UTILIZATION OF SHALLOT BIO-WASTE AS A NATURAL SOURCE OF DIETARY CELLULOSE AND MICROCRYSTALLINE CELLULOSE AND ITS FOOD APPLICATION

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Submitted by

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CERTIFICATE

This is to certify that the thesis entitled "**Utilization of shallot bio-waste as a natural source of dietary cellulose and microcrystalline cellulose and its food application** " submitted in partial fulfilment of the requirements for the award of the degree of **Doctor of Philosophy in Biotechnology** to the Bharathidasan University. Tiruchirappalli is a record of bonafide research work carried out at **National Institute of Food Technology, Entrepreneurship and Management , Thanjavur** (formerly Indian Institute of Food Processing Technology, Thanjavur) by **Shery M Varghese (1D. No. 201801DB007; Ref No.27784-10/Ph.D.K7/Biotechnology/Full time/January 2018/Date 05.01.2018)** under my supervision and guidance and that no part of the thesis has been submitted for the award of any other degree, diploma, fellowship or other similar titles or prizes. However, some part of the research work has been published in peer-reviewed reputed scientific journals (copies enclosed).

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Dedicated to Amma and Appa who has been a constant source of love, support, care and sacrifice throughout their life. To my brother and grandparents for their unconditional prayer for not giving up my path. To my best friend, almighty god for guidance, strength, protection and emotional power...

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(Shery M Varghese)

Abstract

Allium cepa var. aggregatum is a small-sized onion harvested from southern India, well-known for its distinct flavour with greater pungency and is widely used in many cuisines. During the harvesting and processing of shallot, 35% of waste is produced, specifically onion skins, outer fleshy scales, stalk, and blossom. These wastes are biodegradable, however, they are not suitable for a landfill due to the increased development rate of opportunistic infections, which have a negative influence on the environment. Shallot biowaste is a rich source of crude fibre with 55.97, 49.72 and 41.49% in skin, stalk and petiole, respectively. Because of expanded MCC production, a huge quantity of hardwoods and certain softwoods are used as raw materials. These will contribute to increased pollution and rapid deforestation. Thus, the above problem can be overcome by utilising biomass from non-woody agricultural waste like shallot wastes.

Thus, the objective of this research is to transform the underutilised shallot biowaste streams such as peel (P), stalk (S) and petiole (PT) into a low-cost and renewable source of valuable nutritional cellulose and microcrystalline cellulose. Peel along stalk and petiole were found to be with relatively high fibre content with 54.76, 49.51, and 40.73%, respectively.

Two major hydrothermal pre-treatments (hot water(W) & steam (A)) are used for the extraction of α-cellulose. WP (hot water pre-treated peel) had the highest yield per cent of α-cellulose, followed by WS (hot water pre-treated stalk) and WPT (hot water pre-treated petiole), with values of 34.48, 31.58 and 30.41% respectively. Also, increased total ash was found in WP, WS & WPT of 3.11, 3.64, and 2.74 % respectively. The autoclave pre-treatment, cellulose samples treated at 120°C for 30 min at 15psi and 2.0N NaOH hydrolysis exhibited quite good quality cellulose with yield of 29.31, 27.94, and 25.70% in APM (steam assisted pre-treated Peel extracted MCC), ASM (steam assisted pre-treated stalk extracted MCC), and APTM (steam assisted pre-treated petiole extracted MCC) respectively. Since, these pre-

treatments, 21.98, 17.36, and 16.92% of SBWs MCC are recovered from AS (steam pre-treated stalk), AP (steam pre-treated peel), and WPT (hot water pre-treated petiole), with reduced ash contents of 0.065, 0.185, and 1.195 percent, respectively.

The most prominent, characteristic band detected by FT-IR spectra was between 1430-1429, 1364, 1157, and 895cm^{-1}. These peaks elucidate a sharp peak bending symmetric CH_2 and asymmetric CH representing a pure crystalline cellulose band. Induced pre-treatment and chemical hydrolysis are critical in the conversion of cellulose to MCC. In comparison to hot water treatment, autoclave aided pre-treated samples exhibited well-defined cleavage, porous, and smooth surfaces in SEM images. The particle size and agglomerations are mostly determined by the composition of the raw material, pre-treatments, and hydrolysis methods. The longer the reaction period, the greater the aggregates and, consequently, the surface area and interior area of the particle in micro fibrils. The MCC samples such as CM (17.56), ASM, APTM and WSM (SBWs MCC) had the average particle size of 16.14, 21.82 & 24.84μm respectively.

Crystalline index was considerably high in autoclaved pre-treated MCC samples specifically APTM and ASM with 79.9 and 76.75%, respectively, via the peak intensity approach. While, peak area convulsion formulae, APTM & ASM, showed excellent CI of 83.68 and 82.50%, respectively, when compared to the CM. As increase in crystallinity index, thermal properties also seem increased in the autoclaved pre-treated samples. Further the thermal stability was observed by DSC technique and found an increased thermal stability was found in SBWs MCC samples of ASM, APM and WPTM with values of 263, 259 and 255°C respectively.

Low fat stirred yogurt was prepared from skim milk with the addition of SBW's MCC (0.25 to 1.0%) and transferred to a sterilized glass bottle with skimmed milk control to different temperatures at 37˚C (T_1) & 45°C (T_2). The addition of SBW's MCC had not significantly

affected the whole fermentation process. Rheological parameters like flow index(n) showed improvements as compared to Commercial MCC samples and blank samples. The highest flow index was observed in PT_4 and S_4 (1%) with values of 5.947 and 4.866 respectively at T_2 than CM and SMC. The highest firmness was observed in the PTY_4 samples along with PY_4 with values of 29.54 and 26.45g at T_2 respectively with significant indifference ($p<0.05$) between the samples. Also, consistency of the samples treated at T_2 showed increased values mainly in P_4, WMC and P_3 with values of 684.5 and 576.7g.s respectively with no significant difference from other samples. The serum separation was higher in SMC with 68% at T_1. While at T_2, the same phenomenon was observed with 66% at 15min. The least serum separation of 40, 42.7% was observed after 15 min at T_2 by addition of PT_4 and P_4 as compared to SMC. A higher rate of syneresis was found in SMC at 68 % (T_2). While MCC added samples, PT_4 and P_4 were more stable than other concentrations. Significantly Increased WHC ($P\leq0.05$) was found out in P_4 and PT_4 with 66.6 and 64.45% respectively at T_2 as compared to control samples.

Because of the lactic acid produced during the fermentation process, plain yoghurt has a sour and bland flavour. Fruits based puree or crush along with sweeteners are additional blended in to yogurt to improve flavour, taste and colour for greater product acceptance. Panellists preferred the fruit crush concentration of 10% (JBY5) for the greatest overall acceptability score compared to other samples. Following the inclusion of the fruit crush and optimised SBW's MCC (1%) in low fat stirred plain yoghurt, it was refrigerated for 28 days. After fermenting, the addition of the fruit crush decreased the pH and also there was a significant decrease for overall yogurt samples at the end of the cold storage period. TSS, moisture, protein, and fat were somewhat reduced during storage, however, SBW's MCC remained constant in concentration in each sample when compared to SMC (control sample without addition of MCC).

The colour parameters of MCC's incorporated stirred yogurt showed a slight decrease as compared to SMC. With the addition of fruit puree (10%) along with SBW's MCC, PTFY (Petiole MCC) showed better lightness as compared to PFY (Peel MCC) and SFY (Stalk MCC). Also, the syneresis rate was significantly increased after the 14th day. But as compared to control, the syneresis rate was lowered in PFY and SFY and also lower than SMF (without MCC) control. The probiotic survival of *Lactobacillus bulgaricus* and *Lactobacillus cremoris* was observed on the 14th day of the product storage period. SMF showed the highest probiotic growth along with SFY and PTFY of 8.73, 7.6 and 7.46 CFU/ml. Textural attributes showed an inconsistent deviation during the storage period. But, the 14th day showed better firmness in PFY and PTFY samples with 25.63 and 25.19g respectively but showed a slight reduction on the 28th day. Panellists assessed the acceptability of stirred yogurt preparations with scores of 8.1 and 7.55 points which indicated that they liked JSY (Stalk MCC) and JPT (Petiole MCC) samples. The results showed better acceptability index in JSY and JPY (Peel MCC) of 85.75 and 84.91%. MCC samples showed excellent syneresis reduction in stirred yogurt by improving the firmness and cohesiveness by desirable structural changes to the yoghurt matrix during the whole process.

In a nutshell, optimized SBWs MCC samples were proven to be a source of good quality MCC with economic potential. Also, overall results pointed out that, microcrystalline cellulose from SBWs has a lot of potential as a functional component in new product development with health benefits such as dietary fibre, binder, thickener, stabiliser, pickering emulsifier, bulking agent, anticaking agent and fat replacer.

Key words: shallot biowastes, microcrystalline cellulose, crystallinity, low fat stirred yogurt, fat replacer

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SYMBOLS AND ABBREVIATIONS

Symbols

g	:	Gram
%	:	Percentage
mg	:	Milligram
ml	:	Millilitre
rpm	:	Revolution per minute
°C	:	Degree Celsius
mV	:	Millivolt
OD	:	Optical density
ΔH_f	:	Enthalpy of decomposition
2θ	:	Two theta
MMT	:	Million metric tonnes
µm	:	Micrometer
nm	:	nanometer
D	:	Crystalline size
g/ml	:	Gram per millilitre
CFU/ml	:	Colony forming unit per mililitre
Pa.s	:	Pascal seconds
Jg^{-2}	:	Joules per square gram
≤	:	Less than or equal
Min	:	Minute
h	:	Hour
cm^{-1}	:	Inverse centimetre

kV	:	Kilo volt
g/cm^3	:	Gram per cubic centimeter
mS/cm	:	Millisiemens per centimetre
°B	:	Degree Brix
g.s	:	Gram second
ha	:	Hectare
CFU/ml	:	Colony forming unit per millilitre
2θ	:	Two theta

Abbreviations

Association of Official Analytical Chemist	:	AOAC
Shallot waste streams	:	SBWs
Cellulose		CC
Microcrystalline cellulose	:	MCC
Steam Assisted Pre-treatment	:	A
Hot Water Assisted Pre-treatment	:	W
Commercial	:	C
Petiole	:	PT
Peel	:	P
Stalk	:	S
Hot water pre-treated peel	:	WP
Hot water pre-treated stalk	:	WS
Hot water pre-treated petiole	:	WPT
Steam pre-treated peel	:	AP
Steam pre-treated stalk	:	AS

Steam pre-treated petiole	:	APT
hot water assisted peel microcrystalline cellulose	:	WPM
hot water assisted stalk microcrystalline cellulose	:	WSM
hot water assisted petiole microcrystalline cellulose	:	WPTM
Steam pre-treated peel microcrystalline cellulose	:	APM
Steam pre-treated stalk microcrystalline cellulose	:	ASM
Steam pre-treated petiole microcrystalline cellulose	:	APTM
Normality	:	N
Equation	:	eqn
Sodium hydroxide	:	NaOH
Sodium hypochlorite	:	NaOCl
Acetic acid	:	CH_3COOH
Hydrochloric acid	:	HCL
Crystalline index	:	CI
Acceptability Index	:	AI
Optical density	:	OD
Revolution per minute	:	RPM
Million metric tonnes	:	MMT
Fourier transform infrared	:	FTIR
Scanning electron micrograph	:	SEM
Differential scanning calorimetry	:	DSC
Crystalline size	:	D
X-ray diffraction	:	XRD
Bulk density	:	BD
True density	:	Tr

Tapped density	:	TD
Hausner's ratio	:	HR
Water holding capacity	:	WHC
Swelling capacity	:	SC
Water solubility index	:	WSI
Oil holding capacity	:	OHC
Foaming capacity	:	FC
Foaming stability	:	FS
Foaming capacity	:	FC
Foaming stability	:	FS
Carr's index	:	CI
Lightness	:	L*
Redness to greenness	:	a*
Yellowness to blueness	:	b*
Enthalpy of decomposition	:	ΔH_f
Endothermic melting temperature	:	T_M
Onset temperature	:	T_0
Peak maximum temperature	:	TP
Total viable count	:	TVC
Weight	:	W
Volume	:	V
Lactic acid bacteria	:	LAB
Low fat stirred plain yogurt	:	LFSPY
Low fat fruit flavoured stirred yogurt	:	LFFSY

CHAPTER 1

INTRODUCTION

Agro-industrial waste was regarded as the unrecognized raw material that disposed in harvests and production inlets during processing. The dumping of these waste stream leads to large environmental problems. This problem can be solved by sustainable utilization of biomass through interdisciplinary approach that can contribute the development of innovative value-added products by technology interventions. Thus, extracted byproducts are eco-friendly and economically potential. This leads to zero-waste processing and enhances the profits for farmers and stakeholders. Allium cepa. var aggregatum, commonly known as shallot largely cultivated in European and Asian countries. During its harvesting and processing, a huge quantity of the waste streams was generated and dumped or burned. The need for the present research on the utilization of shallot waste streams such as peel, stalk and petiole into derived forms of cellulose and microcrystalline cellulose by an effective comprehensive low-cost pre-treatments and extraction parameters. These primes will improve the resource efficiency and restoration of environmental safety.

1.1 Background

Enormous agro-industrial biomass was generated from the harvesting and processing of food products and is increasing due to the huge demand from the growing population for each year(Oluseun Adejumo & Adebukola Adebiyi, 2021). Inappropriate handling of these waste streams leads to burning and unprotected landfills which produce unfavourable odour, soil and water pollution and are harmful to human health (Tripathi et al., 2019). India is the second-largest producer of crops which also generates a tremendous quantity of agro-

industrial biomass. This unexploited biomass was largely utilized as biofuel, bioenergy and construction material (Gontard et al., 2018b).

According to the ministry of new and renewable energy (2021), 32% of total primary energy was produced from agricultural biomass. Around 250 million metric tonnes of agro-industrial biomass was produced per annum (*Current Status | Ministry of New and Renewable Energy, Government of India*, n.d.).

The sourcing of raw material for the extraction of the by-products from renewable biomass was very challenging for the researcher. This issue that can be solved by promoting this biomass as an alternative and inexpensive natural source in various food applications.

In spite of farming activities, each phase of agriculture and the food supply chain is also associated with the generation of biomass(Tian et al., 2018). Fig. 1.1 illustrate the broad type of agricultural biomass involved in various stages like preparation, transportation, manufacturing, processing, preservation and consumption (Galanakis, 2015; Otles et al., 2015).

Lignocellulose is the major material utilized as a source of parent cellulose for the production of paper, food additives, building composite, biodegradable packaging material, pharmaceutical, medical accessories, genetic engineering and nutraceutical ingredients etc., mainly derived from hardwood and softwood (Ling et al., 2021; Thomsen et al., 2008).

Increased population cause increased demand for essential material which cause huge depletion of renewable sources such as forest and its residues which leads to the greenhouse effect, carbon dioxide emission, air and water pollution and global warming (Hassan et al., 2018). India ranks the second-largest producer of fresh vegetables and fruits. The 21.4% of biomass was expelled during harvesting and processing in the form of leaves,

peel, seeds and stem portions which were outsourced for landfilling and incineration (Sadh et al., 2018). Many literatures showed the benefits in the recovery of many bioactive components and pigments from agro-industrial waste but lack coverage in the utilization of lignocellulose for processing into modified cellulose.

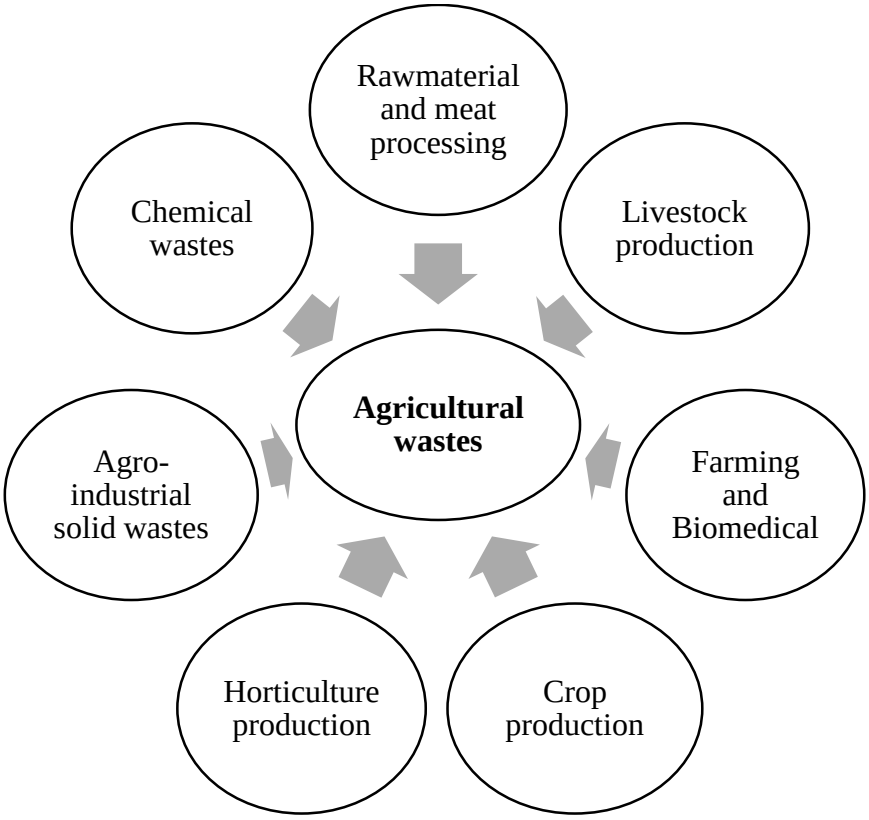


Figure 1.1 Source of agricultural solids wastes during post harvesting processing

1.2 Shallot (Allium cepa var. Aggregatum)

Allium cepa L is the second largest root vegetable belonging to the *Liliaceae* family and biennial plant commonly known as Onion recognized as a therapeutic herb and top-notch spice in culinary ingredients on various cuisines worldwide due to its pungent flavour and sweet taste(A. Nile et al., 2018). Major cultivating countries are from Central Asia and

limited areas of European countries which includes China (31.19%), India (28.51%), United States (3.96%), Egypt (3.85%), Turkey (2.75%) and Pakistan (2.60%)(Food and Agriculture Organization of the United Nations (FAO), 2018). Onion varieties are classified according to their colour (Red, Yellow & White), size (large, medium and Small) and flavour (mild & pungent)(Lim, 2015). According to APEDA (2020-21), India is the second largest country that exports the red onion varieties of 1578016.59 MT to the other countries worth Rs.2826.5Cr. The major producers of onion in India are Maharashtra, Karnataka, Andhra Pradesh along with Rajasthan, Haryana and Telangana (APEDA, n.d.).

Moreover, one variety that got more popular and commercialized due to its culinary and therapeutic property was shallot which was commonly known as Small onion or Griselle(Peterson, 2000). Shallot was classified into two varieties as *Allium oschaninii* and *Allium cepa var. aggregatum. Allium oschaninii* commonly produced in France, were commonly termed as French grey shallot or grisaille which is true shallot with a single bulb whereas *Allium cepa var. aggregatum* is generally termed as multiplier onion due to its multiple bulb formation from a single plant with the subsequent smaller size as compared to griselle or small onion(Swamy & Veere Gowda, 2006). These varieties are largely cultivated in Europe and Asian countries such as India, Singapore, Indonesia, and Brunei.

The shallot cultivation was mainly concentrated in the southern part of India, among this, 75% of shallot production was accounted by Tamil Nadu which shares 12 tonnes/ha per year. In the southern part of India, shallot or small onion plays an important part in cuisines owing to its delicate pungency and taste. Also, an inseparable ingredient in traditional medicine due to its therapeutic properties(Saraswathi et al., 2017; *Small Onion Cultivation Will Be Profitable, TNAU _ Business Standard News*, n.d.).

1.3 Shallot waste

Increased consumer demand of shallot onion will lead to increased production and processing of shallots produce a large amount of agricultural and industrial waste biomass. As per the cultivation and processing of shallot onion, 37 percent of the conventionally non-edible peel (skin), leaves (stalk), pseudostem (petiole) and flower, as well as a small fraction of the roots, are disposed. Therefore, increased dumping of this waste streams face a challenging drawback in the disposal of shallot waste due to the increasing growth rate of pathogenic organisms and the extremely pungent odour of these wastes, they should not be buried or used as feed (Bhosale et al., 2022a). Hence, there is a need to find a sustainable

Figure 1.2 Waste streams derived from post harvesting and processing of shallot onion

agricultural processes and develop solutions for the recovering of key natural products. thus the sustainable production of allium requires valorisation of shallot biowaste streams as food waste into food by-products. These valuable byproducts often used as functional and nutraceutical ingredients in food development, pharmaceuticals, packaging and cosmetics industries.

1.4 Valorisation of shallot waste streams

Underutilization of bio-waste led to increased problems by landfilling which cause an impact on environmental pollution and human health. These biowastes are tremendous and

considered as an alternative source of raw material for different fields. In recent years, researchers are keener in the utilization of biomass into value-added products due to the rich source of functional components such as bioactive components, pigments and polysaccharides which are of great interest to the processors. The application of onion waste streams depends on the presence of various constituents in each part of the onion morphology.

The onion peel consists of quercetin and its derivatives having an antioxidant activity that can be widely utilized for the treatment of various cancers found in the liver, kidney, breast and ovaries. Integration of various extraction and purification methods was challenging but more cost-effective than other sources (Benitez et al., 2011). It also comprises 48 fold flavonol glucosides as compared to fresh onion which showed effective functionality such as antioxidant activity, enzyme and cancer cell inhibitory action etc.,

The data proved that onion peel can be an alternative raw material for the resupply of natural flavonoid derivatives in the field of medicines and cosmetic applications (Choi et al., 2015b). Many studies were performed to find out the components present in onion peel and stalk which are an excellent source of sixteen variances of flavanols which consist of aglycone and glycosylated derivatives of quercetins etc., (A. Nile et al., 2018). Choi and team (2015) also demonstrated the extraction of 98.5% of natural glucose through enzymatic treatments which were highly efficient for the recovery of natural sugar. Onion waste streams are also a good source of polyphenolic acids such as protocatechuic, vanillic and ferulic acid mainly found in the inner and outer scales of onion. Other bioactive components such as diallyl disulphide and diallyl trisulphides were found at a reasonable level too (Shea et al., 2012).

Studies also showed onion stalk and peel are good sources of soluble and insoluble dietary fibre which includes pectin, lignin, hemicellulose and cellulose (Reddy & Rhim, 2018). In prior literature, a lack of research was found in the exploration of the application of dietary fibre from different onion waste streams due to the nature of onion waste streams. Depending on the *Allium cepa* varieties, the estimated quantity of dietary fibre varies in different parts of waste streams. The total dietary fibre content found in the peel layer was 68.3%. Around 27–34% of pectin and pectic substances were observed in onion peel, which can also be an alternative source for a new generation of a prebiotic component known as pectic oligosaccharides, which helps in the production of short-chain fatty acids in the process of colonic fermentation (Benítez et al., 2011). The aforementioned constituents in waste streams can be explored for the development of non-food and food-grade raw materials for the production of various products using various sustainable technologies. Previously, due to its extreme pungent smell, not used for the feedstock and inorganic fertilizer. Some of the researchers also found the technologies for conversion of onion waste converted to bioethanol, biochar, dyes, carbon paper and activated carbon, lignocellulose adsorbent etc., which are low cost and were reusable (Chadorshabi et al., 2022a; Mourtzinos et al., 2018b; Sharma et al., 2016a, 2016b; Sun-Waterhouse et al., 2008).

Shallot waste streams are excellent source of polysaccharides which were unexplored due to more consideration for the extraction of phytocomponents which can act as an antioxidant, anti-inflammatory, apoptotic and antitumor components, especially for pharma industries (Pezeshk et al., 2011; Phaiphan et al., 2019; Seyfi et al., 2010). Also, researchers were studied only on peel portion of onion varieties. Until now, no research has been conducted on the other parts of onion waste, such as the stalk, petiole, and flower. However, it is a structurally excellent source of soluble and insoluble dietary fibre, which

can be utilised for the extraction of oligosaccharides, pectin, and dietary cellulose. Chemical characterization and understanding of bioactivities in these parts are vital for the creation of optimum and efficient waste recycling strategies. Thus, it can be utilised in the production of pharmaceutical, nutraceuticals, and functional ingredients in the food system.

Morphology and functional properties of insoluble fiber can be improved with the help of some technological interventions namely modified hydrothermal, microwave, ultrasound, enzymatic methods along with combination of chemical process. As a result, utilising these non-edible parts and transforming them into valuable products will profit onion growers and production companies (Panesar & Bali, 2015; Shea et al., 2012).

For many centuries, cellulose is a versatile biodegradable polymer that was produced from hardwood, softwood as well as cottonseed linters. As rapid depletion of deforestation causes non-availability of wood which triggered hazardous environmental pollution. Alternatively, many kinds of research proved that agro-industrial bio-wastes are inexpensive and abundantly available that can be recycled for the production of parent cellulose. These parent materials can further act as a precursor of cellulose derivatives via., the thermal, enzymatic and chemical procedure to perform outstanding physical, functional and perfunctory properties in food applications (Dyk et al., 2013; Pastell et al., 2019).

In these derivatives, the popularity of microcrystalline cellulose (MCC) gained high ground in various fields such as pharmaceutical, cosmetics, packaging, and new food product development. MCC isolated from agro-industrial waste such as sorghum, wheat, rice, tea, sago, jute, coir, corn, date palm, oil palm, groundnut, coffee etc., showed comparatively good characteristics of commercial grade (Collazo-Bigliardi et al., 2018a; Harini et al., 2018).

MCC is a short chain of glucose moiety to form a linear crystalline lateral configuration by depolymerisation of vigorous acid hydrolysis that destroys the amorphous region of cellulose through different hydrothermal methods. Currently, microcrystalline cellulose (MCC) is popular as a versatile additive along with combined attributes such as thickener, stabilizer and fat replacer in the dairy industry (BeMiller, 2019; Bhandari, Roy Maulik, et al., 2020a; Hindi & Z Hindi, 2017). It shows multidimensional functionality with stable viscosity, gelling power, emulsification capacity, textural modifier, fat replacer or mimicker, heat resistance and water binder.

The milk industry is the largest in the world, with a wide range of milk products. Among the countries, India has the largest milk sector with an annual production of 198.4MMT with growth rate of 6.2% (NDDB, 2020). Milk producers and processors face major challenges in the marketing of dairy products due to presence of unsaturated fatty acids and lactose, which cause hypercholesteremia and lactose intolerance. Nowadays, consumers are more concentrated and choosy on nutritious and healthy diets to overcome lifestyle diseases. The criteria for selecting a food, do not depend on the appearance or taste or satiety but also on the nutrient supply and ability to prevent nutrition-based lifestyle diseases.

From these viewpoints, yogurt gained more popularity being a probiotic food that is the carrier of probiotic microbes, protein, and essential minerals that could promote a stable immune system by modifying the gut microflora, reduction of carcinogens and β-glucuronidase and urogenital infections, anti-inflammation and allergies. Thus, with the increasing demand of consumers, processers are more focussed on the development of low-fat based yogurt that is marketed and consumed in the form of plain, set and, stirred types incorporated with additives.

According to market status, the growth rate of drinkable or stirred yogurt is predicted to reach a Compound Annual Growth Rate of 18% in 2023 with the takeover of 30% of the Indian yogurt market (Drinkable Yogurt Market Overview 2017-2023). Moreover, low fat based yogurt products face the major challenges due to the absence of fat micelles such as flavour deformity, textural imbalance, mouthfeel and appearance defects. To overcome this defects, application of hydrocolloids can play an important role to overcome the deformities such as appearance, texture, viscosity consistency, mouthfeel and especially removing the serum separation (Fazilah et al., 2018) .

Major hydrocolloids sourced in dairy products are cellulose, starch sodium caseinate, whey protein concentrate, carboxymethylcellulose, pectin and gums from plant or animal resources (Hashemi & Hosseini, 2021; Hassan et al., 2018). Especially, modified cellulose becoming more popular in recent years due to its better air entrapment, water holding capacity, creaminess, binding capacity and stabilizer (BeMiller, 2019). In modified cellulose, microcrystalline cellulose and carboxymethylcellulose were mainly utilized in milk beverages and ice cream products as a stabilizer and thickening agent due to its good water holding capacity to form a gel-like structure that improvises the overall attributes and rheological properties.

Therefore, in the current study, it is planned to incorporate shallot biowaste MCCs as a dietary fibre in yogurts products and analyse the effects.

1.5. Requisites of research

The demand for microcrystalline cellulose application is rising with a CAGR rate of 6% from 2019-to 2027. Shallot biowaste is the rich source of crude fibre with 55.97, 49.72 and 41.49% in skin, stalk and petiole respectively which includes lignin, hemicellulose and

cellulose. This cellulose can be further modified depending on the utilization as a gelling agent, fat replacer, texturizing and thickening agent in various food product development(Bhosale et al., 2020; Nair et al., 2021; Thivya et al., 2021a).

Hence the current study focuses on the extraction of cellulose from shallot biowastes and the conversion to MCC. Further, the application of MCC in food products as a fat replacer in low fat stirred fruit yogurt and positive correlation with physical, chemical, microbiological properties along with improved textural characteristics were also studied.

1.6 Objectives

Agreeing to the literatures, the following objectives were outlined to extract and analyse the cellulose and its derivatives from the shallot bio waste and further incorporation and safety assessment of value-added food products.

1. To extract, characterize and purify the microcrystalline cellulose from shallot waste streams
2. To incorporate the extracted dietary MCC in food products
3. To study the prebiotic and functional properties of shallot biowaste streams extracted MCC samples in developed products and its shelf life

CHAPTER 2

REVIEW OF LITERATURE

This chapter presents an outline of the study undertaken by many investigators to convert agro-industrial biowastes into value-added products. It primarily focuses on agro-industrial waste generation, the various types of agro-industrial biomass, and the exploitation of lignocellulose in various waste management systems. Also, explains the pre-treatments and extraction methods of Microcrystalline cellulose from diverse agro-industrial waste biomass and its use as a hydrocolloid in a wide range of industries. Also explores the benefits in numerous applications and therapeutic functions through replacement in various industries

.2.1 Agro-industrial waste

The significance of agricultural output in socioeconomic development cannot be under estimated. The necessity to feed the world's ever-growing population is being highlighted by steadily increasing agricultural production (Isaac Oluseun Adejumo, 2020). The increasing population's demand for food and other resources has resulted in the expansion of agro-industrial endeavours (Tripathi 2019). According to the Food and Agriculture Organization, one-third of the total mass of digestible portions of food produced for human use is lost each year (FAO, 2018). Agricultural by-products and wastes are made up of a variety of complicated components, many of which have a considerable financial value. As the agro-industry grows progressively mechanised, the waste generated has a significant environmental impact. Agriculture generates 5 million tonnes of waste annually (Rajeev Ravindran 2018). The fast growth of population and industrialization generates a massive amount of waste streams of 170 billion tonnes per year. As a result, agriculture and industry are inextricably linked. However, as a byproduct, the food companies generate highly

polluting biomass. To complete the agro-industry chain, waste streams must be carefully maintained to collect harmless material used in agribusiness as an efficient approach and sustainable.

2.2 Types of biowaste streams

According to prior literature, the sources of waste streams can be categorised into four groups: agricultural waste, forest, industrial, and food waste. Under this, agro-industrial wastes, both agriculture and industrial waste, were generating huge sources of biomass, which were classified into six sources and shown in fig 2.1. The three main sources are agriculture, residential, and industrial processes. In these, agricultural waste streams primarily comprise agricultural and livestock biomass, as well as municipal bio-waste. These organic wastes are expelled from urban areas; municipal waste, market waste, and food streams; and manufacturing industries that include textiles, food, and forest resources. Moreover, a large percentage of these industries rely on agricultural production for natural resources (Ravindran et al., 2022).

Agriculture wastes are further classified as field wastes and process wastes. Field waste are the remnants after the harvesting of crops. It includes leaves, stalks, flowers, seeds, and stems, whereas process wastes are remnants that remain after the crop has been processed into another useful sources (Pardeep Kumar Sadh 2018). Cereal crops account for a significant portion of the massive amounts of agricultural residue generated worldwide per year. Globally, cereal straw (stem, leaves, and rind debris) accounts for 66 % of residual plant biomass, with poor nations producing more than 60% of these waste streams (Tripathi et al., 2019). Sugarcane waste streams includes stems and leaves are the second largest providers, followed by plantation crops, roots and tubers, fruits, and vegetables. These resource have the potential to be used in the energy production.

Thus, utilization of these agro-industrial waste streams was critical for aiding the decoupling of human development and economic well-being from resource utilization, minimizing environmental constraints, getting a negative impact on biodiversity, and endangering world food security. It is a seemingly limitless stock of biowaste which might pose environmental and economic challenges. These could be transformed into biofuels and organic goods via sequential bioconversion within the framework of the economy and must be recognized as waste streams. For example, by upgrading vegetable by-products and processing waste streams, people are being used to create unique products such as dietary fibres, food flavours, supplements, phytochemicals, glucosinolates, protein isolates, pectin, phytonutrients, and enzymes. Milk and milk product potential wastes contain proteins, peptides, salts and ions, lipid molecules, and sugars. Furthermore, meat-

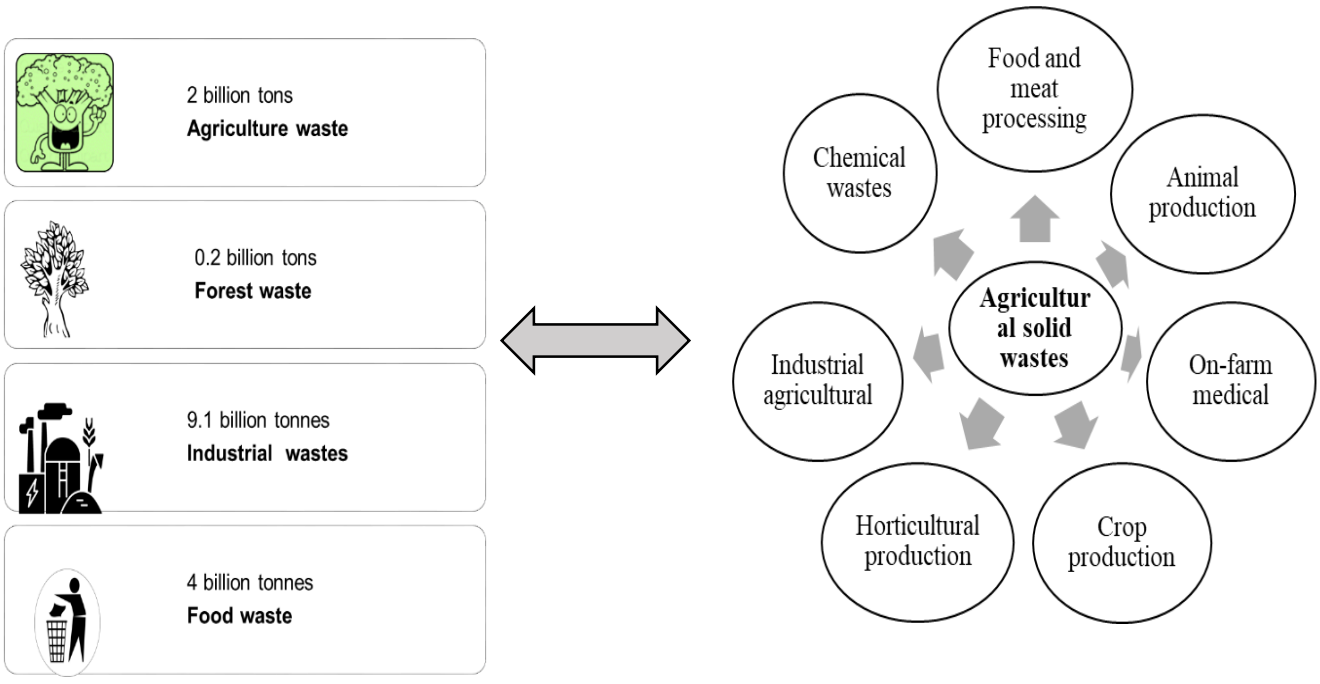


Figure 2.1 Production mass and type of waste streams generated from agro-industrial waste

processing biomass may be a significant source of protein and functional hydrolysates (Galanakis, 2015). Thus, biowaste valorisation in the food processing sector might be regarded via the "zero waste economy" perspective. Furthermore, this trash may be converted into new value-added items, providing specific aims for new sectors while conserving significant energy. *Allium cepa L*. is one of the largest root vegetables produced worldwide. The onion varieties are the world's second most important vegetables after tomatoes. The past decades, onion production and export has increased by more than 25%. Increased consumption of processed onions had also resulted in significant wastage. As a consequence, over 600,000 tonnes of onion biowastes are produced annually in the EU (European Union), predominantly in Spain, United Kingdom, Holland, Germany, Japan, Netherlands and Russia. *Allium cepa var. aggregatum* is a small-sized onion, one of the varieties included in *Allium cepa L*., which is commonly known as shallot or small onion and is more famous in the southern parts of India, especially Tamil Nadu, where 75% of shallots are cultivated with the production of 12 T/ha.

During production and processing, 25–30% of biomass portions are wasted, which is highly perishable and creates a vast unease about environmental pollution (Kambli et al., 2017). Commercialization of these products will lead to increased revenue as well as employment opportunities to improve people’s livelihoods.

The following section mainly discussed the botanical history, varieties, and cultivation of onion and shallot species. Additionally, waste generated during harvesting and processing and the application of by-products in different fields were explained.

2.3 Allium family

Allium is a recognised genus with over 900 species and is typically found in Central Asia, notably Afghanistan, Tajikistan, Pakistan, and sections of Serbia, and is mostly produced in China and India (Bede & Zaixiang, 2020). Also cultivated in Europe, North America, and Africa. According to Food and Agriculture Organization statistics (2016), Asia was the largest producer of Allium species, accounting for 65.5%, and European countries account for 10.9%. In Asia, China has become a major producer of Allium species, producing 23.84 MT of onion per year, followed by India, with supplies approximately 19.42 MT of onion per year, and Egypt and the United States of America, each produce approximately 30MT of onion simultaneously.

Allium species are differentiated by their herbaceous geophytes with a distinct bulb variety that is also grown in the form of rhizomes and has a notable pungent onion-like aroma and taste. Almost 30 varieties have been employed for food applications with onions, garlic, leeks chives being the most popular and scientifically well known in *Allium sativum L, Allium ampeloprasum as well as Allium schoenoprasum* species.

The genus Allium is used in many herbal medicines for the treatment of fever, pain, wheeze, skin infections, respiratory problems, flu, pneumonia, whooping cough, bronchitis, soreness, meningitis, arthritis, laryngitis blood clotting, cardiovascular diseases, liver infections, viral diseases and other pathogenic infections. Beneficial effects of the Allium family have often been linked to phytoconstituents contents with substantial pharmacokinetic properties. Numerous secondary metabolites have been isolated and categorised with significant action within the major Allium species (Alam et al., 2022).

2.3.1 History of the allium family

Ancient literature going back to approximately 1500 B.C., namely the Egyptian papyrus "Codex Elsers", defined at least 22 Allium-based medicines to treat a variety of diseases such as migraines, extreme fatigue, and throat infections. Hippocrates, a Greek physician, advocated garlic to alleviate pneumonia and injuries, including as a diuretic, in the 4th century B.C.

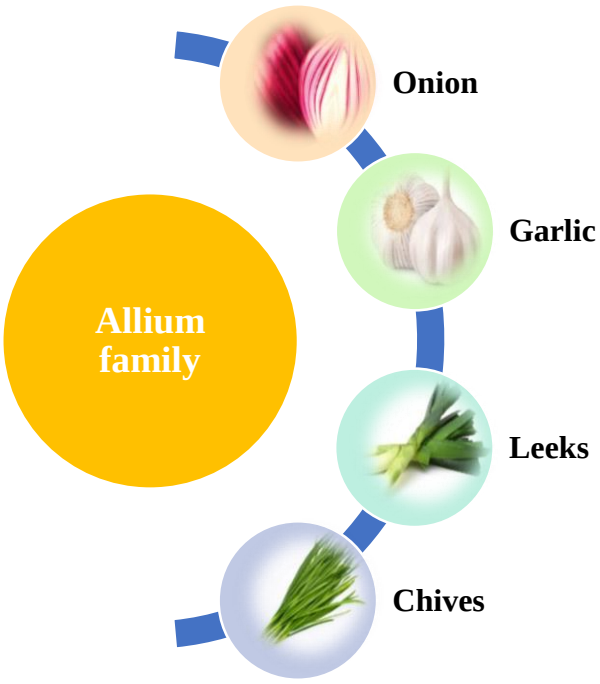


Figure 2.2 Schematic representation of major allium species cultivated worldwide

Throughout the first century (AD), the Indians compiled their decade's pharmaceutical knowledge into a single collection titled "Charaka Samhita," in which garlic and onion were described as diuretics, rheumatic healing agents, including treatment for cardiovascular, gastrointestinal, and ophthalmic ailments. Also Pliny the Elder, recorded sixty garlic-based formulas that were often used to cure diseases such as nausea, ulcers, rheumatism, and haemorrhoids. These ancient literatures identified the genus Allium as a true goldmine of curative herbs, leading to its widespread popularity among physicians and herbalists.

Allium variety of species thrives in broad, bright, somewhat dry environments in dry and moderately humid regions. Allium species can be located in a variety of habitats, including woods, European subalpine pastures, subalpine and alpine grasslands in the Himalayan and Central Asian high ranges, as well as areas near riverbeds. Some of the variants survive in saline and alkaline circumstances. There are allium species that bloom in the springtime, midsummer, and autumn. It may go inactive throughout the summer or winter. Numerous varieties of annual growth are constrained to the first few months in spring season and early summer climate when the cycle between leaf sprouts to seed development will be completed within the three months.

2.3.2 Onion

Onion is the most commonly cultivated root vegetable in the world, accounts for a significant proportion of the food processing industries is being familiarized in a range of functionalities. Onions are well-known for their high polyphenolic compound content, specifically phenolics, anthocyanins, and flavonoids, which have a wide range of biological activities. Apart from the polyphenolic compounds, onions contain a substantial amount of carbohydrates, which has generated a huge interest in healthcare professionals due to their pharmacological uses. it also offers dietary complexity and promising properties in nutraceutical food production, and also antioxidant, antibacterial, anticancer, and anti-browning characteristics.

Onions are categorized per their aroma as sweet or non-sweet, along with their pigmentation as red, white, or yellow, which also constitute the most prominent type of such varieties. Red onions exhibited better antioxidant activity than white and yellow onions because they included more anthocyanins and phenolics, including quercetin and its

derivative. Onions are extensively cultivated and used as a vegetable as well as a spice around the world for a range of functional culinary purposes, food supplements production, and the making of pharmaceutical applications given the vital nutritional, functionality, and therapeutic capabilities (A. Nile et al., 2018, 2021; S. H. Nile et al., 2017).

2.3.3 Production scenario of onion

Onions are grown in approximately 170 countries around the world for both domestic and international trade. Colour, shape, dry matter content, and pungency are all factors which vary in onions grown around the world. This diversity is also reflected in the species' ability to adapt to a wide range of environmental conditions. Global onion production has been steadily increasing, from 36.36 million tonnes in 1994 to 99.97 million tonnes in 2019. China is the world's largest onion producer, accounting for nearly 24.91 million tonnes, or 38 percent of global production. Table 2.1 shows that China is followed by India (22.82 million tonnes) and the United States of America (3.17 million tonnes). It is a particularly important vegetable crop, not only for domestic consumption but also as the vegetable with the highest exchange rate. Table 2.1 shows the top ten countries' onion production status.

Table. 2.1 Production status of onion in top ten countries

Country	Annual production (Tonnes)	Total Production (%)	Area production (ha)	Yield (hg/ha)
China	2,49,08,392	24.92%	11,27,609	2,20,896
India	2,28,19,000	22.83%	12,20,000	187,041
USA	31,70,270	3.17%	52,370	605,360
Egypt	30,81,047	3.08%	87,948	3,50,326
Turkey	22,00,000	2.20%	68,713	3,20,172

Pakistan	20,79,593	2.08%	1,48,272	1,40,255
Sudan	19,19,308	1.92%	104,463	1,83,731
Bangladesh	18,02,868	1.80%	1,72,456	1,04,541
Iran	17,79,457	1.78%	45,417	3,91,804
Russia	16,70,129	1.67%	58,226	2,86,836

According to reports, production in India has increased in recent decades as a result of advancements in technology and increased awareness of proper cultivation techniques. In 2019, the total area under cultivation was 1.22 million hectares, with a total production of 22.82 million tonnes, placing it second in the world.

2.4 Shallot

Allium cepa var. aggregatum is also known as shallot, Giselle (little onion), potato onion, multiplier onion, and nested onion. The aggregatum variety may be distinguished genetically and physiologically by aggregates and the formation of bulbs of diverse sizes, forms, and colours, which are influenced by increasing ecological and climatic conditions. Shallot is a popular root vegetable in many different regional cuisines throughout the world. Shallot is a perennial crop that is cultivated year after year, along with its cluster of small bulbs. Shallots, like medicinal herbs, are helpful spices for flavouring foods. Taxonomically, they are the species of proliferating bulb distinguished by their small size.

2.4.1 Morphological characterization of shallot onion

Shallots differ from onion bulbs in such a way that they can proliferate vegetatively through laterals and that a singular shallot bulb often contains numerous initial shoots. Each bulb can be planted, and it will sprout multiple leafy shoots as shown in fig 2.3. Each shoot

then generates a tiny bulb rapidly, developing a clustered group that remains connected to the original base plate. These bulbs could be detached, or the practice can be continued the following seasons. shallot onion appears to be extremely similar to onion morphologically, there are notable variances. Perennial plants with smaller flowers, inflorescences, bulbs, and leaves than big onions(Kamenetsky & Rabinowitch, 2017; Major et al., 2018).

As compared to the leaves of onion, shallot leaves are narrow, delicate, often curved, particularly flatter, practically concave mostly on the inner lining of the leaflets. Buried bulbs are properly matured, including an oval form. The clusters constitute a substantial quantity of shallot bulbs. Upon planting, every bulb sprouts a cluster of interconnected side bulbs. Each new bulb produces its leaves, giving the plant a bushy appearance. On the other hand, big onion normally has a developed petiole (stem) (Krontal et al., 2000).

2.4.2 Cultivation and harvesting

Red to black soils with proper drainage is ideal for onion cultivation. It grows in soil pH limits of 6-7 and throughout mild seasons with few excessive heat and chill conditions.

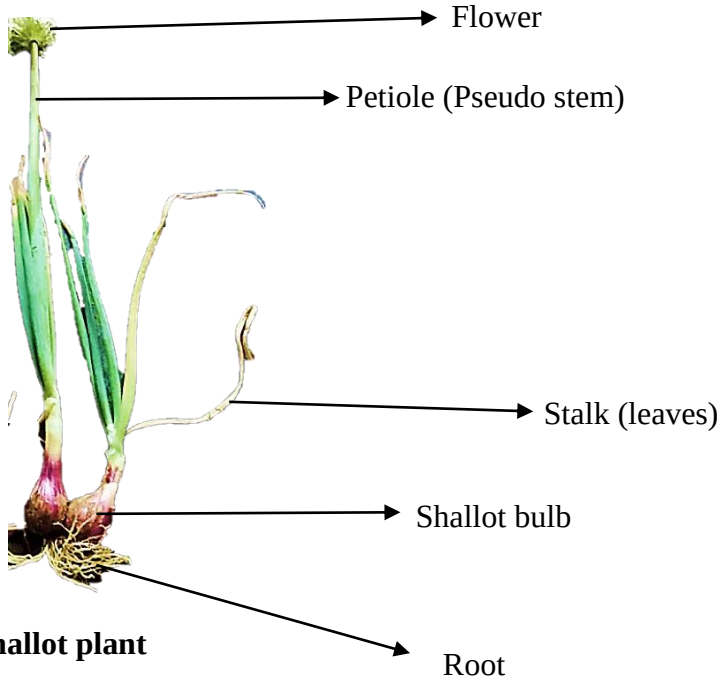


Figure 2.3 Structure of shallot plant

During April – May and October – November, look for medium-sized bulbs. It requires adequate soil moisture during its growing season, however strong rains during bulb germination and bulb formation harm crop growth. Shallots are seeded 7 cm apart on raised beds in sandy loam. By one hectare of planting, approximately 4 tonnes of bulbils are required. Each hectare demands 900–1000 kg of quality seeds. Plants are spaced 15-20 cm apart relying on the cultivar. If the bulbs are dormant, the tips are clipped when planting. Every cluster of a plant will generate two to fifteen small bulbs.

Planting individual bulbs, 5 cm deep and 10 cm apart in rows 30–60 cm apart is how the crop is propagated. Bulbing was facilitated by warm temperatures and prolonged photoperiods. Shallots may also tolerate higher temperatures and humidity levels than onions. As a result, they are a good substitute for onion. When 70- 80% of the leaves are pulled out manually after bulb growth is complete. Before storage, shallots are cleaned, the outer layer of peel is peeled off, and roots are trimmed before being wrapped into bunches and sun-dried for 5-14 days. For each kg planted, aim to produce 5–7 kg of shallots. This corresponds to 8 to 10 shallot per each shallot plant planted. Whenever the leaves start to fall and the bulb size exceeds 2.5 cm in diameter and ready for its harvest. Exempting water supply or weakening the root system can promote bulb maturity. After 50% and more of the stalk has fallen off, bulbs for storage could be harvested however, the bulbs must be cured and dried well before being stored. When 15–25 percent of the tops are down, bulbs meant for immediate consumption can be harvested. Thick necked shallot bulbs must be treated instantly since they will have spoiled during storage. Harvesting starts after 70–80percent of the leaflets have become yellow, typically happens up to 70 days after sowing in the lowlands and 80 to100 days after planting.(Askari-Khorasgani & Pessarakli, 2020; Horticulture :: Vegetables::Small onion, 2015; Tabor, 2018)

After the shallots have attained a size of a minimum of 0.5–0.6 cm, they are manually pulled. The outer layer is peeled off and also roots were also clipped, after which they are washed and tied into 1-kg bunches. Shallots are dried for 5–14 days in the field for dry bulb production and stored by a protective covering. When shallots are produced for their dried bulbs after the harvesting and processing are comparable to that of onions.

Persian shallots such as "Mooseer" in Iran include *Allium hirtifolium Boiss, Allium altissimum Regel,* and *Allium hollandicum R.M. Fritsch*. Grey shallots and Jersey shallots are two different species. The latter is divided into three categories: Mean-Long shallots, Long shallots, and Round shallots. Grey shallot *(Allium oschaninii* cv. Griselle) is grown primarily in France's south and east. Indonesia has reported cultivars of Ampenan, Cloja, Bima, Bima Kuning, Bauji, Balizo, Suminep, Bawang Lambung, Betawi Cipenos, and Hejakuning. (Fitriana & Susandarini, 2019; Marlin et al., 2021; Wenli et al., 2019)The Indian cultivars CO 1, CO 2, CO 3, CO 4, MDU 1, Agrifound Red and Arka ujjwal are grown entirely by bulblets. While the CO 5 variety generated seeds and spread by both bulblets and seeds (Askari-Khorasgani & Pessarakli, 2020; Horticulture :: Vegetables::Small onion, 2015).

2.4.3 Compositional characterization of shallot

Shallots have larger quantities of lipids and soluble solids, including sugars, than bulb onions with 16–33%, which along with sulphur components, makes shallot a key role in cooking preparation. The shallot is composed of 70–85 % carbohydrates, namely glucose, fructose, sucrose, fructan and oligosaccharides. Cell wall components consist of both soluble and insoluble polysaccharides such as lignin, hemicellulose, cellulose and pectin, which comprise 10–15 % of the carbohydrate fraction, as in the bulb onion.

The red pigmented shallot onion comprises anthocyanin (cyaniding and its derivatives), while the yellow colour is primarily due to the Quercetin derivatives. Generally, shallot onion composed of moisture (79.8 %), carbohydrate (16.8 %), protein (2.5 %), fat (0.1 %), fibre (0.7%), total ash (0.9 %), minerals (potassium (334 mg), phosphorous (60 mg), calcium (37 mg), Iron (1.2 mg), sodium (12 mg), and vitamins (thiamine, riboflavin, niacin, vitamin C with values of 0.06, 0.02, 0.2 and 8mg respectively) (Charoenchai et al., 2018; Fattorusso et al., 2002).

Flavonoids and related glycosides, sulphur components, and saponins are the most common chemical ingredients found in shallot onion. In French and Italian shallots, flavonols glucosides such as quercetin kaempferol, and isorhamnetin mono and diglucosides were discovered (Tan Sian Hui Abdullah et al., 2021). Sulphur antioxidant compounds such as diallyl disulphide, diallyl trisulphide, and allyl propyl disulphide are detected in it (Charoenchai L, C Luprasong, 2018).

The existence of flavonoids and polyphenol derivatives such as quercetin, quercetin 4-glucoside, quercetin 7,4-diglucoside, quercetin 3,4-diglucoside, and quercetin mono D-glucose was already observed in shallot extracts (Leelarungrayub et al., 2006). The overall flavonoid content of shallot was 20% higher than that of big onion, at 8%, with flavonoid concentrations highest in the outer layers and decreasing toward the middle portions of the bulb. Black shallot extract comprises seven bioactive constituents, notably quercetin 3,4-diglucoside, isorhamnetin 3,4-diglucoside, quercetin 3-glucoside, quercetin 4-glucoside, isorhamnetin 4-glucoside, quercetin aglycone, and isorhamnetin (Tran & Nguyen, 2020).

In 15 Indonesian shallot cultivars, a total of 64 volatile components were identified. Cycloartenol was the most prominent volatile among the volatiles. Thiols, mono-sulfides,

disulfides, trisulfides, thiophenes, and antioxidants are formed from shallot oil. Furthermore, methyl propyl trisulfide, dimethyl trisulfide, propyl propenyl disulfide, and 1-methylthiopropylethyl disulfide were the chief constituent of raw shallot oils (Lim, 2015). Several foods, including Persian shallot, can be supplemented with fructan dietary fibres without affecting the product's taste. The recommended daily intake of dietary fibres (30 g) might be exceeded with an additional 10–15 g of fructans daily (Setyadjit & Sukasih, 2015; Sukasih et al., 2018)

2.4.5 Preservation, Processing and storage of shallot cultivars

Curing is required after harvesting unless the crop is promptly dispatched to the market. The goal of curing is to dehydrate the shallot skin layers, providing an effective barrier against microbial invasion even while limiting the bulb's weight loss. So when the neck is firm, the exterior layers completely dry, and 3 to 5% of the initial bulb mass is lost during the curing process. Based on the cultivar, temperatures, and air humidity, bulb forming commodities such as onion, shallot and garlic have a prolonged storage life. Storing at room temperature implies adequate ventilation, while long-term storage needs refrigeration (0 to2°C). At 30 C, shallot quiescent bulbs can be stored for a longer period (Kamenetsky & Rabinowitch, 2017; Sukasih et al., 2018; M.-R. Wang et al., 2021; Yuvraj Khasherao, 2021).

Cell division, differentiation, elongation, and leaf growth are mostly promoted by storage at 15°C. Except when sets are used for propagation and/or bulbs directed at seed generation, pre-harvest preparation of sprouting inhibitors for the destruction of axillary buds, with the subsequent prolongation of storage life, is employed in Allium cultivars (Kamenetsky & Rabinowitch, 2016).

Fungal damage can occur if the bulbs are exposed to the sun directly, especially while they are wet. Fungal injuries can result is if bulbs were exposed to sunlight directly, particularly while they are moist. Induced warm air, vacuum chilling, cold rooms, and infrared radiation exposure are all types of artificial curing processes. In shallot bulbs, gamma irradiation is an efficient sprouting inhibitor. Studies have shown that irradiation causes discolouration, must not affect rotting and make onions extra vulnerable to aflatoxin growth (Swamy & Veere Gowda, 2006). Processing of shallot can be done in different methods such as minimally processed cut or paste products under freeze conditions to keep their freshness. Dehydration shallot flakes and powders. Also using preservation techniques, shallot can be converted into sauces and volatiles oils.

2.5 Exploitation of shallot waste into value-added products

Shallot onion production and processing generate a wide range of wastes that have never been investigated compositionally for practical benefits. The safety of these waste streams should be investigated and proved to be suitable for food and others applications. Although shallot onion produces a huge proportion of nutritionally solid waste, these are still not encouraged for use in the food material. Among all these wastes, only the big onion skin was already investigated from the perspective of value addition. Even though shallot onion produces a huge proportion of nutritionally solid waste, these are still not encouraged for use in the food material (Bhosale et al., 2022b).

The shallot onion waste stream consists of peel, petiole, stalk and flower during harvesting and processing. Among all these wastes, only the big onion skin was already investigated from the perspective of value addition. It contains high quantities of both soluble and insoluble polysaccharides, with 58 mg along with 10.6% minerals with increased

concentration of potassium and magnesium respectively (Kumar et al., 2022; Sukasih et al., 2018).

Onion waste leftovers contain rich phytochemicals that can be exploited in the pharmaceutical, culinary, and cosmetics sectors. Several researches have been conducted to determine the dietary fibre content, the sulphur content of the onion waste, as well as excellent source of minerals and fatty acids (Kumar et al., 2022; Michalak-Majewska et al., 2020). These processed and cured onion wastes can also be used as a substitute for food supplements, owing to rising customer response for food supplements and health ingredients. Compounds such as crude fibre, fructo oligosaccharides, flavonoids, and sulphur components can all be found in onion wastes (Sharma et al., 2016b).

The colour extracted from the outer layer can be used as a natural colorant. Flavonoids have been detected in the onion's dry skin. The skin of onion also is used to extract onion essence, often used as a flavour enhancer (Mourtzinos et al., 2018a).

2.5.1 Application and benefits of byproducts from onion waste

Several research has been conducted to determine the dietary fibre content and sulphur content of the top to bottom peels of onion waste, as well as a rich source of minerals and fatty acids. These processed and cured onion wastes can also be used as a substitute for synthetic food supplements, due to rising customer demand for food supplements and health-related ingredients.

Compounds such as crude fibre, fructooligosaccharides, flavonoids, and sulphur components can all be found in onion wastes (Nile et al., 2018). Flavonoids have been detected in the onion's dry skin. The skin of onion also is used to extract onion essence, often used as a flavour enhancer.

2.5.1.1 Flavonoids and phenolic components

Anthocyanins and quercetin and their derivatives, which contribute yellow to purple colour to onion skins, are the 2 key flavonoids distinct groups found in onion. Quercetin, quercetin diglucoside, quercetin aglycone, quercetin glucoside, and kaempferol are the predominant onion flavonoids. Flavonol glucosides have been derived from red onion waste streams and their functional characteristics are being determined to develop an alternative approach for medicinal benefits and source of bioactive compounds. Its main flavonol glucosides isolated from red onion waste streams were identified as quercetin-3, 4′-O-diglucoside, quercetin-3-O-glucoside (isoquercetin), quercetin-4′-O-glucoside (spiraeoside), isorhamnetin-4′-glucoside, quercetin glycoside and quercetin glycoside. As a result, onion waste streams and flavonol glycosides could be considered promising antioxidant and anti-inflammatory agents (Nile et al., 2018). Extraction of Flavonol glucosides was done using microwave and ultrasound-assisted extractions methods.

Onion skin extract has numerous health benefits, such as anti-carcinogenic, hypocholesterolemic and anti-asthmatic characteristics. Several studies have been conducted to evaluate onion skin as a rich source of dietary fibres, polyphenols as well as Fructooligosaccharides. It has been revealed that the formation of reactive oxygen species (ROS) relates to hyperglycaemia, coronary artery disease, hypertension, cardiomyopathy, and cardiac arrest. In terms of industrial waste, the outer scales contained the most flavonols, while the brown skin and inner scales contained the least. Flavonols were the key part of flavonoids in fleshy scales (Chadorshabi et al., 2022b).

Further research concluded that the skin of red onions contained most and the skin of yellow onions contained the least, with 20.22 and 10.69 mg QE/g dry weight of total

flavonoid, respectively. in the brown peel, flavonols indicated a fraction of the overall flavonoid and its derivatives (Benítez et al., 2011; Pareek et al., 2017). In whole onion, inner and outer scales, and top to bottom, main flavonols accounted for more than 80% of total flavonols. while white skin had the lowest antioxidant activity of 23.40%. Besides that, the Radical scavenging activity of red and yellow peel onions has been evaluated, so it was revealed that red peeled onions had better activity than yellow skin with 74.7% and 40.8%. The occurrence of flavonoids is a key factor that influences the potent antioxidant activity, with quercetin having a critical role.

The treatment doses of quercetin were ascertained to be approximately 250 and 500 mg threefold per day. Quercetin and its derivatives were generally accessible, in the form of a pill or tablets ranging in doses from 50mg to 500mg, like nutritional supplements. The dosage of quercetin was already recommended based on the health condition to be regarded but no recommended dose for quercetin has been recommended. Also, the anthocyanin and its derivatives, including the 3- malonylglucoside, 3- dimalonylglucoside, and 3,5-diglucoside derivatives of cyanidin, peonidin 3,5-diglucosides, and two 3-glycosylated derivatives of pelargonidin, were found in various cultivars of onion from Norway region (Kothari et al., 2020).

Brown peels had the highest concentration of total polyphenols and flavonoids in onion waste, whereas inner peels had the lowest. As either an outcome, from the outer towards the inner part of the onion flesh, total phenolic content and flavonoids dropped significantly. Protocatechuic acid was prevalent in the onion peels but not in any of the inside layers. Ferulic acid is been found in massive quantities both in dry and fleshy peels of the onion. Eventually, vanillic acids are known predominantly in the onion peels (Jiang et al., 2020; Simin et al., 2013).

As previously stated, industrial onion wastes are a valuable source of phytochemicals and natural antioxidants, and their use in food, which enhances their health-promoting characteristics, is a promising area. Besides that, if effective recovery and production process from commercial onion wastes, environmental impacts might be rectified (Sharma et al., 2016b).

2.5.1.2 Sulphur and its derivatives compounds

The inner scales had the highest Sulphur content, while brown skin had the lowest. Sulphur is found in a variety of compounds, including onion flavour precursors. Sulphur content in onion was only 19 percent of total Sulphur in onion and 15–35 percent in onion wastes, with inner scales and brown skin having higher Sulphur content. Trans (+)-S-1-propenyl-L-cysteine sulfoxide (PECSO), which is commonly found at the largest percentage as well as gives rise to the components liable for the lachrymatory impact. Also, (+)-S-methyl-Lcysteine sulfoxide (MCSO) and (+)-S propyl-L-cysteine sulfoxide (PCSO) that is found in lesser concentrations, seem to be three of the naturally occurring sulphur compounds found in onions Whenever an onion is fresh, these compounds are crucial.

Sulphur containing derivatives sourced from onion waste streams can hinder blood clotting but can boost heart health. Moreover, such volatile compounds do have the ability to significantly induce the free radical scavenging, induction of apoptosis, as well as inflammation processes in humans.

Onions contain sulphur compounds with low molecular weight which also constrain polyphenolic compounds (PPO). Thiol (SH) classes have been effective PPO enzyme inhibitors which also help in limiting the reactive oxygen species levels by inhibiting nicotinamide adenine dinucleotide phosphate oxidase, which is an oxygen radicals source.

Organosulfur compounds also help to keep antioxidant activity like glutathione s-transferase from deteriorating.

Other studies have shown that organosulfur derivatives help prevent heart conditions such as hypertension by intervening like an angiotensin-converting enzyme inhibitor, leading to high blood vessel distention as well as lowered reaction, also and prevent thromboembolism by hindering cyclooxygenase HMG-CoA and blood clotting (Bede & Zaixiang, 2020). The enzyme is responsible for the browning effect that occurs even before or after the handling of fruits and vegetables. The browning reaction alters the product's organoleptic, phenolic, and appearance attributes.

2.5.1.3 Fructooligosaccharides (FOS)

Non-structural carbohydrates (NSC) such as glucose, fructose, sucrose, and fructooligosaccharide may account for up to 65% or more of the dry mass and it varies in varieties. Kestose, nystose, and fructofuranosyl nystose are the main FOS observed in onion varieties. The therapeutic effects of this carbohydrate intake were broadly reported previously, which has their prebiotic influence. In terms of FOS, the existence of these compounds throughout onion waste streams with pharma, food products and skincare applications has been affirmed.

Even though FOS is not digested by human gastrointestinal enzymes, that has fascinating prebiotic features as immunomodulatory and could be regarded as a source of dietary fibre. As a byproduct, they can be used in food manufacturing as a prebiotic additive ingredient (Beatriz Santiago 2019).

2.5.1.4 Dietary fibre

The majority of the dry matter in onion skin was fibrous, with 50 percent defining the insoluble fibre. minerals, sugars, protein, soluble fibre, and fat were the other components in decreasing the order of composition. Also, it was observed that insoluble fibre was the most plentiful fraction throughout the onion peel with 59.3–66.6 % of the dry matter, which varies based on the onion diversity. Onion waste streams comprise inner, outer, and peel parts, and hence contain more dietary fibre.

Both soluble and insoluble fibre and their soluble/insoluble fibre ratio are higher than that of other vegetables, which are associated with various physiological and metabolic influences. The fleshy layer had the minimum dry matter content, and the brown skin layer had the maximum DM content. Brown skin contains the most dietary fibre, followed by outer to inner peel layers. it was made up of a mixture of inner, outer, and peel portions and thereby, increased levels of dietary fibre can be related to the enhanced input of skin to the upper layer.

Non-structural carbohydrates may account for up to 65% or above of the NSC. Dietary fibre is remarkably essential for public health. This helps in the prevention and control of water retention as well as the emergence of hard bowel movements, which also can give rise to bloating.

2.5.1.5 Vitamins and minerals

Vitamins found primarily in *Allium cepa* are B complexes such as B1, B2, B6, and also A (669.63mg), C (3.48mg), and E (277.12mg), as well as biotin, nicotinic acid (Pareek et al., 2017)varies in onion varieties. The mineral proportion in onion waste streams is determined by minerals found in the soil where the onion is produced.

Top-bottom layers always had the highest concentrations of magnesium, iron, zinc, and manganese, while inner scales have the increased concentrations of potassium and selenium. Likewise, brown skin has a high calcium concentration. The outer scale has the highest concentration of calcium, up to 3.05 %, followed by the top-bottom contains a concentration of 2.08 %.

Chromium is only found in bottom waste, nickel is only located on the outer scale, as well as manganese content is excessive in bottom waste. This waste stream contains several metal complexes known for catalytic properties, including manganese, iron, nickel, titanium, and chromium, and it can be explored as a functional food to enhance digestion mechanisms and metabolism in the body.

2.5.2 Edible and non-edible applications of onion and shallot varieties

Onions' health benefits are frequently underappreciated. Throughout Europe, the white, yellow flesh onion is primarily used in cuisine practise. Red or purple onions, on either hand, are becoming widely attractive in meals. Due to their pungent flavour and high stability, these onions are prominent throughout food preparation, processed food, and fine dining. due to its popularity in cuisine practises, there is still a demand for the onion to be supplied in an even more feasible way, creating a demand for industries to generate prepared onions, including pre-chopped as well as peeled and dehydrated forms (Shea et al., 2012).

During harvesting and processing, a large amount of waste is generated, which primarily includes onion peel, stalk, petiole, flower and roots, and undersized or deformed and infected onions. Unfortunately, these streams are not acceptable for the regular vegetable dumping sites. Its strong aromatic characteristic makes, unsuitable for animal feed, and its phytopathogenic microbes prevent it from being used as a biofertilizer.

As a result, scientists are working through its promising use as a raw resource for both non-edible and edible implementations, based on the composition as well as the functionality of complex constituents. Table 2.2. explains the applicability of onion and shallot waste streams in different fields.

2.6 Lignocellulose

2.6.1 Structure of lignocellulose

The most abundant and available polymer is lignocellulose waste, which is composed mainly of cellulose, hemicellulose, and lignin, with a minimal quantity of crude protein, soluble pectin, ash, and extractives. The occurrence of these constituents in wide variation amounts within a hetero matrix in a diverse selection of lignocellulose structures with varying and relative contents. Hemicellulose and lignin are found in natural fibres. The cell wall has quite a compact structure. The bond formation of cellulose, hemicellulose and lignin varies a lot. An H- bond is the principal way to the linkage between cellulose, hemicellulose, and lignin compounds. Aside from the H-bond, also there is a chemical linkage between lignin and hemicellulose, that results in lignin, once detached from pure lignocellulose biomass, very often possessing a limited carbohydrate.

Chemical bonds between lignin and hemicellulose mainly refer to chemical bonds between galactose residues, arabinose residues on the side chains of hemicellulose and lignin, and carbs, even though revealed through research on separated lignin-carbohydrate complexes. In 4:3:3 ratios, cellulose, hemicellulose, and lignin make up the majority of cell walls. Sources such as hardwood, softwood, and herbs have different ratios (H. Das & Singh, 2004; Hakeem et al., 2015; Puri et al., 2020).

Table 2.2 Application in non-edible and edible formulation from onion waste streams

Portions	Products	Applications	Benefits	Reference
		Non-edible		
Onion peel	Fluorescent carbon dots	Biosensor & imaging agent	Stabilizer	(Bandi et al., 2016)
	Carbon Nanoparticles	Energy storage devices		(Jung et al., 2020)
	Supercapacitor	Porous carbon		(Mehare et al., 2019)
	Fungicide	Powder	Stimulant	(Shea et al., 2012)
	Homespun	Bio-piezoelectric Nanogenerator	Biomedical	
Onion Peel, stalk	Active film	Quercetin	Packaging	(Sholichah et al., 2018)
	Film	Extract	Antioxidant,	
Onion seed	Packaging film	Extract	Anti-browning	(Chadorshabi et al., 2022b)
Onion peel	Active film	Extract	Antioxidant	(Sholichah et al., 2018)

	Biochar	Whole	Carbon	(Jaya et al., 2021; Nihayah et al., 2020; Quilliam et al., 2020)
Shallot stalk	Packaging film	Extract	Antioxidant	(Thivya et al., 2021b)
Onion Peel	Silver nanoparticle	Extract	Biosensor	(Tan Sian Hui Abdullah et al., 2021)
	Natural dye	Extract	Colourant	(Sarwono et al., 2019)
	Edible applications			
Onion peel	Yogurt	Natural colorant	Additive, antioxidants	(Mourtzinos et al., 2018a)
	Biosugar	Glucose	Antioxidant	(Choi et al., 2015a)
	Filler	Powder	Antioxidant	(Setyadjit & Sukasih, 2015; Sukasih et al., 2018)
Onion Stalk	Food	Extract	Anti-browning	(Phaiphan et al., 2019)
Onion Peel	Fortified bread	Extract	Fibre enriched, Bioactive components	(Bedrníček et al., 2019; Santiago et al., 2020)

	Natural dye	Extract	Colourant	(Sarwono et al., 2019)
Shallot Stalk, peel and petiole	Cookies	Flour	Supplementation	(Bhosale et al., 2022a)
Onion Peel	Pasta	Flour	Antioxidant	(Michalak-Majewska et al., 2020)
	Candy and Jelly Glaze	Extract	Natural Colorant	(Chadorshabi et al., 2022b; Sarwono et al., 2019)
	Patties	Extract	Antioxidant, preservative	(Bedrníček et al., 2019)
	Supplements	Extract	Encapsulation, Oxidative stress marker	(Milea et al., 2020; Piechowiak & Balawejder, 2019)
	Nutraceutical	Extract	Prebiotic	(Shery M Varghese, Aruna Nair U K, 2021)

Flavour enchanter	Extract	Natural additive	(Benítez et al., 2011)
Emulsifier	Extract	Natural additive	(Dahlawi et al., 2020)
Preservative		Antimicrobial agent	(Hamza, 2014)
Antioxidant		Radical scavenger	(Khiari et al., 2009)

Lignin has a structural component that is made up of a cross-linked polymer matrix of phenolic monomers, notably p-coumaryl alcohol, coniferyl alcohol, and sinapyl alcohol. Plants with a supporting function and mechanical action typically have a high lignin content in their cell walls. Woody plants have a lignin content of 27–32 percent, while herbaceous plants have a lignin content of 14–25 percent. Lignin plays an important role in blocking the destruction of cell membranes caused by microbial attacks, and the separation of biowaste. The presence of lignin determines the strength of lignocellulose structures.

Softwood, for example, has quite a higher lignin content than other biomass, which increases its resistance to the cellulose separation process (Ghaemi et al., 2019). As a result, it is critical to remove lignin to reach the cellulose fibre separately by increasing the digestibility of the biomass.

Hemicellulose is a mixed polymer made up of diverse C5 and C6 sugars like xylose, arabinose, mannose, and galactose. Hemicellulose is the second most prevalent carbohydrate component, contributing to 25–35 % of the wood material. Following, Cellulose is the primary structural polysaccharide of the main plant cell wall, accounting for 30–50% of lignocellulose's dry weight and consisting of linear chains of (14) linked d-glucose units (H. Chen, 2014). Extraction of the aforementioned components is pre-treated to separate into individual components for further modification was clarified in the next subsection.

2.6.2 Isolation methods for the separation of lignocellulose components

Throughout, each of the common pre-treatment approaches behaves differently to break down the complex structure of the lignocellulose waste streams. Even though some of the steps outlined had already efficaciously transformed from the part of the strategy to the commercial phase, gaps remain, including the source of ecologically hazardous waste in

some circumstances (Hassan 2018, M. Duque-Acevedo 2020). The major pre-treatments applied for the isolation of lignocellulose are given in the table 2.3.

Table 2.3 Diverse methods involved in the separation of lignocellulose components

Pre-treatments	Advantages and disadvantages	Reference
Milling process	• Particle size reduction • Easy handling • Loss of crystallinity • Great energy depletion	(Chang & Li, 2019; Cheng et al., 2017; Gontard et al., 2018a; Hassan et al., 2018; Ilyas et al., 2018; Li et al., 2016; Mansour et al., 1989; Thomsen et al., 2008)
Extrusion process	• Breakdown of the polymeric chain • Defibrillation	
Chemical process	• Isolation of each component from the lignocellulose • Chemicals are toxic and corrosive • Formation of reactive components	
Solvent extraction process	• Isolation of cellulose, hemicellulose, lignin, fatty acids and other pigments • Formation of metabolites • Costly and corrosive	
Oxidation process	• Isolation of hemicellulose and lignin • Partly breakdown of the cellulosic chain • Highly expensive	
Hot water process	• Separation of soluble pectin, pigments, lignin and hemicellulose • High usage of water • High energy intake	
Ionic process	• Partial removal of hemicellulose and lignin • Destruction of crystal formation in cellulose	

	• Highly toxic and formation of reactive species • Costly	
Steam Explosion	• Particle size reduction • Easy isolation of cellulose • Low cost and energy requirements	
Deep eutectic reagents	• Greener solvents • Effective removal of hemicellulose and lignin • Less stability in high temperatures and	
Supercritical process	• Reduced crystallinity • High cost and energy • Greener solvent and reagents	
Enzymatic process	• Separation of targeted components • No environmental issues • Long process • Low output	
Microwave process	• Superior to traditional heating techniques • Convert agricultural wastes into higher-value products. • Highly expensive	
Ultrasound	• Cellbreakdown and destruction, fragmentation • Effective greener process • Oxidising radicals are generated • Reduced crystallinity • Expensive and energy consumption	
Radiation process	• Lignin alteration and cellulose crystalline region degradation	

	• Effective γ-irradiation on the bioconversion efficiency of microcrystalline cellulose • Lower the thermal stability • High cost and energy consumption	
Electron-beam irradiation	• Disrupt the structure of lignin, cellulose and hemicellulose • Effective in combination with further pre-treatments • Lower the degree of polymerisation • Low crystallinity • Costly	
Pulsed electric field	• Omission of lignin from lignocellulose waste • Structural deformation of cellulose • Non-thermal technology • High cost	
High hydrostatic pressure	• Particle size reduction • Inhibit the enzymatic reaction • Affect the polymeric bonding • Destruction of crystalline forms • Structural modification • Highly expensive	
High-pressure homogenization	• Cell wall destruction and extraction of the lignin, hemicellulose and cellulose • Increased extraction of sugar molecules • Size reduction • Depolymerisation • Crystallinity reduction • Expensive	

2.6.3 Derivatives of cellulose

The manufacture of cellulose in a variety of colloidal forms has recently improved thanks to the development of novel sources and separation technologies. Although cellulose is frequently utilised as a functional component in food, the link between the colloidal phases of cellulose is not well understood (Krawczyk et al., 2009). Due to its unique structure and distinguishing qualities, such as bio-compatibility, renewability, non-toxicity, and environmental friendliness, cellulose has been widely used in various domains, including food additives, paper manufacturing, bio-materials, and pharmaceuticals in latest years.

Physical, chemical, and biological approaches are used to modify cellulose, with chemical modification being the most diverse and significant, including etherification, esterification, crosslinking, and surface polymerization. A combination of acid hydrolysis and mechanical treatments are used on natural cellulose sources, cellulose of diverse forms and sizes, including microcrystalline cellulose, crystalline nano cellulose, and nanofibrillated cellulose, can be generated (Seddiqi et al., 2021). The type and degree of substitution, as well as the crosslinking pattern throughout the polymer chain, define the attributes of cellulose derivatives was showed in fig 2.4. Because of cellulose's low solubility in organic solvents and severe steric hindrance due to the stiff and bulky cellulose main chain, the synthesis of cellulose derivatives is restricted. On the cellulose chain, the -OH group is the most targeted active group.

The native cellulose may be changed by the attachment of functional groups; it is possible to create water-soluble cellulose derivatives. By substituting the surface hydroxyl groups with methyl, carboxymethyl, and hydroxypropyl methyl groups via etherification processes, methylcellulose (MC), carboxymethyl cellulose (CMC), and hydroxypropyl methyl cellulose (HPMC) and carboxymethyl hydroxyethyl cellulose (CMHEC),can be

made. Chemical alterations can result in unique features in cellulose, allowing it to be used in a larger range of applications. Many key properties of cellulose, like size, charge, crystallinity, and wettability, may be characterised as colloidal states.

Recent studies have mostly concentrated on the manufacture of cellulose with desired colloidal states utilising various isolation approaches. Under regulated circumstances, for example, cellulose with varying diameters, surface charges, and crystalline structures may be generated.

Chemical modification via functional groups or physical complexation with other polymers might change the properties of cellulose. Cellulosic materials are recognised as potential functional ingredients or dietary fibres for food applications since they are "generally recognised as safe" (GRAS) substances. Based on its dimensions, surface charge, crystallinity, and wettability, cellulose has the potential to demonstrate exceptional features including emulsibility, foamability, film-forming ability, encapsulation, fat replacement, and so on(X. He et al., 2021).

The dimensions of cellulose are related to its shape. MCC is rod-like and has a length of 50-200μm, whereas NCC and NFC exhibit needle/whisker-like and fibril-shaped characteristics, respectively. Mineral acid hydrolysis is the most practical method for controlling cellulose dimension. MCC is microscale in length because it is partially depolymerised by dilute mineral acid hydrolysis, whereas NCC is nanoscale in dimension because it is generated from a high concentration of acid. The extraction methods for each type of cellulose can have a direct impact on the final morphology, and the treatment method is chosen depending on the structure and quality of the extracted cellulose (K. Liu et al., 2021).

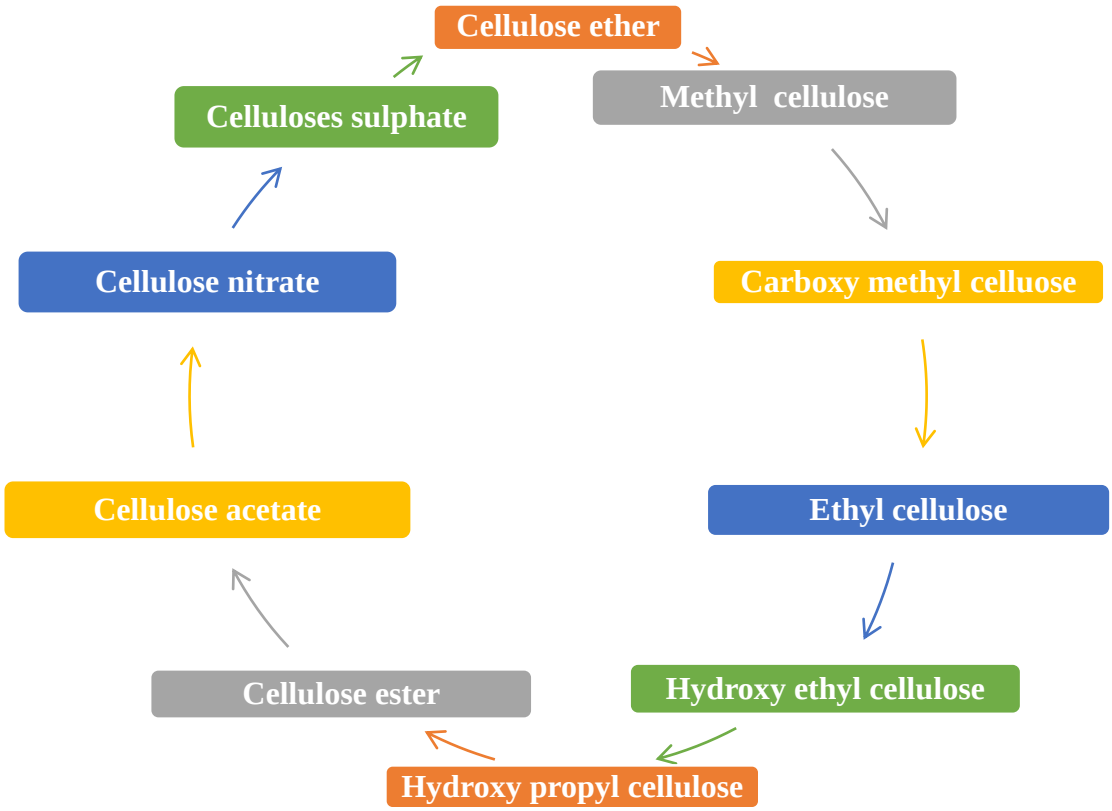


Figure 2.4 Categories of cellulose derivatives produced from cellulose sources.

2.7 Perspectives on microcrystalline cellulose and its applications

2.7.1 Production scenario of MCC

By 2025, the Microcrystalline Cellulose Market is expected to grow at a CAGR of 6.3 percent, reaching 1540 million US dollars. Microcrystalline cellulose (MCC) is a white porous powder that is odourless, tasteless, and generated from wood pulp or refined cotton. MCC is frequently utilised in pharmaceutical, food, and other industries due to its wide variety of chemical, technical, and economic benefits. According to statistics, the top three manufacturers, FMC, JRS, and Mingtai, held 53.8 percent of the population market share in 2017. Demand for microcrystalline cellulose is primarily concentrated in the United States, Europe, South America, China, Japan, and India. North American and Asia-Pacific-led industries account for half of the market.

The pharmaceutical sector in India is predicted to reach USD 65 billion by 2024, according to Invest India, and is now valued at USD 41 billion (*Microcrystalline Cellulose Market | 2022 - 27 | Industry Share, Size, Report*, n.d.). South Korea boasts one of the best beauty markets in the world. Novel skincare concepts and advancements are frequently on exhibit including sophisticated components and visually appealing designs. During the projected timeframe, 2020-2025, the South Korean cosmetics products, market was valued at USD 1,578.4 m in 2019, and it is predicted to exceed USD 1952.05 million by 2025.

2.7.2 Manufacturing of microcrystalline cellulose

Agro-industrial waste streams are rich in valuable ingredients such as micronutrients, pigments, polyphenols, and complex carbohydrates, but they are neglected. More research into using bioactive components from agro-industrial raw material for the production as additives or nutrients is gaining attraction, as the commercial exploitation of these waste streams can enhance economic value, and the recuperation of those phytochemical compounds is often cost-effective.

Most research has explored the utilisation of by-products, with strong attention on the recovery and exploitation of polyphenol compounds. As a result, independent of the insoluble part, water or solvent extraction was performed to obtain a fraction of the polyphenols. Indeed, those worthless insoluble residues contain some useful substances, such as conjugated phenolic content, proteins, and, especially notably, insoluble fibre. Valorising this insoluble fibre concentrate can contribute to a long-term food recovery chain, both in terms of environmental issues and the economy, as well as health and wellbeing.

For many decades, the papermaking industries, textile products and derivative products, or even MCC production were the primary uses of cellulose. Hardwood, softwood, agricultural and industrial wastes, and grasses are all examples of lignocellulosic biomass. Nevertheless, obtaining this biopolymer and meeting input materials demand is therefore

needed to enhance renewables such as lignocellulosic biomass. Moreover, packaging components are formed primarily of fossil-based and lignocellulosic materials that can be used to minimise or substitute materials generated by non-renewable sources. The finding of distinct cellulose scales, on either side, has expanded the potential of cellulose derivatives in the pharmacological, food product development, textile, water systems, healthcare, power, and paper industries(Nsor-Atindana et al., 2017, 2020).

Cellulose is the most abundant natural polysaccharide in the universe and was found in all plant cells. It is a linear macromolecule comprised of D-glucose molecules bonded together by a (1–4) glycosidic bond to generate cellobiose units. Lignocellulosic wastes are a major source of cellulose; their percentage ranges from 40 to 78 percent. Additional, materials of interest in the production of cellulose derivatives from Lignocellulose wastes also are considered to be low-cost raw materials for MCC synthesis(Ventura-Cruz & Tecante, 2021). The quantities and qualities of cellulose are defined by the isolation methods, the source, and the lifecycle of the natural origin. Such sources are often made up of cellulose, hemicellulose, lignin, extractives, and microelements. The spirally oriented cellulose in their cell walls acts as reinforcements in the soft hemicellulose and lignin matrix. Pure cellulose is produced by effectively removing hemicellulose, lignin, and other contaminants (Beroual, Boumaza, et al., 2021; Beroual, Trache, et al., 2021; Tarchoun et al., 2019). Cellulose is tasteless, hydrophobic, odourless, chiral, renewable, and biodegradable, as is widely known. It is hydrophobic but in the majority of organic solvents. Hydroxyl groups are primarily responsible for cellulose's stability and crystallinity

Microcrystalline cellulose is synthesized by acid treatment of fibrous cellulose into redispersible gels or clusters of crystalline cellulose. There are crystalline and amorphous zones in microcrystalline cellulose, which is purified and partially depolymerized cellulose. The crystalline regions are known as cellulose crystallites, and they are synthesized via

Vander Waals force and H- bonding among cellulose strands. Those crystallites are around the same size as cellulose fibrils in regards to diameter. When acid hydrolysis is used, its amorphous phase is effectively hydrolyzed. Synthesis yields MCC, which are shorter as well as more crystalline fragments. As an outcome, the cellulose chain's degree of polymerization is lowered while almost minimal weight is lost (. MCC was already produced commercially for decades, as well as its abundance, cheapness, low density, and high mechanical resistance have prompted interest as a composite reinforcer.

For powdered grades, this material is either dried to a purified, fine particle form and processed with a water-soluble copolymer, including such cellulose gums, that acts as a barrier dispersion, allowing the copolymer to be dispersed in water with high shear. The formulation will have reconstituted to produce microcrystalline cellulose in a colloidal form. As compared to other soluble food hydrocolloids, those colloidal dispersions are peculiar (Krawczyk et al., 2009).

2.7.3 General Stages of microcrystalline cellulose production

The major stages of MCC isolation are i) extraction and purification of α-cellulose and ii) hydrolysis of extracted cellulose to crystalline cellulose. Several stages are required for the extraction of pure alpha-cellulose depending upon the source of raw material were given in fig 2.5.

The first process is to grind the fibres to lessen the size of the particles so the chemical treatments may react with the cell wall. It increases the contact area and boosts the effects of further treatments. The milling process is an important step in obtaining a powder with consistent particle size and enhancing the fibres' liquid swelling capacity. To eliminate soluble contaminants, the swelled fibres are filtered and washed. Depending upon the

source, if this step is not enough, the washed fibres are extracted using combinations of solvents to remove wax, dyes, and sugars.

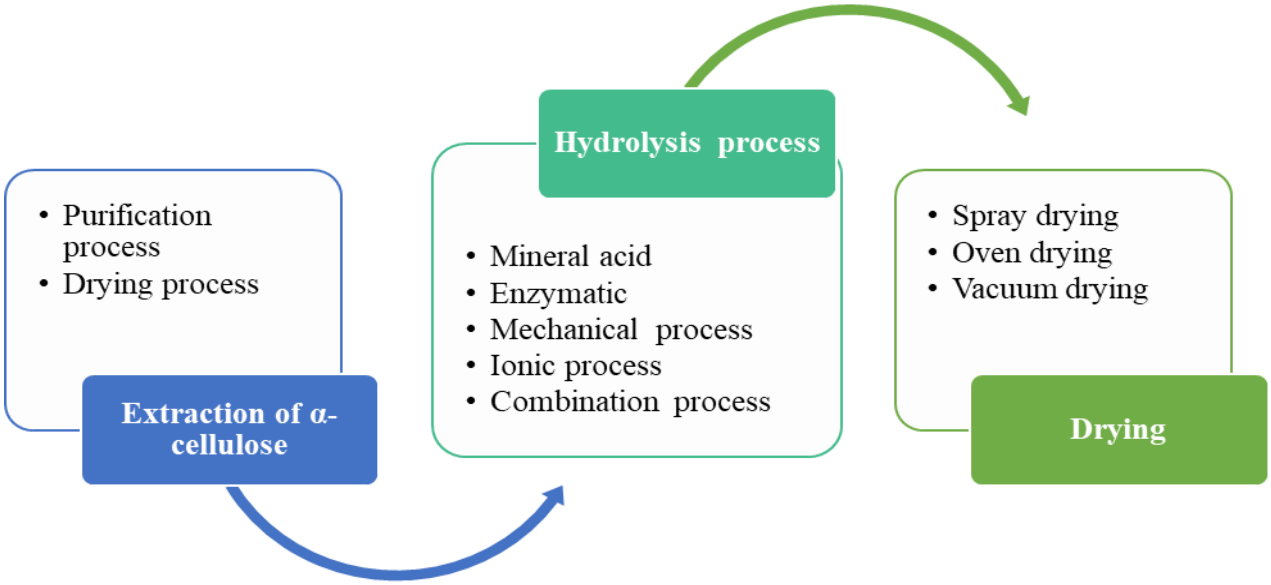


Figure 2.5 General layout of the isolation of the microcrystalline cellulose from alpha-cellulose

The fibres are again reacted with either an alkaline solution of sodium hydroxide or potassium hydroxide in the second phase. Hemicellulose is partially hydrolysed and remaining waxes, lignin, pectin, and native fats are separated. The lignin in the lignocellulosic fibres is eliminated in the third process, known as delignification or bleaching.

This procedure is required to remove residual hemicellulose, but primarily lignin, to obtain crystalline cellulose. In the prior literature, two methods are frequently mentioned. Former, the fibres are bleached in acidic settings with chlorinated chemicals, whereas in the latter, the fibres are bleached in alkaline pH of hydrogen peroxide.

Following the third process, various techniques to isolate MCC from cellulose fibre were described in the literature such as acid hydrolysis is considered to be the most effective in eliminating lignin. The most critical terms to regulate during hydrolysis are acids and dosage, hydrolysis duration, and temperature.

Whenever the necessary level-off degree of polymerization (LODP) is reached, the hydrolysis process is terminated. The required level of DP varies depending on the raw material and is commonly found in the 180–350 range, as in the cases of 180–210 for hardwood pulps, 210–250 for softwood pulps, and 182 for agro-industrial biomass.

The remnant matters were neutralised and thoroughly washed to remove contaminants after acid treatment (Nsor-Atindana et al., 2017; F. Ohwoavworhua et al., 2009). MCC was made from different sources using a range of techniques, and the traits and efficacy of a novel MCC were revealed to be extensively impacted by the cellulose fibre and process circumstances.

Acid, alkaline and enzymatic processes, steam explosion, and mechanical deconstruction are some of the commercially patented MCC manufacturing operations. As a result, several MCC grades with diverse physicochemical, thermal, and functional properties were commercially available. The table 2.4. gives an overview of prior studies on pre-treatments, extraction layout, and quantification of crystal cellulose extracted from agro-industrial lignocellulose waste stream.

Table 2.4. Source, pre-treatments and crystallinity index of extracted microcrystalline cellulose from agro-industrial waste streams through prior literatures.

Source	Extraction process	Crystallinity (%)	Reference
Wheat straw	During alkali treatment, one coarse milled (CM) and two passes ball milling wheat straw materials were treated at various sodium hydroxide concentrations (1–10 %), and the lignocellulosic ingredients and crystalline-structural alterations were find out in quantity and quality-wise evaluated.	61.0%	(Gao et al., 2017)
Water guard bark	Cellulose was isolated from the fruit of Lageriana siceraria and modified by controlled acid micronisation to generate microcrystalline cellulose (LS-MCC) using 2.5N hydrochloric acid, with Avicel pH 101 providing as a control.	44 %	Achor M 2014
Date seeds	A three-step technique was used to effectively isolate MCC from date seeds through dewaxing, bleaching and mineral acid hydrolysis. The dried cellulose from date seeds was hydrolysed for 45 minutes at 105 °C with 150 mL of 2.5N HCl (1:11 w/v ratio) under continual agitation.	70%	(Galiwango et al., 2019) Nedal Y. Abu-Thabit
Bagasse, rice straw, and cotton stalks	Bleached cotton stalks fibre was produced in the lab by milling cotton stalks using NaOH (15 percent per weight) at 150 °C for two hours and afterwards bleaching the pulp with the sodium chlorite bleaching process. During reflux for 45 minutes, bleached samples were hydrolyzed with 2N hydrochloric or 2N sulfuric	77.64, 75 & 71%	(El-Sakhawy & Hassan, 2007)

	acid (1:10 w/v). The hydrolyzed pulps have been thoroughly rinsed with distilled water and freeze-dried.		
Bamboo chips	Using sodium hypochlorite, the dried cooked pulp was bleached to remove lignin and hemicellulose. The procedure was carried out for 1 hour at 70–80 degrees Celsius. It was then dried in an oven The bleached fibres were treated with 8% (w/v) sodium hydroxide (NaOH) (1:50 w/v). For 30 minutes, the reaction was carried out at room temperature. The obtained pulp was filtrated and washed before being dried in an oven at 60 degrees Celsius for 4h. The treated pulp was filtrated and washed until being dried in an oven at 60 degrees Celsius for 4h. 2.5 mol/L HCl was applied to the alkali-treated fibre. The hydrolysis process was carried out at 85 C for 30 minutes, with a solid-to-liquor ratio of 1:30 (g/mL1).	82.6%	(Rasheed et al., 2020)
Roselle stems	Over 1h at 70–80°C, a 10 % (w/v) sodium hypochlorite solution was used to bleach the material. A fibre to sodium chlorite ratio (1:60 w/v) was added and the acidified to pH 4 by acetic acid. Afterwards, filtered and rinsed with distilled water until it acquires a white-yellowish colour. Then, dried overnight in a 60°C oven. Dried samples were hydrolysed with 2.5M HCl at 85°C for 30 min (1:30). The hydrolysis was carried out with constant agitation speed. At room temp, the hydrolysed products were chilled. Thereafter, distilled water was filtered and washed until pH 7 was acquired. The extracted MCC were dried to constant weight in a vacuum oven for 5 hours.	78%	(Kia et al., 2017)

Pomelo peel	Pomelo peel material was processed for 4 hours at 80 °C with a 4% NaOH containing 0.9% hydrogen peroxide. To get crude cellulose from the pomelo peel, the solution was filtered and rinsed with distilled water until it reached neutral pH. Then subjected to a fast purification process at 100 °C for 15 minutes with an 80% acetic acid and 68% nitric acid blend to remove hemicellulose and lignin linked to cellulose. The extracted cellulose was freeze-dried. MCC was made using the hydrochloric acid hydrolysis technique.1g of cellulose was mixed with 20ml of a 6 percent (w/w) hydrochloric acid aqueous solution in a 90 °C water bath for 100 minutes. The solution was then filtered and rinsed with distilled water. After that, the extracted MCC was freeze-dried.	40.53%	(Y. Liu et al., 2018)
Oolong tea waste	The results revealed that the time and temperature of hydrolysis, the concentration of HCl, and the acid-to-material ratio all had a significant impact on the DP and yield. Under ideal conditions (1:20, HCl of 1.5 M 65 °C, 90 min)	81%	(T. Zhao et al., 2018)
Fodder grass (Setaria glauca	The samples were rinsed with water until they reached neutral pH, then bleached for 40 minutes at 100 °C using a 1:1 solution of NaClO and hydrogen peroxide. The samples were rewashed with water until they reached a neutral pH, revealing the -cellulose fibres. 10 g of -cellulose was acid hydrolyzed with conc. HCl (2.5 N) at 100 C for 30 minutes to extract MCC.	80%	(Baruah et al., 2020)
Soybean hulls	MCC was produced from SH by a two-stage extrusion process in which the SH was first extruded with conc. NaOH followed with H2SO4 in the second step. MCC synthesized from SH by reactive extrusion was made up of short and rod-	70%	(Merci et al., 2015)

	shaped fibres. Reactive extrusion is an innovative and successful approach for producing MCC from lignocellulosic residues that is simpler and less damaging than traditional processes.		
Corncob	The method involved in lignin removal of the raw material with 3.5% HNO_3 and sodium sulphite(0.01%) at 90°C for 2h following alkali hydrolysis with sodium hydroxide extraction (2%) and sodium sulphite (2%) at 50°C for 1h and NaOH (75%)at 80°C for 60min. Bleaching was done at 40°C for 1.5 hours with NaClO (3.5%). The corn cob cellulose sample was hydrolyzed at room temperature with HCl (2N) under reflux for 15 min (1:10). The extracted MCC was extensively rinsed with chilled distilled water till it was neutral pH and then air-dried.	78%	(Azubuike & Okhamafe, 2012; Shao et al., 2020)
Rice husk	Mechanical and chemical pre-treatment is used to generate microcrystalline cellulose (MCC). A 10% NaOH treatment followed by varying concentrations of sodium chloride (0.5, 0.7, and 1%) treatments led to a significant weight reduction. The sample was mixed with the acidic media with strong stirring at 650 rpm for 20 minutes at 105 C. It turns dull coloured microcrystalline cellulose into milk-white, hydrogen peroxide bleaching was used.	64.74%	(Bhandari, Roy Maulik, et al., 2020b)
Kans grass	MCC was extracted from kans grass using an eco-friendly and durable process that combined with delignification, bleaching, and acid treatment. Samples were chemically treated via immersing in a NaOH solution ((1:20) for 14 h. They were autoclaved for 45 min at 210 °C with a pressure of 20 psi. The bleaching process was then carried out using a standard solution with a specified volume of H_20_2	83%	(Baruah et al., 2020)

	and alkaline solution. The cellulose (white coloured)was purified by 95% ethanol and distilled water until it attain pH neutral. Dried for 8 h in a vacuum oven at 70–80 °C to a constant mass. It was recovered and acid hydrolysed to generate MCC.		
Waste cotton	The catalytic digestion of phosphotungstic acid was used to develop an innovative and environmentally friendly approach for the manufacture of microcrystalline cellulose (MCC) from waste cotton. The effects of phosphotungstic acid concentration, reaction temperature, response time, and solid to liquid ratio on degradation were evaluated. Its follows was considered to be the best process conditions with HPW (3.47 mmol/L, 1:40 w/v) at 140 °C r for 6 hours	85.2%	(Sheng et al., 2018)
Rice straw, banana waste	After chemical treatment, the cellulose materials were obtained in the first step from rice straw and banana plant waste, mostly using alkali-acid or acid-alkali pulping, followed by the NaClO bleaching process. after that, the cellulose was enzymatically treated to produce MCC. To create MCC, the bleached fibres were treated with enzymes. Trichoderma reesei cellulase were used. The fibres were mixed in a sodium acetate buffer (pH 4.7–5.0 in 100mL) with cellulase (2.0 mL) at a 10% (w/v) initial sample concentration. The enzymatic treatment took place at 50 C for 1 and 2 hours at 75 rpm. Filtration was completed, and the resultant MCC was rinsed in hot water and then dried.	88.8 and 96.3 %	(Ibrahim et al., 2013)

Cotton linters	MCC was isolated from cotton wool with the help of microwave-assisted pre-treatment. On the yield, the effects of conc. H2SO4, time, and temperature were examined. The best results were obtained at acid content was 55%, for 1 minute and at 30℃. Furthermore, the structural structure of cellulose I changed to cellulose II. Without mercerization or ionic liquid treatments, cellulose II was discovered.	53.4%	(Kusumattaqiin & Chonkaew, 2015)
Sorghum stalk	The stalk was cut up into tiny fragments and crushed. These obtained fibres were ground and rinsed numerous times with water and oven-dried for 5 days. The MCC obtained from the stalk of Sorghum obtained by delignification using NaOH followed by bleaching (NaClO) and acid (2.5N HCl) or enzymatic hydrolysis was examined for its physical and chemical and pelletization properties in comparison with commercial microcrystalline cellulose grade.	75.19 & 89.43%	(F. O. Ohwoavworhua & Adelakun, 2010; Ren et al., 2019)
Sago seed shell	After bleaching and oven-dry sample (2 g), holocellulose was treated with 17.5 percent NaOH (25 ml) in three additions for roughly 45 minutes. The solution was filtered before being rinsed in distilled water and 10% acetic acid. The residue was rinsed until it reached a neutral pH, then dried in the oven until it reached a constant weight. The a-cellulose was hydrolyzed for 15 minutes with 2.5 N HCl at boiling temperature. Following continuous stirring, the boiling acid mixture was poured into 50 ml of ice water and left overnight. The MCC was filtered, rinsed to neutral pH and oven-dried at 60 ° C.		(Naduparambath & Purushothaman, 2016)

Pineapple leaves, banana stem, coconut coir, sugarcane bagasse	For alkaline hydrolysis of waste fibres, NaOH (2 per cent w/v) was added to a water bath and left for 5h at 80°C. After quite a washing with distilled water and filtration, this was bleached for 15 min at 75°C of an aqueous solution of NaClO (1:1). For the extraction of alpha-cellulose, 500 mL of NaOH (12% w/v) was used at 80°C for 1h. MCC extraction was proceeded by hydrolysing with 2.5N HCl at 100°C for 15 minutes, aggressively stirring with a spatula, and allowing to stand overnight before drying at 60°C.	75%, 74.55%, 72.73%, 66.50%	(Sainorudin et al., 2018)
Groundnut husk	The effects of several pulping procedures (NaOH and multistage pulping) and different bleaching times on groundnut husk alpha-cellulose yield were investigated. At 80°C, the powder was delignified with aqueous NaOH. To extract lignin in the form of soluble nitrolignins, the samples were treated with 3.5 percent nitric acid containing 40 mg sodium nitrite for 2 hours in a water bath at 90°C. The alpha-cellulose was hydrolyzed for 15 minutes with 2.5 N HCl. This microcrystalline cellulose was washed with water until neutral, pressed, and dried for 60 minutes in a fluidized bed dryer at an inlet air temperature of 57-60°C.	58-69%	(Azubuike et al., 2012; F. Ohwoavworhua et al., 2009)
Date palm stalk	To begin with, MCC was extracted by bleaching the fibre with 10.0 (w/v) NaCIO. After that, bleached fibres were treated for 30 min at room temperature with an 8.0% (w/v) NaOH (1:50 w/v)). The bleached pulp was washed and dried a 60°C for 24 h. Using 2.5M HCl, undergo hydrolysis of the alkali-treated bleached pulp for 30 min at 85 °C based on a 1:30 (g/ml) with continuous agitation for hydrolysis, then cool to room temperature.	79.4%	(Alotabi et al., 2020; Baruah et al., 2020)

Coffee husk	Powder coffee husks with a particle size of 2-3 mm were alkali treated (4% NaOH) for 3h before being bleached (acetate buffer, sodium chlorite (1.7 %), and acid hydrolysis (Conc. H2SO4, 64 percent, wt/wt at 50ºC for 40 min).	92%	(Collazo-Bigliardi et al., 2018a)
Alfa grass	Extracted alfa cellulose was immersed in concentrated acid and boiled at reflux conditions using continuous stirring, and cooled to normal condition for 30 min. The deteriorated cellulose was neutralised using diluted 1M NaOH, filtered again, rinsed with distilled water, and dried at 50°C . Using a grinder, the resulting Alfa-MCC was ground into a fine powder. The hydrolysis studies were carried out in 2.5 M of HCl (1:10) at 85°C for 120 minutes with continual agitation.	73 %	(Baruah et al., 2020; Beroual, Boumaza, et al., 2021; Trache et al., 2013)
Damaged Palm fruit, Stalk and spikelet	The samples were combined and treated with a NaCIO2 reagent, which was hydrolyzed with acetic acid (pH 4). The fibres were again boiled for 2h (80°C) in acidified NaCIO2 (1:50) and dried at 60°C. The bleached fibres were again treated for 2 h with a 17.5 % NaOH. After that, the mixture was filtered, washed, and dried in a 60°C oven for 24 h, yielding fibrils. The MCC was made by hydrolyzing cellulose fibres in sulphuric acid (55%) solution for 45 min at 45°C. whilst stirring frequently.	82.5, 82.2 & 86.5%	(Xiang et al., 2016)
Jackfruit peel	Three processing stages were compared for effective removal of impurities, fragments and isolation of cellulose from jackfruit peel powder i) sodium chlorite method, ii) acetic acid + nitric acid treatment, and iii) formic acid treatment.	83.42%	(Trilokesh & Uppuluri, 2019)

2.7.4 Benefits and Application of MCC

Critical attributes of microcrystalline cellulose play an important criterion for its functions and applicability in different fields is demonstrated in table 2.5. These properties are mainly classified into three categories as follows- i) degree of polymerization (DP), ii) degree of crystallinity index, iii) mechanical and thermal properties, iv) structural characterization etc., The linkage of DP with physical and chemical parameters will be valid unless the material was acquired from the same source and produce under the same process parameters. The impact of DP influences MCC's inherent properties. Increased DPs of cellulose have been reported to have greater water holding capacity compared to smaller DPs, due to the relatively large amount of hydroxyl groups making them more hygroscopic. Moreover, longer DP of MCC solid particles are expected to be less crystalline since the presence of even more amorphous parts, which are more hygroscopic, if compared to short DPs.

Table 2.5 Applications and benefits of microcrystalline cellulose in different fields

Application	Benefits	Reference
Pharmaceutical industries		
Pickering emulsion	• Its inherent rigidity and strength • Experimental mechanical coupling to yield a high-strength hydrogel.	(Buffiere et al., 2017; Shahbazi et al., 2022)
Tableting & drug delivery	• Carrier of drugs such as Isoniazid, Ranitidine, Cephalexin, 5-fluorouracil.	(Diarsa & Gupte, 2021; Seera et al., 2020)
Hydrogel	• The mixture of microcrystalline cellulose and sodium carboxymethylcellulose • Bacterial cellulose and chitosan composite hydrogel • Cellulose-based conductive hydrogels • Cellulose hydrogel microspheres	(Bai et al., 2018; Dai et al., 2020, 2021; Jo et al., 2019; Seera et al., 2020)

Filler	• Compressible filler- the number of active components that a diluent can adequately transport using direct compression • Wet granulation filler- Water-insoluble yet hydrophilic filler with swelling characteristics and excellent water-absorbing or spreading ability • Roller compaction- Roller compaction is a dry method that entails the compression of particles into the ribbon, which gets pulverized to generate granulated especially for moisture sensitive drugs	(Yohana Chaerunisaa et al., 2020)
Binder	• Dry binder- help in spray-dried drugs or dried powder to compressible tablet • Wet binder- It can be used to granulate both soluble and insoluble powders as supplementary binding material in wet granulations.	
Disintegrant	• When wet, disintegrants expand and dissolve, causing the tablet to disintegrate in the gastrointestinal tract and release the bioactive constituents for assimilation.	
Lubricant	• Keep the components from aggregating and adhering to the pill presses or capsule filling	
Glidant	• Reduce interparticle friction and cohesiveness to improve powder flow. eg: Proslov, which is high functionality silicified MCC	

Sensory enhancer	• Smooth palate, that helps to mitigate the negative impact of its insolubility. • Also helps to lower the overall amount of disintegrant required.	
Cream stabilizer	• Preserve suspension consistency and avoid settling, add thixotropic viscosity, and improve formulation stability	
Packaging applications		
Films	• Spherical composite • Composite film • Biocomposite films • Gelatin composite film • Thermoplastic Composites • LDPE-based composites • Starch-based films	(Law & Deasy, 1998; Limwong et al., 2004) (Noishiki et al., 2002) (Kale et al., 2018) (Pan et al., 2020) (Kiziltas et al., 2014) (Piechowiak et al., 2020)
Food applications		
Pickering emulsion gel	• 3D Food Printing	(Ahsan et al., 2019; Shahbazi et al., 2022; K. Wang et al., 2021)
Aerogel	• Wastewater treatment	(Wei et al., 2018)
Hydrogel	• Toxic metal removal	(Dai et al., 2021; Huang et al., 2020; Seera et al., 2020)
Fat replacer	• Improve the textural attribute in low-fat food products such as beverages, yogurt shakes, chocolate beverages and sausages.	(Gibis et al., 2015; Schuh et al., 2013; Zbikowska et al., 2018)
Thickeners and stabilizers	• Processed milk and milk products • Emulsions	

Anticaking	• Reduce the aggregations, sticking in hygroscopic powders, sauces, dressings	
Encapsulations	• Act as a matrix as a carrier of probiotic microorganisms and deliver to the stomach	
Cosmetics applications		
Abrasives, absorbent, peeling agents and lubricants,	• Consistency regulator in creams • Emulsifiers in sunscreen lotions • Waterproof skin products • Talc and baby powder	(Nsor-Atindana et al., 2017, 2020; Xiang et al., 2016)

2.8 Gap and need for the research work

The demand for microcrystalline cellulose application is rising with a CAGR rate of 6% from 2019-to 2027. Shallot biowaste is a rich source of crude fibre with 55.97, 49.72 and 41.49% in skin, stalk and petiole respectively which includes lignin, hemicellulose and cellulose. This cellulose can be further modified depending on its utilization as a gelling agent, fat replacer, texturizing and thickening agent in various food product development.

Hence the current study focus on the extraction of cellulose from shallot biowaste and the conversion to MCC. Further, the application of MCC in food products as a fat replacer in low fat stirred fruit yogurt and positive correlation with physical, chemical, and microbiological properties along with improved textural characteristics were also studied.

CHAPTER-3

MATERIALS AND METHODS

The chapter describes the procurement of materials and the methodology adopted for the current research. It gives an account of the selection of materials, experimental setup, and parameters for the valorisation of shallot biowaste into valuable products for various food products. The extraction of products, viz. cellulose and microcrystalline cellulose, from SBW's in a probiotic system was elaborately explained. Furthermore, the importance of physicochemical, functional, morphological, and crystalline properties was emphasized. Also, the incorporation of extracted MCC into low-fat stirred plain and fruit yogurt as a stabilizer and fat-mimicking agent was detailed in the methodology.

3.1 Materials

3.1.1 Procurement of raw materials

Shallot bio-waste that comprises the petiole (PT), peel (P) and stalk (S) was derived from the CO-2 variety, was procured from the small onion processing company in Chettikulam village, Perambalur district, Tamil Nadu, on a seasonal basis. Analytical grade chemicals and reference standards used mainly, microcrystalline cellulose (Sigma-Aldrich, Bangalore), sodium hydroxide, sodium hypochlorite (30%), hydrochloric acid (37%), acetic acid, ethyl alcohol, acetone, etc., were purchased from Himedia and Loba chemicals. Furthermore, to prepare yogurt, skim milk and fruits (ripe jamun) were purchased from the local market. Starter cultures were procured from commercial companies.

3.1.2 Sample preparation

Foreign matter such as dust, sticky muds, stones, weeds, plants, and damaged parts was manually cleaned and sorted from the collected samples. The samples were dried at 50°C in a solar hybrid drying chamber until it attained a moisture level of 5%. To yield

powder form, dried samples were cut and pulverised to powder using a pin mill (EssEmm Corporation, Coimbatore, Tamil Nadu, India) and filtered through sieve of 0.75 mm opening. For later use, finely powdered samples were kept in sealed containers and incubated at room temperature(Varghese et al., 2022).

3.1.3 Chemical investigation of shallot waste

The shallot waste streams were evaluated using AOAC methodologies(AOAC, 1995). Using a modified fractionation method, the insoluble and soluble constituents of pectin and lignocellulose were analysed(Adel et al., 2011; Collazo-Bigliardi et al., 2018a)., 2018).

3.1.3.1 Moisture

The gravimetric method was used to determine the amount of moisture in shallot waste streams (AOAC 930.15). A known amount of sample was deposited in a moisture dish and dried for 12 h in a drying oven (Leads India Laboratory Equipment, Chennai, India). Equation (3.1) was used to calculate the amount of moisture in the samples.

$$Moisture\ content\ (\%) = \frac{(W_1 - W_2)}{W} \times 100 \qquad \ldots 3.1$$

where W_1 and W_2 are the masses of the samples before and after dehydration, and W is the initial mass taken for drying.

3.1.3.2 Protein content (P)

The protein quantity was calculated using the micro-Kjeldahl method (AOAC 920.87). 0.5 g of sample was weighed in the digestion tube, followed by 1 g of copper sulphate, 10 g of sodium sulphate, and 10 ml of sulphuric acid for the digestion procedure. The sample was then thermally digested for 3 hours at 420°C, followed by distillation and titration against 0.1N HCl solution in the presence of a mixed indicator (Kirk, 1950). The total nitrogen content and crude protein were estimated using the calculations following:

$$Nitrogen\ Content\ (\%) = \frac{(T\ - B) \times N \times 14}{W \times 1000} \qquad \ldots\ldots\ldots (3.2)$$

where, T is titre value, B is blank value, N is normality of HCl used for titration and W is weight of sample.

$$Protein\ (\%) =\ Nitrogen\ Content \times 6.25 \qquad \ldots\ldots\ldots (3.3)$$

where, 6.25 is the multiplication factor.

3.1.3.3 Fat content

With slight changes, the fat content of shallot biowastes was measured using the Soxhlet method (AOAC 948.22). A known quantity of sample was weighed and packaged in filter paper. After that, the filter paper is preserved in a thimble and placed in a pre-weighed glass vessel. Following that, 90 ml of hexane was added, and the extraction was carried out using the regular programme in the automatic soxhlet equipment (Socs Plus, SOS 06 RTS, Pelican Equipments, Chennai, India). The following equation was used to compute total crude fat.

$$Fat\ (\%) = \left(\frac{W_2 - W_I}{W_3}\right) \times 100 \qquad \ldots\ldots\ldots (3.4)$$

where, W_2 is final weight, W_1 is initial weight of glass vessel and W_3 is weight of the sample.

3.1.3.4 Fiber content

The acid-alkali technique was used to determine the fibre content of shallot waste samples (AOAC 985.29). A predetermined amount of material was dried and defatted. The moisture and fat-free sample was first added and boiled with 1.25 percent H_2SO_4, followed by 1.25 percent NaOH. The digested sample was then filtered and dried using pre-weighed Whatmann filter paper. The dried sample was then weighed and subjected to ash content determination. The crude fibre content of shallot waste streams were calculated using Equation (3.5).

$$Fiber\ (\%) = \left(\frac{W_2 - (W_I + W_4)}{W_3}\right) \times 100 \qquad \ldots\ldots\ldots (3.5)$$

where, W_2 is final weight, W_I is initial weight of empty filter paper, W_4 is weight of ash content and W_3 is weight of sample.

3.1.3.5 Soluble and insoluble fractions of fibre content

The peel (P), stalk and petiole from the shallot waste streams were milled to fine powder. The samples were treated with 0.1 HCL (pH 2.0) at 80°C for 2h. solid liquid ratio should be maintained by 1:15. The soluble fraction was separated from the mass and concentrated to 1/3 parts of the initial volume. Cool and precipitate the liquid by 95% of the ethanol (1:2). Keep the precipitate overnight without disturbing at 4°C and separated the precipitate by filtration through whatmann filter paper. Thoroughly wash the precipitate with distilled water and oven dry the precipitate at 70°C. The precipitate weight gives the total pectin content (%). Insoluble fractions of the samples were treated with bleaching agent (pH-11.5) at 60°C for 18h with solid to liquid ratio of 1:20. Separate the filtrate from the insoluble fraction and adjust to pH 5.5 with acetic acid. precipitate the sample by 95% ethanol and concentrate the liquid for the separation of ethanol and wash the precipitate with excess of distilled water and filter through whatmann filter paper and dried in hot air oven. These precipitates are known as lignin content (%). The residue fractions were again washed thoroughly with distilled water which is termed as alkali insoluble hemicellulose (%). Remaining insoluble fraction was washed by 3 volumes of distilled water until it attains the neutral pH followed by purification method (ethanol and acetone). The insoluble fraction was dried in an oven kept at 60°C for 16h. Difference in the final and initial weight of the sample gives the % of total cellulose content (Adel et al., 2011; Lu et al., 2013).

3.1.3.6 Total ash content

The ash content determines the quantity of mineral present in given products (AOAC 942.05). As a result, the potential of shallot biowaste to supply minerals was analysed using the procedure modified by Bicu and Mustata (Bicu & Mustata, 2013). In pre-weighed crucibles, a known amount of powdered shallot waste streams was deposited. The samples were then charred with a hot plate, followed by 4 hours of ashing at 600 °C, and final calculations were formulated as follows.

$$Total\ Ash\ (\%) = \left(\frac{W_2 - W_1}{W_3}\right) \times 100 \qquad \ldots\ldots\ldots 3.6$$

where, W_3 is final weight, W_1 is initial weight of empty crucible and W_3 is weight of sample.

3.1.3.6 Total carbohydrate content (TC)

The sample's carbohydrate content was estimated using the differences in composition. The macronutrients in the commodity, together with moisture, were assumed to be 100 %, and the amount of carbohydrate was calculated by subtracting the other constituents from 100. The calculations were carried out using the following formula (3.7).

$$Total\ carbohydrate\ (\%) = 100\ - (M + P + F + Fb + A) \qquad \ldots\ldots\ldots 3.7$$

where, M is moisture content, P is protein, F is fat, Fb is fibre and A is total ash content of sample.

3.2 Methodology

3.2.1 Pre-treatments and extraction of cellulose

The extraction and isolation procedures for cellulose and MCC from the shallot bio-waste petiole (PT), peel (P), and stalk (S) are depicted in fig.1. The main pre-treatments for cellulose isolation were a combined of time and concentration of NaOH followed by mineral acid hydrolysis(Varghese et al., 2022). Two pre-treatment processes were used to completely separate cellulose and MCC viz., hot water treatment in water bath (W) and steam treatment in autoclave (A). In the case of steam assisted autoclave method, variations

were made in concentration of NaOH (1, 1.5, 2N) while maintaining a constant time, temperature and pressure as 30min, 121℃ and 15 psi respectively.

Meanwhile, in the case of hot water treatment in water bath, the samples were subjected to pre-treatment at 60℃ with variations in concentration of NaOH (1, 1.5, 2N) and time (30, 60, 120 min). Accurately weighed samples were taken in glass beaker. The fractionation of cellulose starts with addition of water in the ratio of 1:10 (W/V) and subjected to pre-treatments as per aforementioned procedures. These pre-treatments play an important role in the removal of water soluble components like sugar, volatile components and impurities and soften the structure of the petiole, peel and stalk (Feng et al., 2018; Seehra et al., 2014).

Dewatered samples were treated twice with NaOH solution with varying concentrations of 1, 1.5 and 2N in ratio of 1:20 (W/V) at 80℃ for 30min. The alkali hydrolysis leads to protein denaturation and delignification. Furthermore, samples were bleached with 10% sodium chlorite in combination with acetic acid to adjust the pH to 4 which will significantly solubilize the pigments, wax and lignin completely (Bicu & Mustata, 2011; Wijayanti et al., 2021).

This process is continued for 3 or more cycles applied until its colour changes from dark brown to white gel of cellulose. It is further filtered and washed with deionized water until all the residues were removed along with the residual odour of chlorine(Alba et al., 2018).

3.2.2 Transformation of alpha-cellulose to Microcrystalline cellulose

Extracted cellulose was mixed in a 1:20 (w/v) ratio with concentrated HCl (2.5N) and allowed to treat at 100°C for fifteen min. To stop the reaction by expelling the acid, the derived MCC was cleaned and filtrated with surplus deionised water. Moreover, the depolymerized components were cleansed by washing them with suitable solvents like

distilled water, ethanol, and acetone till its end product attained pH of 5.6-7. The procured MCC solution is then oven dried at 60°C for 24h for further evaluation.

Table 3.1 Different pre-treatments, time and concentration of NaOH applied for isolation of cellulose and MCC from shallot waste

<table>
<tr><th>Time (min)</th><th>Pre-treatment Temperature (°C)</th><th>Concentration of NaOH</th><th>Responses analysed</th></tr>
<tr><td colspan="3">Hot water assisted pre-treatment (W)</td><td rowspan="8">Cellulose %
Ash %
MCC%
Colour analysis</td></tr>
<tr><td>30</td><td rowspan="3">60</td><td>1.0N
1.5N
2.0N</td></tr>
<tr><td>60</td><td>1.0 N
1.5 N
2.0 N</td></tr>
<tr><td>120</td><td>1.0 N
1.5 N
2.0 N</td></tr>
<tr><td colspan="3">Autoclave assisted pre-treatment (A)</td></tr>
<tr><td>30</td><td>121</td><td>1.0 N
1.5 N
2.0 N</td></tr>
</table>

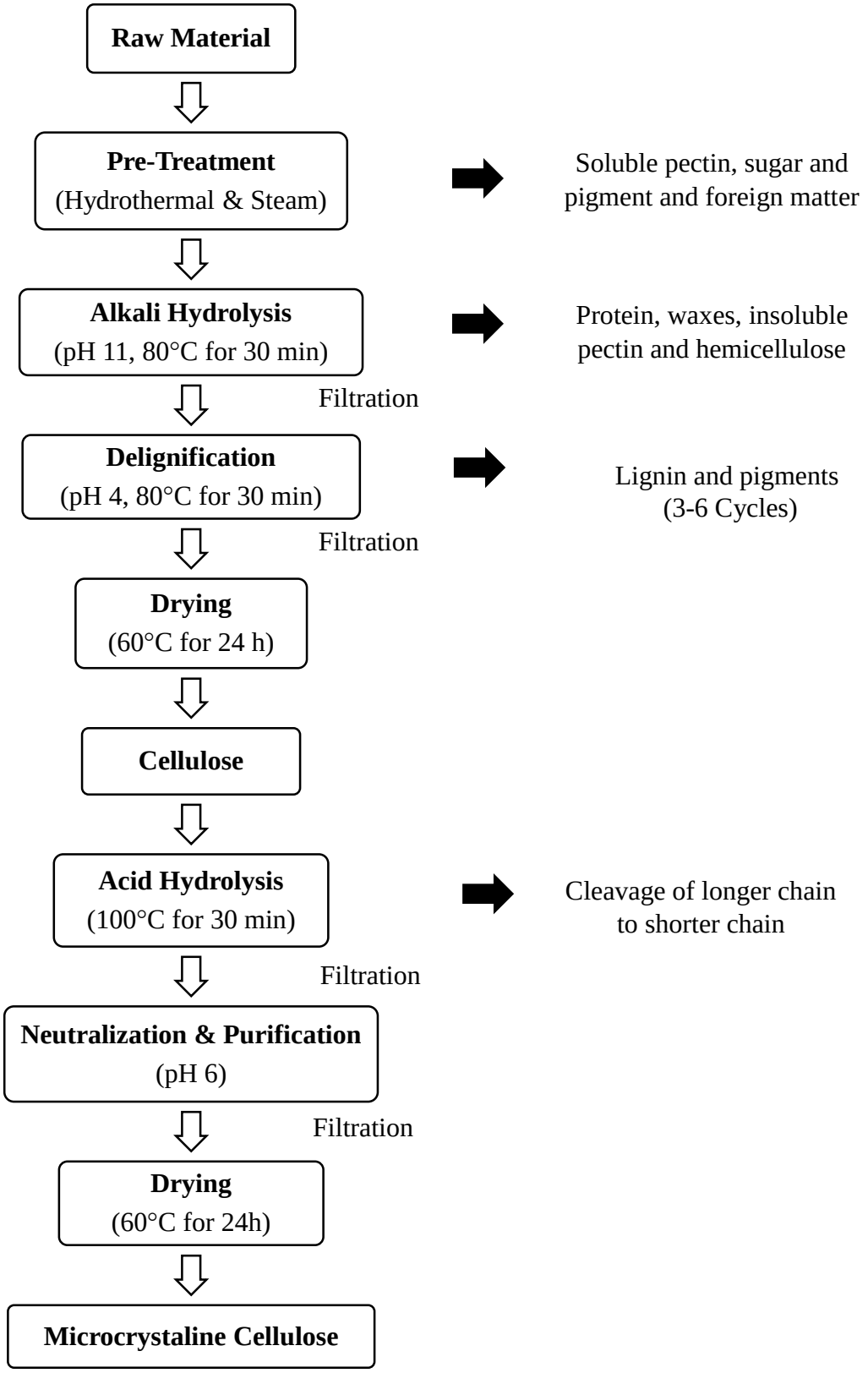


Figure 3.1. Process flowchart for extraction of cellulose and MCC from shallot waste streams

3.3 Quality evaluation of extracted MCC

3.3.1 Yield and recovery %

The percentage of isolated cellulose and MCC were compared to initial mass of the shallot waste to determine the yield and recovery % (Ding et al., 2020; Ilyas et al., 2018)by following formulae detailed in equation (3.8) and(3.9)

$$Yield\ \% = (W_c/W_b) \times 100 \qquad \ldots\ldots\ldots 3.8$$

where, W_C is the isolated shallot cellulose and W_b is the initial weight of the SBW.

$$Recovery\ \% = \left(\frac{W_c}{W_m}\right) \times 100 \qquad \ldots\ldots\ldots 3.9$$

where, W_C is the initial weight of the extracted cellulose and W_m is the weight of the MCC content.

3.3.2 Colour analysis

Colour determination is done by hunter lab colorimeter (ColorFlex EZ, Hunter Associates Laboratory Inc., Virginia, USA) (Karaman et al., 2017). The ΔE gives the total colour difference of samples and is calculated as given in equation (3.10)

$$\Delta E^* = \sqrt{\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2}} \qquad \ldots\ldots\ldots 3.10$$

where, ΔE is total colour difference and L^{*2}, a^{*2}, b^{*2} were the values of lightness, redness and yellowness of corresponding to the extracted samples

Hue angle, a quality property of colour attribute by which colours have been designated as reddish, greenish, and so on, and is often used to make the distinction between a specific hue and a grey colour of the same luminance (Sumonsiri et al., 2018). Its

characteristic is about the absorbance variations at different wavelengths were calculated by equation (3.11)

$$h^* = \frac{tan^{-1} b^*}{a^*} \qquad \ldots\ldots\ldots 3.11$$

where, h^* is hue angle exemplify the different angles such as 0°, 90°, 180°, 270° which includes red-purple, yellow, bluish-green and blue traits.

3.4 Characterization of microcrystalline cellulose

The isolated MCC were characterised using various spectral and analytical techniques.

3.4.1 Fourier transform infrared spectroscopy (FTIR)

Fourier transform infrared spectroscopy is an instrument which determine the functional groups present in extracted sample. The samples were pulverized, dried and mixed with potassium bromide and compressed to tablets. The transmittance (%) determination is carried out in the spectral range of 4000-400 cm^{-1} with resolution of $4cm^{-1}$ using 32 scans using Nicolet iS50 model (TESCAN, VEGA 3, model no. 99065) (Akatan, 2022).

3.4.2 Scanning Electron microscopy (SEM)

The scanning electron microscopy(SEM) was mainly used to scan the structural pattern of MCC powder. A small quantity of dried samples was spread uniformly in a conductive adhesives and coated with thin gold. Scanning is completed beneath the specific distance and accelerating voltage of 5kV using VEGA 3 TESCAN model with different magnifications. Micrographs of each MCC samples magnified in 500µm and 1000µm. Also through SEM images, 100 particles per screen were taken for quantification of particle dimension using image analysis program (1.52V, USA)(Azubuike & Okhamafe, 2012; Jiang et al., 2020; Satyamurthy et al., 2011; Uesu et al., 2000).

3.4.3 X-ray diffraction analysis (XRD)

The crystallinity of the isolated MCC samples was investigated using an X-ray diffractometer (Malvern pAnalytical-XPERT-3). Powdered samples were examined with Ni-filtered Cu-K radiation (=0.15406 nm) at various angles of incidence ranging from 10° to 50° (2), with a generated voltage of 45kV. With a ramp rate of 1°C/min, all samples were analysed. Origin software 2018 was used to determine the peaks formed by the diffractogram. This software was used to compute the full width at half maximum (FWHM), area of crystalline and amorphous regions, crystalline size (D), and crystallinity index (CI). Scherrer's equation was used to calculate crystalline size (D) by equation (3.12).

$$D = \frac{K\lambda}{\beta} cos\theta \qquad \ldots\ldots.3.12$$

where, *D* is the crystalline size, *K* is Scherrer's constant (0.9), λ is wavelength of X-ray, β is full width at half maximum in radians and θ is Bragg angle.

The quantification of crystallinity index (CI) was calculated by using equations (3.13) and (3.14) namely by peak-height ratio and peak deconvolution method. Both methods have 10- 15% difference in the CI, while the deviation in line are usually fitted(Agarwal et al., 2018; Ahvenainen et al., 2016; Collazo-Bigliardi et al., 2018b; Ju et al., 2015). But for the convenience and easy application, peak-height method is mainly used for the quantification of crystalline index. Here, both models were used for the estimation of CI. Former method commonly known as Segal method that quantify the relative crystallinity index depending on the least intensity peak and highest intensity peak as per the given equation (3) from PANalytical software 2019.

$$CI = I_{200} - I_{110}/I_{200} \times 100 \qquad \ldots\ldots\ldots 3.13$$

where, I_{200} and I_{110} are the peaks corresponding to fraction of crystalline and amorphous regions respectively. Crystalline and amorphous peaks are deconvoluted by integrating the

area of peaks reconnoitred by fitting the Gaussian curve using Origin pro 2018 software (SR1, origin lab Corporation, USA).

$$CI = \left(\frac{A_C}{A_C} + A_{Am}\right) \quad \ldots\ldots\ldots 3.14$$

where, A_c is the integrated area of crystalline peaks and A_{Am} is the integrated area of the amorphous region.

Crystalline properties were also evaluated by finding d-spacing and Microstrain. The d-spacing was calculated by the following formula (3.15)

$$d = \frac{K\lambda}{D} Cos\theta \quad \ldots\ldots\ldots 3.15$$

where, D is the crystalline size, K is Scherrer's constant (0.9), λ is wavelength of X-ray and θ is Bragg angle.

Microstrain (nm^{-2}) was calculated by the following equation (3.16):

$$\varepsilon = \frac{\beta}{4} tan\theta \quad \ldots\ldots\ldots 3.16$$

where, ε is denoted as microstrain, β is full width at half maximum in radians and θ is Bragg angle.

3.4.4 Thermal analysis

A Differential Scanning Calorimeter was used to measure the thermal resistance of the extracted MCC from the SBW (DSC3, Mettler Toledo, Switzerland). Heat was applied at 40°C-350°C at a rate of 20°C/min to approximately 6mg of powdered sample in an aluminium pan. The nitrogen flushing rate was kept constant at 20ml/min (Alotabi et al., 2020).

3.5 Functionalities of extracted cellulose and MCC

Flow and hydration properties of extracted cellulose and MCC from shallot biowaste were used to determine functional properties. Prior literature is being used to assist the procedures, for determining the properties.

3.5.1 Flow properties

The flowability of powder samples is all about breaking through the flow resistance and allowing the powder particles to pass over each other. The bulk, true and tapped densities, hausner's ratio, carr's index, and porosity of the extracted cellulose and MCC powder samples were evaluated. By adding 2g of sample powder to a 100 ml graduated measuring cylinder and dividing the weight of the sample (g) by the volume of the sample, the bulk density was calculated (ml).

Tapped density is calculated by placing 1g of sample in a measuring cylinder and tapping it 300 times on a flat surface. The initial and final volume were measured and calculated as a percentage. The difference in the tapped and bulk densities was estimated and divided by the tapped density (Jiang et al., 2020; Kolakovic et al., 2011).

This ratio, expressed as a percentage, is known as hausner's density (Koç et al., 2014). The following relationship between bulk density and particle density was used to calculate particle porosity (ε)(Heng & Koo, 2001). All of these properties were measured in triplicate.

$$BD = \left(\frac{M}{V}\right) \qquad \ldots\ldots\ldots 3.17$$

Where, M and V is the mass and volume of the samples

$$TD = \left(\frac{M}{V_{TD}}\right) \qquad \ldots\ldots\ldots 3.18$$

Where, M is the mass of the sample and V_{TD} is the volume of after tapping

$$CI = \left(\frac{TD - BD}{TD}\right) \qquad \ldots\ldots\ldots 3.19$$

Where TD is the tapped density of samples and BD is the bulk density of the sample

$$HR = \left(\frac{TD}{BD}\right) \qquad \ldots\ldots\ldots 3.20$$

where, HR is the hausner's ratio and BD is the bulk density of the sample

$$Py\ (\%) = \left(1 - \frac{BD}{T}\right) \qquad \ldots\ldots\ldots 3.21$$

3.5.2 Hydration properties

The hydration properties included water holding capacity (WHC), water solubility index (WSI), swelling capacity (SC) and oil holding capacity (OHC). These properties are important to determine powder samples with insoluble fibres as compared to commercial powders(Rodríguez et al., 2006).

3.5.3 Water holding capacity (WHC)

The WHC of powder samples are determined by the amount of water retained by the sample. Weighed sample(1g) dispersed in 50ml of the distilled water, vortexed(1min) and hydrated for 18h. Centrifuge the samples at 3000rpm for 20min. Decanted sample kept carefully inverted to drain off and weighed and expressed in g/g as dry basis (Twarogowska et al., 2020).

$$WHC = \left(\frac{W_F - (W_I}{W_S}\right) 100 \qquad \ldots\ldots\ldots 3.22$$

where, W_F is final weight, W_S is weight of sample and W_I is initial weight.

3.5.4 Water solubility index (WSI) and swelling index (SI)

The sample (0.1g) was suspended in 30ml of distilled water in centrifuge tube. Mix and vortex for 10min at 90°C for 30min. centrifuge for 3000rpm for 20min. Decant the sample and dried(105°C) until it reaches constant weight. The weight is expressed as g/g of dry basis. Weigh the sample (0.5g) and poured to measuring cylinder containing 15ml of distilled water and incubated for 18h at 20-25°C. Record the initial and final bed volume of the sample. Swelling index can be expressed as the volume in ml/g of the dry basis (Dong et al., 2020).

$$WSI = \left(\frac{W_F - W_I}{W_S}\right)100 \qquad \ldots\ldots\ldots 3.23$$

where, W_F is final weight, W_S is weight of sample and W_I is weight of initial weight.

$$SI = \left(\frac{V_3 - V_2}{W}\right) \times 100 \qquad \ldots\ldots\ldots 3.24$$

where, V_3 is final volume, W is sample weight and V_2 is initial volume.

3.5.5 Oil holding capacity

1g of the sample added to a 50ml of the centrifuge tube with 10ml of sunflower oil and vortexed for 1min and stand for 18h at room temperature. Then centrifuged at 3000rpm for 20min, decant the supernatant and drain off the excess oil. Weigh the wet residue and expressed as g /g dry basis(Dong et al., 2020; Twarogowska et al., 2020).

$$OHC = \left(\frac{W_F - (W_D)}{W_S}\right)100 \qquad \ldots\ldots\ldots 3.25$$

where, W_F is final weight, W_S is weight of sample and W_D is weight of empty petri dish.

3.5.6 Foaming properties of extracted SBW's cellulose and MCC

In a blender, 0.5g of the sample powder was whipped in 25ml of distilled water for 5 minutes. This mixture was transferred quickly to a 50ml measuring cylinder. The final volume of foam was recorded and expressed as ml/ml for foaming capacity. The foam stability was expressed as a percentage of the initial foam volume. The volume of foam after 1 h of whipping was recorded to determine the foam stability (Bhosale et al., 2022a; Çalışkan Koç & Özçıra, 2019; Mokhtar et al., 2018; Sundarraj & Ranganathan, 2018).

$$FC = \left(\frac{V_W}{V_I}\right)100 \qquad \ldots\ldots\ldots 3.26$$

where, V_I is initial volume and V_W is volume after whipping

$$FS = \left(\frac{V_F}{V_W}\right)100 \qquad \ldots\ldots\ldots 3.27$$

where, V_W is volume after whipping and V_F final volume after 1h resting time.

3.6 Rheological investigation

The extracted cellulose and MCC viscosity was measured using a Rapid –Visco Analyzer (RVA) (MCR 52, Anton Paar GmbH, Austria). For 12h at room temperature, the sample was dispersed in distilled water while vigorously stirring in a rotor shaker. Its rheological behaviour was investigated using a rheometer equipped with a parallel plate (PP25). The steady viscosity of each sample was determined using a flow curve and shear rates ranging from 0.1 to 100 counts per second at 25°C. Using power law fitting, the flow behaviour was evaluated using the Herschel-Bulkley model.

3.7 Particle size distribution and zeta potential capacity

The Zetasizer (Nanoplus, Malvern Instruments, Malvern UK) was used to perform a dynamic light scattering (DLS) analysis of 0.001 wt. % cellulose and MCC suspension at a temperature of 25°C (L. Chen et al., 2018; S. He et al., 2019). In air, a diffraction pattern

study of a stream of dry powders was done. As a result, D10, D50, and D90 denote the 10, 50, and 90 percent of cumulative percentiles of particle sizes respectively (K. Das et al., 2009; Nakamori et al., 2008).

3.8 Optimization and replacement efficiency of MCC as fat replacer and stabilizer in low fat stirred plain yogurt

3.8.1 Preparation of stirred yoghurt with added P-MCC (Peel), S-MCC (Stalk) and PT-MCC (Petiole)

As showed in fig 3.2, skim milk was pasteurized by a water bath at 83°C for 30min and subsequently cooled down to 42°C by keeping in iced water bath. The samples were transferred to a 100ml glass bottle and P-MCC, S-MCC and PT-MCC were added at 6 treatments different concentration ranging from 0.25% to 1.0%.

In each sample along with WMC and SMC (without MCC) at different temperatures (37˚C & 45°C). Stirred well for 10min until it disperses properly. Afterwards, commercial starter culture comprising of *Streptococcus thermophiles*, *Lactobacillus cremoris* and *Lactobacillus bulgaricus* directly inoculated with 0.2% (w/v) as mother culture and a known quantity was transferred into samples and gently stirred for 1min.

The samples were prepared according to fig. 3.2 and the fermentation process was carried at T_1 and T_2 until it reaches a pH value of 4.6. The finished products were homogenized at 9500rpm for 2min for the uniform distribution of the MCC. The yogurt samples were immediately cooled at 4˚C and stored for 48h. The quality parameters were inspected throughout the processing and storage of developed low fat stirred yoghurt (Costa et al., 2019; Shery M Varghese, Aruna Nair U K, 2021; Torrico et al., 2020).

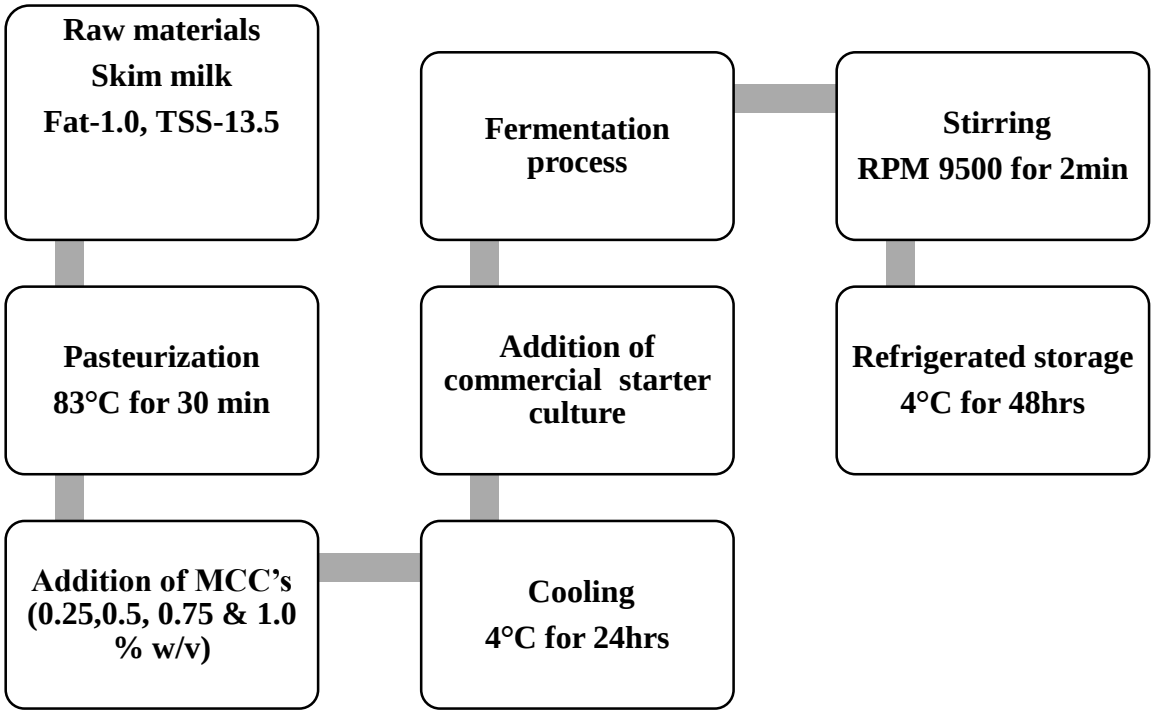


Figure 3.2. Flow diagram for preparation of low fat stirred yogurt with the incorporation of MCC extracted from shallot waste streams

3.8.2 Post fermentation analysis

The post fermentation analysis helps to determine the incubation time and also monitor the fermentation factors such as pH, Titratable acidity (TA) and turbidity after the incorporation of C-MCC, P-MCC, S-MCC and PTMCC and starter culture (Altemimi, 2018; Buchilina & Aryana, 2021).

3.8.2.1 pH

The pH of samples was determined using a digital pH meter (King's lab, Model; KLPHM-114) which is attached to glass electrode after the buffer calibration.

3.8.2.2 Titratable acidity

The presence acidity in the samples can be determined by the titration with the standard base. Thus, Titratable acidity (TA) were expressed by % of lactic acid by 0.1N NaOH by chemical titration.

1ml of 0.1N NaOH = 9mg of lactic acid

3.8.2.3 Total soluble solids

Total soluble solids were evaluated using a digital refractometer (Model 3810, Atago, Fukayashi). Homogenized samples were drop wise added and expressed as °B (Brix)

3.8.2.4 Probiotic growth analysis

Samples were inoculated by 2% of the culture at 45°C and incubated at 37 and 43°C. 3µl of the samples were withdrawn at each 1h for the quantification of lactic acid bacteria(LAB) using UV- visible micro-plate reader (Spectrum max Id 3, molecular devices, LLC USA) at 600nm(Zhang et al., 2015).

3.8.3 Physiochemical analysis

Similarly, the finished product was also analysed for moisture, protein, fat, total ash, total fibre and carbohydrate along with colour analysis using AOAC method. The detailed methodology was described in section of 3.1.3 under this chapter.

3.9 Effect of incorporation of SBW's MCC in production of low fat stirred yogurt as stabilizer

3.9.1 Serum separation rate and Syneresis

Syneresis or serum separation gives an account of whey separation from solid mass and the extent of resistance of MCC's incorporated in stirred skim milk yoghurt. Ten grams

of samples were taken in a centrifuge tube and were centrifuged at 4°C for 0, 3, 6, 9, 12 and 15min at 1200rpm (Model-E-Spin Lark Make)(Kycia et al., 2018). The whey separated from the supernatant was quantified with a measuring cylinder. Following, formula was used for the determination of serum isolation rate(Gilbert et al., 2020).

$$SSR = v_2 - \frac{v_1}{v_1} \times 100 \qquad \ldots\ldots\ldots 3.28$$

Where v_1 is the volume of the sample and v_2 is the volume of serum separated.

3.9.2 Rheological characteristics

The flow behaviour and viscoelastic behaviour of the low fat stirred plain yogurt samples was determined by Rapid –Visco Analyser (RVA) (MCR 52, Anton Paar GmbH, Austria) instrument equipped with cone and plate probe (CO 25). Each Sample were placed in the RVA plate and rest for 3min to minimize the disturbance during transfer to the plate. The steady viscosity of each sample was measured by flow curve using shear rate in the range of 0.1-100 counts per seconds under 25°C along with triplicates. The fitting of the curve was done using Herschel bulkley model to find out the flow index (n)(Sah et al., 2016).

$$\tau = k\gamma^n \qquad \ldots\ldots\ldots 3.29$$

Where, τ is the shear stress, k is the consistency index and n is the flow index. Whereas n = 1 (Newtonian fluids), n < 1 (Pseudo plastic fluid), and n > 1 (Dilatant fluid).

3.9.3 Texture evaluation

Texture attributes of stirred skim milk yoghurt incorporated by MCC were evaluated by using a texture analyser, TA.XT. plus (stable microsystem) with a 5kg load cell. The texture profile carried out by cylinder-shaped probe with 35mm diameter(A/BE-d35) with specific test speed of 1.0mm/s, penetration distance (25mm) and surface trigger at 10g by

Firmness, consistency, cohesiveness, work of cohesiveness and intrinsic viscosity was estimated using data processed by test Xpert software 2018.

3.9.4 Microbiological analysis

Microbial quality of the stirred yogurt was determined by total plate count, mold and yeast count and E-coli count(Bhat et al., 2018). The following procedures were used for the same. The samples were homogenized by vortex mixer and were serial diluted by distilled water.

3.9.4.1 Total plate count

Determination of total plate count gives an account about the bacterial load in the developed products. From the serial dilution (10^{-5} and 10^{-6}), 0.1ml of the sample were spread plated into sterilized Petri plates containing 15ml of molten nutrient agar. Triplicates plates were incubated at 37°C for 72h. Results were expressed in CFU per gram of sample products.

3.9.4.2 Mold and yeast

Mold and yeast were estimated by using spread plate by Potato dextrose agar (PDA) media with serial dilution of 10^{-3} and 10^{-4}. This plates were incubated at 25°C for 5 days. Results were expressed as mold and yeast per gram of sample product(X. Wang et al., 2019).

3.9.4.4 E-coli count

Faecal Coliform bacterial count was determined using eosin methylene blue method (EMP media) by spread plate method and incubated at 37°C for 24h. EMP media is a selective and differential stain for identification of E-coli. The detection of metallic green colonies was observed as faecal E-coli forms and results were expressed as count per gram of stirred yogurt.

3.9.4.5 LAB (lactic acid bacterial) count

The probiotic bacterial growth rate was detected by MRS agar (de Man, Rogosa & Sharpe) method. Sterilized plates were filled with 15ml of sterilized MRS media. Using spread plate method, samples were taken from the serial dilutions (10^{-5} and 10^{-6}) and incubated for 24h at 37°C. The colonies formed was expressed in CFU per gram of sample products.

3.9.5 Sensory quality of developed low fat stirred plain yogurt

Sensory attributes help to evaluate the acceptability of the developed stirred yogurt. In this evaluation, 30 semi-trained panel members, aged between, 21 and 50, group of male and female students and staff were participated from NIFTEM-T. The hedonic method which include 9 –point scale like 1- Dislike extremely, 2- Dislike very much, 3- Dislike moderately, 4- Dislike slightly, 5-Neither like nor dislike, 6-Like slightly, 7-Like moderately, 8-Like very much and 9-Like extremely was used. In-addition to compositional properties, sensory attributes help to optimize the products depending on the consumer's expectation and preference which act as effective alternative for the selection of the enriched products (Vanegas-Azuero & Gutiérrez, 2018).

The low fat stirred yogurt samples were provided at 10°C in sterilized glass container labelled with code numbers (Nair et al., 2021). Each of the samples were assessed for appearance, color, taste, texture, aroma, aftertaste, syneresis and overall acceptability. All the panellist were instructed about the yogurt samples before starting the evaluation with questionnaire. Thus, ranking of each product was observed for selection of the final product.

3.9.5.1 Acceptability Index

Acceptability index of the low fat stirred yogurt incorporated with SBWs MCC was determined by following equation (3.31)(Lafarga et al., 2019; Lucas et al., 2018b)

$$AI = \left(\frac{Lowest\ score}{Highest\ score}\right) \times 100 \qquad \ldots\ldots\ldots (3.30)$$

3.10 Effectiveness of SBW MCC'S on quality attributes of optimized low fat stirred yogurt flavoured with fruit puree

3.10.1 Preparation of fruit puree

Jamun (*Syzygium cumini*) belongs to Myrtacea family which is an underutilized seasonal fruit, having very attractive dark purple colour, oval shaped, fleshy fruit with a hard seed with astringent taste which was procured from the local market of Thanjavur. The fruits were washed thoroughly for the removal of the ends and other dirt's and foreign matters. Whole fruits were blanched at 90°C for inactivation of microorganism and enzymes. After removing the hard seeds from the jamun flesh and pulping was done using mixer grinder for uniform consistency. The pulp was sieved to remove the coarse skin portions. The pulp was processed along with sucrose at 85°C for 20 -30 min until it reaches the TSS of 60°B and bring down to room temperature. The jamun puree obtained was packed in and stored in airtight container under chilled condition (Turgut & Cakmakci, 2018).

3.10.2 Compositional analysis of fruit puree

The proximate estimation of developed Jamun puree was analysed for moisture, protein, fat, fibre, ash, carbohydrate, acidity and TSS by the standard methods defined in previous sections 3.1.2 and 3.9.2 of the same chapter.

3.10.3 Selection of fruit puree ratio through sensory panellist

Sensory evaluation of the samples was performed by 30 panellists using the 9-point scale for all the sensory attributes such as appearance, colour, taste, texture, aroma, aftertaste and overall acceptability. All the panel members who participated were non-smokers and familiar with milk products. Fruit puree was added in to yogurt samples with different ratio

of 0, 2, 4, 6, 8, 10 and 12% were coded as JBY7, JBY2, JBY1, JBY6, JBY4, JBY5 and JBY3 (in questionnaire) to find out the consumer preference using hedonic scale(Silva et al., 2013; Yekta & Ansari, 2019). Warm water is provided to all panellists to cleanse their palates between each samples.

3.10.3.1 Development of low fat fruit flavoured stirred yogurt (LFFSY) with addition of SBW MCC's to enhance the consumer acceptance

The figure 3.3 shows the schematic representation of preparation of LFFSY and SBW's MCC. Selected jamun crush ratio through sensory evaluation was incorporated into freshly prepared low fat set yogurt along with SBW's MCC. subsequently, the samples were stirred gently to blend with the yogurt. Afterwards, using a probe homogenizer, samples were mixed for 1min for the uniform distribution of the jamun crush along with SBW's MCC. All the samples were stored under 4°C for 48h for further analysis.

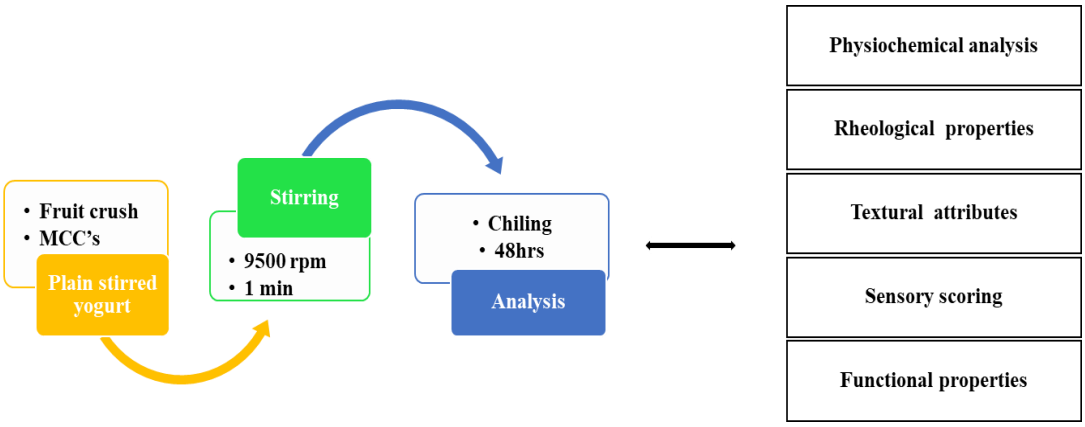


Figure 3.3 Flow chart for preparation of LFFSY incorporated with SBW MCC

3.11 Shelf life and quality of developed SBW MCC's incorporated low fat stirred fruit yogurt under refrigerated condition

The optimized low fat stirred fruit yogurt samples were subjected to low temperature storage for evaluating the effect of added SBW's MCC as stabilizer and fat mimicking agent. To find out the aforementioned, following methods were carried out such as physiochemical analysis, functional properties, rheological stability, textural parameters, microbial quality

and probiotic survival of LAB during storage. The methodology was detailed in the section 3.10.3

3.11.1 Physicochemical analysis

The compositional constituents of SBW's MCC *viz*. moisture, protein, fat, fiber, ash and carbohydrate content along with pH, TA and TSS were analysed with standard method described previously in section of 3.1.2 and 3.9.2 of this chapter.

3.11.2 Functional properties

The functional properties play an important role in the quality of yogurt products. It helps to determine the ability of the SBW MCC as stabilizer. Syneresis index, serum separation rate and water holding capacity were estimated under functional parameters. The detailed procedure of each method was explained in the section 3.10 in this same chapter.

3.11.3 Rheological parameters

The detailed methodology of the rheological parameters was explained in the following section 3.7 under this chapter.

3.11.4 Textural parameters

Textural parameters of the low fat stirred fruit yogurt were measured by using textural profile analysis with back extrusion method. The detailed methodology was discussed section 3.10.3 of this chapter.

3.11.5 Microbial quality

The microbial load of the developed low fat stirred fruit yogurt was determined by TPC, mold and yeast and E-coli count. The detailed methodology was elucidated under the section 3.10.4

3.11.6 Probiotic survival of LAB

Serial dilution and spread plate method was mainly used for the identification and microbial load of LAB in the finished products. The effective survival of the LAB gives an overall probiotic quality of the SBW MCC's incorporated low fat stirred fruit yogurt samples. This was measured by the intervals from 0, 7, 14, 28 days of storage periods. The procedure for the estimation of the LAB growth was described under section 3.10.5

3.11.7 Sensory analysis of low fat stirred fruit yogurt

Sensory evaluation was conducted with 30 participants at age of 21 – 55 which include the students and faculty members of the institute. Overall 6 treated samples were analysed and labelled with JBY, JFY, JCY, JPY, JSY and JTY. The samples were freshly prepared and stored for 3 days of fermentation in sterilized transparent bottles (100ml) at 4°C.

The coded samples were randomly arranged in sensory booth. All participants were instructed about the product nature and strictly prohibited the discussion with each other. Each time of tasting, requested to rinse their palate to avoid the human error. The 9-point hedonic scale was used for the evaluation of the treatments. Detailed methodology given in the section of 3.10.6 of the same chapter.

3.11.8 Statistical analysis

In this study, the statistical significance of pre - treatments for cellulose and MCC extraction, as well as product development and results, were assessed utilising ANOVA with Minitab 17 software (LLB, USA) and Tukey modelling via significant difference ($p<0.05$).

CHAPTER 4

RESULT AND DISCUSSION

This chapter contains the outcomes of the physiochemical characterizations of the shallot waste streams, trailed by the findings of the structural transformation of cellulose to microcrystalline cellulose and crystalline properties from selected shallot waste streams including peel, stalk and petiole. Moreover, this chapter accounts for the addition of MCC powders of different concentrations in low fat stirred yogurt and its outcome on the pre and post-fermentation factors of different blends. Also, the nutritional and sensorial effects of MCC samples incorporated low fat yogurt samples were discussed. To improve the sensory quality of yogurt, fruit puree was added and optimized. Selected SBWs MCC and fruit puree incorporated low fat stirred yogurt were further observed for the storage stability and results were tabulated.

4.1 Chemical characterisation of shallot waste streams (SBWs)

The compositional integrity and usability of shallot waste streams as raw material for further processing were determined via proximate analysis. As a result, shallot waste streams were assessed using the procedures outlined in section 3.2.2 of the materials and methods chapter.

4.1.1 Proximate characterization of shallot wastes

The untreated raw materials from Shallot bio-waste were P, S and PT (Peel, stalk and petiole) varied from part to part depending on the composition of the bio-waste. Table 4.1 shows the proximate estimation of untreated biowaste from shallot from the study by Bhosale et al., 2020, which was undertaken as part of the preliminary work for the current investigation. The carbohydrate and protein compositions of the P and S were lower than those of the PT, with 17.93, 5.18, and 4.58, 5.6%, respectively. The total ash content in P,

S, and PT, on the other hand, was relatively high, with 7.79, 15.89, and 9.21%, respectively. The increasing ash level can disrupt the chemical process that occurs during alkali hydrolysis. It is also a good indicator of contamination levels.

Table 4.1 Proximate composition of bio-waste streams from shallot.

Constituents (%)	P	S	PT	Reference
Moisture	12.28±0.29	19.25±0.43	17.19±0.18	
Carbohydrates	17.93±1.07	5.18±2.15	21.84±0.82	
Fat	2.66±0.18	4.57±0.44	2.48±0.42	(Varghese et
Protein	4.58±0.18	5.6±1.24	8.55±0.17	al., 2022)
Fibre	54.76±1.7	49.51±0.78	40.73±0.74	
Ash	7.79±0.59	15.89±0.14	9.21±0.33	

*Results are represented as mean ± SD (n=3). The terms were denoted as P-peel, S-stalk and PT-petiole.

The significant fibre concentration of the biowaste sources suggests that cellulose and MCC can be extracted for valorisation. This was supported by the high crude cellulose content of all samples. Table 4.2 shown the structural makeup of crude fibre in detail. Peel along stalk and petiole were relatively high fibre content, with 54.76, 49.51, and 40.73%, respectively (Bhosale et al., 2020).

As a result, this proximal estimation may differ from one portion of SBW to the other. In untreated SBW fibre, the peel(P) fibre had the highest cellulose content (31.41%) and the lowest lignin content (6.047%) when compared to the stalk(S) and petiole (PT) samples. The soluble fibre content was relatively high in the PT sample, as well as P and S (9.09, 8.11, and 4.29%) respectively.

Table 4.2 Structural composition of soluble and insoluble fibre in bio-waste streams from shallot

Samples	Pectin (%)	Hemicellulose (%)	Lignin (%)	Crude Cellulose (%)
P	8.11±1.83	2.57±0.85	6.047±1.13	31.41±0.53
S	4.29±0.18	6.45±0.34	13.25±0.36	28.47±0.12
PT	9.09±1.13	5.77±0.57	12.67±2.14	22.29±0.60

*Results are represented as %w/w dry basis in the form of mean ± Standard deviation(n=3)

4.1.2 Effect of extraction process on cellulose and MCC isolation

Two major hydrothermal pre-treatments (hot water & steam) used for the extraction of cellulose gives an overview of fractionation quality which assisted in selecting the best optimization condition depending upon the response parameters like yield% of cellulose, MCC, ash content and colour index as given in fig 4.1 and 4.2 (Collazo-Bigliardi et al., 2018a).

Source, pre-treatments, time, temperature, chemical hydrolysis, and cellulose yield percent all have a role in MCC extraction. In fig 4.1, the yield percent of α-cellulose was comparatively high in WP, followed by WS and WPT, with values of 34.48, 31.58, and 30.41% respectively, along with high ash content of 3.11, 3.64, and 2.74%. However, autoclave assisted pre-treated cellulose samples that were treated at 120°C for 30 min at 15psi and 2.0N NaOH showed relatively acceptable quality of cellulose yield of 29.31, 27.94, and 25.70% in AP, AS, and APT, respectively, when compared to total ash content of 0.54, 3.32, and 0.77% were showed in fig 4.2.

The ash concentration of untreated SBW is excessively high in each section, causing processing and product development problems. MCC was recovered at 21.98, 17.36, and

16.92% from AS, AP, and WPT, respectively, with reduced ash content of 0.065, 0.185, and 1.195%. While the lightness index(L*) of chosen MCC samples of C, APT, AP, and AS were considerably better than hot water pre-treatment, with 91.81, 82.4, 73.82, and 77.50, respectively. Protopectin, pigment matter, and lignin needed more time to dissolve in hot water-assisted samples.

Chemical hydrolysis will take place after the pre-treatment. The glycosidic and ether bonds of lignin and hemicellulose will be cleaved when the NaOH concentration rises. The structural deformations, stability, polymerization, and crystalline phase are also affected by the elongated NaOH penetrations. In hot water treated samples, demineralization and contaminants were significantly reduced in 2N NaOH for 60 minutes, evidenced by the low ash content (Alotabi et al., 2020; Bicu & Mustata, 2013).

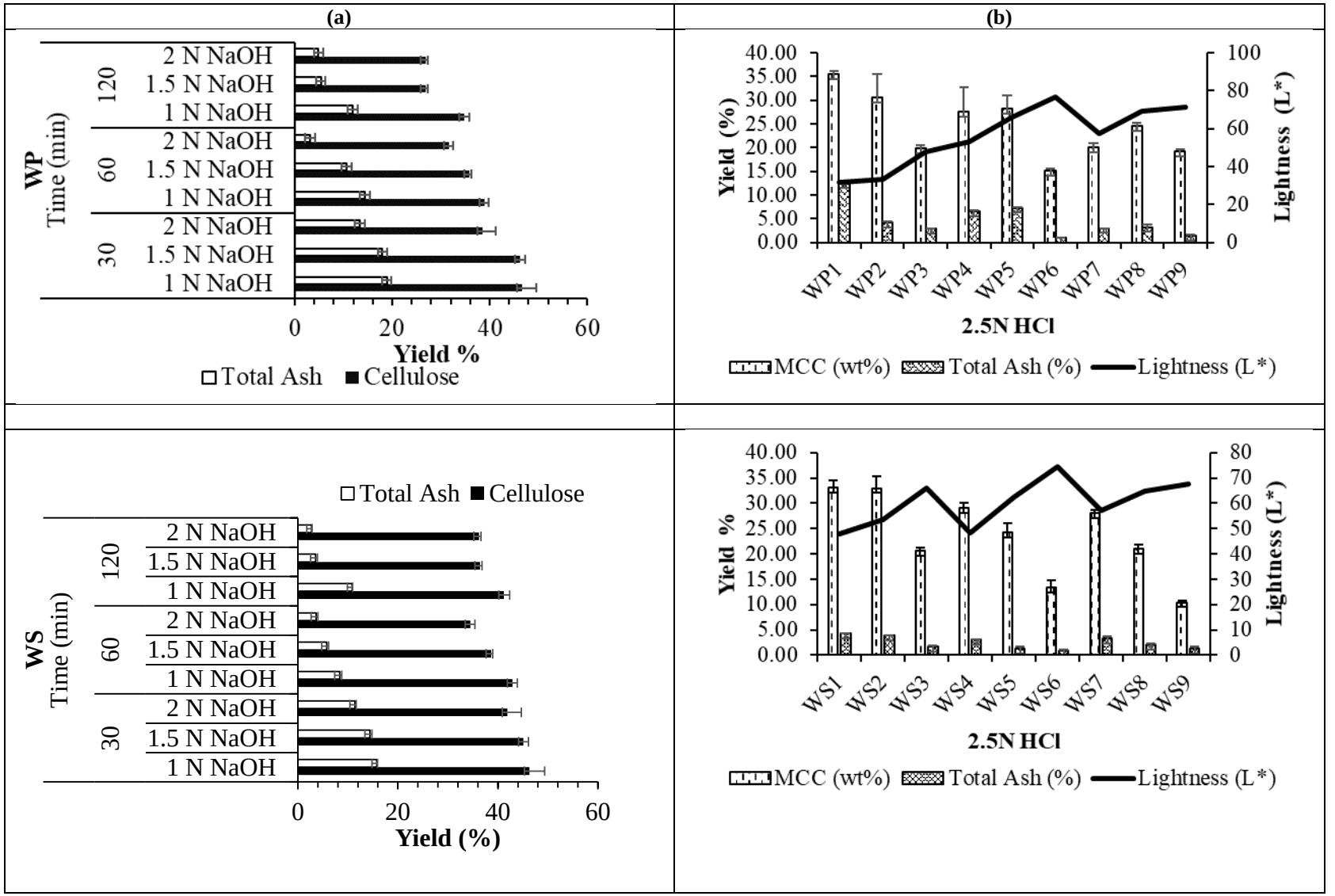
Compared to hot water pre-treatment, autoclave assisted pre-treatment activates maximum depectinisation, deliginification, and demineralization with little time consumption. These findings show that cellulose expands and loosens the structural binding of hemicellulose and lignin prior to autoclave pre-treatment.

Because of the porous surface in autoclaved samples that dissolve the lignin and cleave ester bonds of pectin to generate a complex that dissolution more quickly than the hot water approach, the alkali penetration may be uniform and faster during the NaOH hydrolysis.

In the bleaching process, sodium hypochlorite and acetic acid interaction at acidic conditions obliterates lignin and dissolves polyphenols and other pigments from the

cellulose surface, resulting in white powder. However, cellulose's colour fades to yellowish, affecting the cellulose's properties due to the lengthier bleaching process exposure seen in hot water treated samples (Jacquet et al., 2015).

As the temperature rises to 190°C due to steam explosion, cellulose is degraded into glucose, resulting in the breakdown of the glycosidic bond of the linear chain and an increase in ash content (Hu & Ragauskas, 2012)


(a)
(b)
WP
Time (min)
120
60
30
2 N NaOH
1.5 N NaOH
1 N NaOH
0
20
40
60
Yield %
Total Ash
Cellulose
Yield (%)
Lightness (L*)
WP1
WP2
WP3
WP4
WP5
WP6
WP7
WP8
WP9
2.5N HCl
MCC (wt%)
Total Ash (%)
Lightness (L*)
WS
Time (min)
Yield (%)
WS1
WS2
WS3
WS4
WS5
WS6
WS7
WS8
WS9

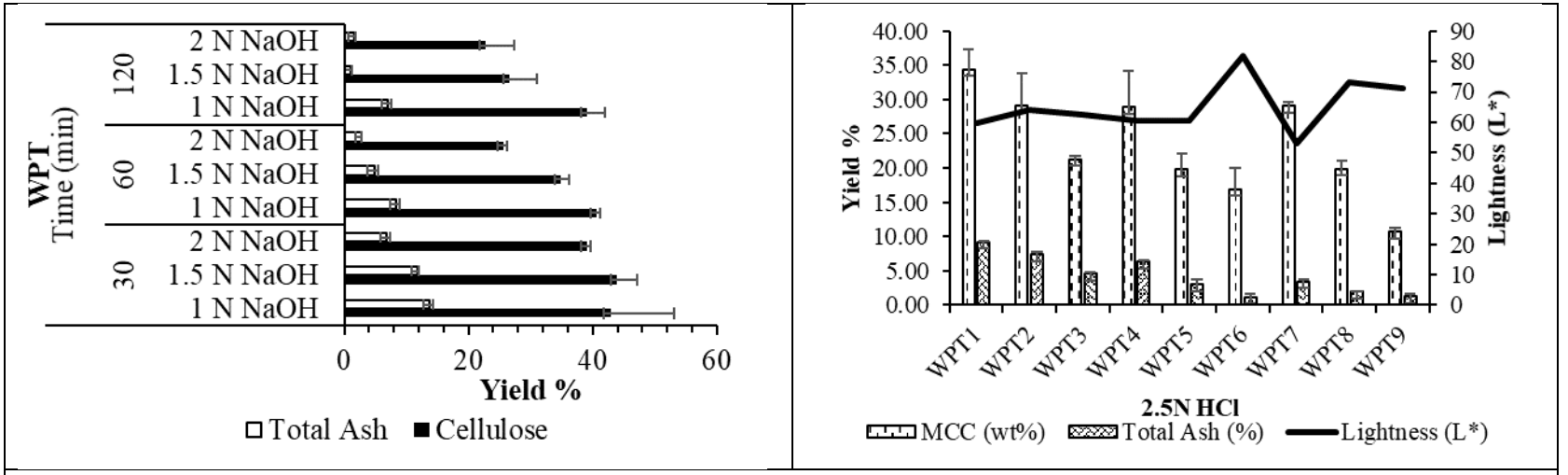


Fig. 4.1 Variations of time, temperature and concentration of NaOH factors convoluted in extraction of cellulose with respect to (a) ash content and (b) MCC yield versus ash content and lightness(L*) index through 2.5N HCl hydrolysis from shallot wastes pre-treated with hot water assistance. *Results were expressed as mean ± SD (n=3). *Note : Hot water assisted extracted MCC samples from peel, stalk and petiole were denoted as WP, WS and WPT

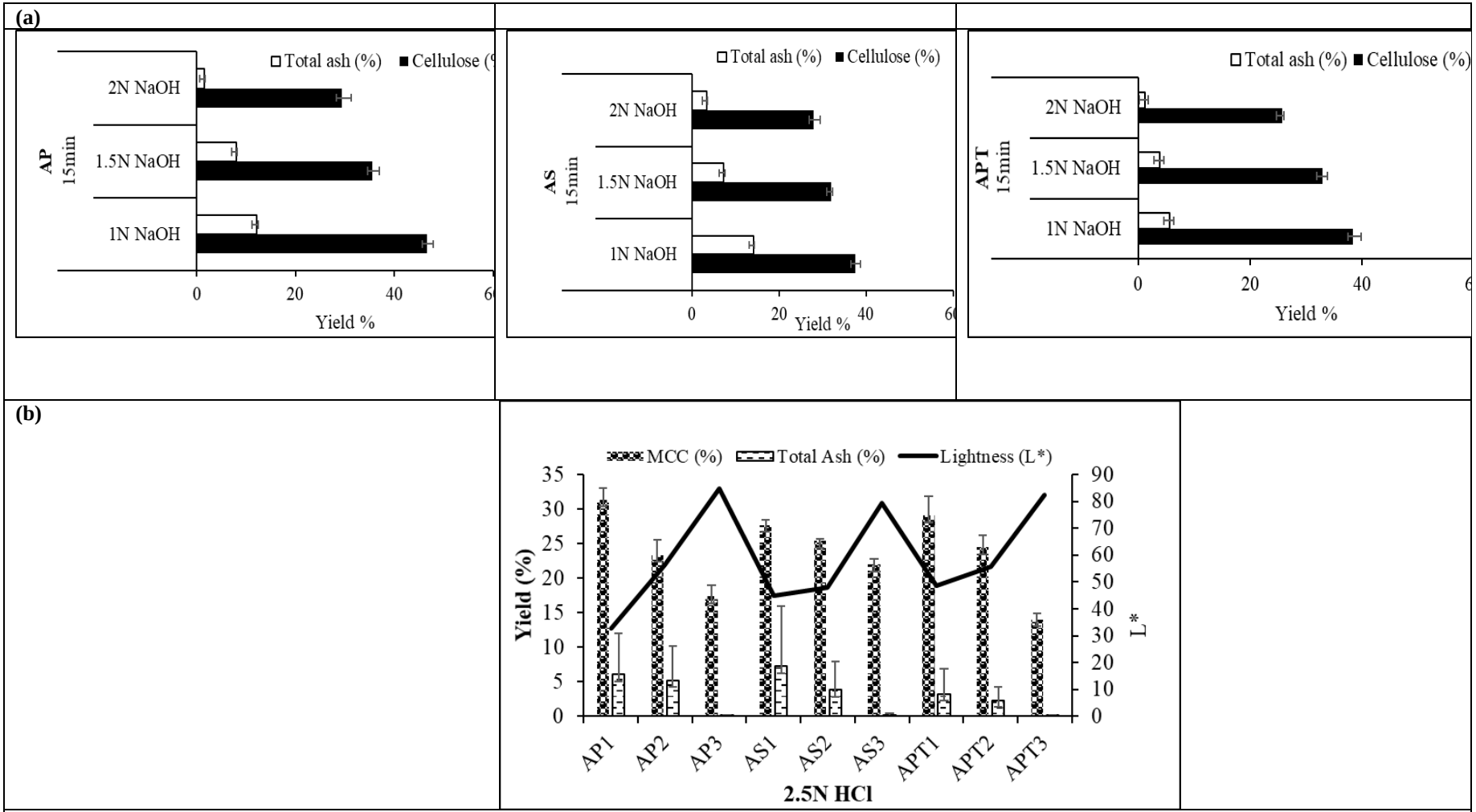


Fig. 4.2 Variation of different concentration of NaOH (a) on yield of cellulose with respect to ash content and (b) MCC versus ash content and lightness(L*) through acid hydrolysis from shallot wastes pre-treated with pressurised steam assistance. (*Results were expressed as mean ± SD (n=3). *Note: Auto clave assisted extracted MCC samples from peel, stalk and petiole were denoted as AP, AS and APT

Fig. 4.3. reveals the MCC yield percent of the selected optimal condition of hot water and steam assisted pre-treatments Overall, MCC recovery was relatively high in AS, AP, and WPT, with 79.39, 59.23, and 55.63%, respectively. Acid hydrolysis is a critical stage in the synthesis of MCC. During acid hydrolysis, linear higher degree cellulose polymers are broken down to smaller degree polymers, resulting in the removal of an amorphous region with a greater specific surface area than commercial cellulose. The reaction begins with a vigorous interaction of 2.5N HCl with extracted cellulose at 100°C. At high temperatures, increasing concentrations of HCl will penetrate the amorphous zone of cellulose where Cl has weakened the glycosidic linkage, and H+ react with β-1,4-glycosidic bonding and breaks into a shorter chain MCC. The extent of pre-treatments and alkali and bleaching hydrolysis will also affect the HCl hydrolysis, such as yield, DP, crystallinity, and MCC colour characteristics.

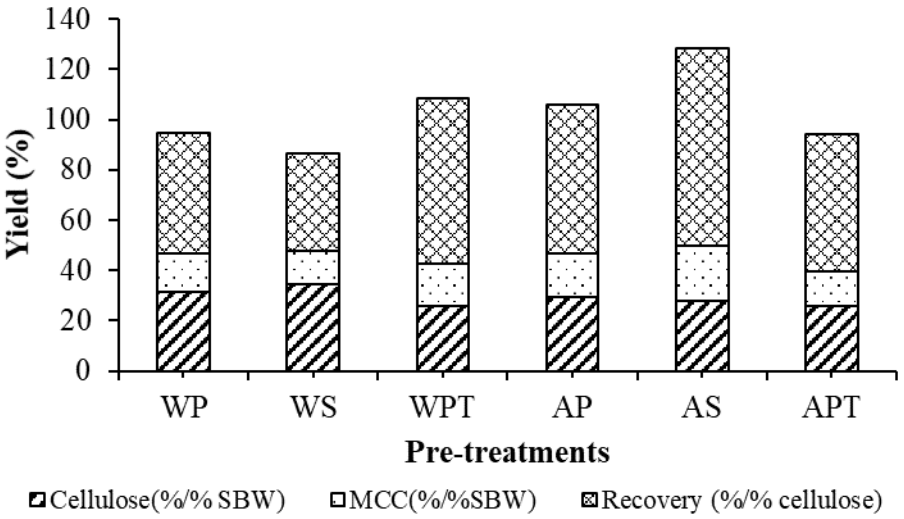


Fig. 4.3 Recovery of MCC from pre-treated cellulose extracted from SBW. * Note: i) Hot water assisted extracted samples from peel, stalk and petiole were denoted as WP, WS and WPT. ii) Auto clave assisted extracted MCC samples from peel, stalk and petiole were denoted as AP, AS and APT. Results are represented in the form of mean ± Standard deviation(n=3)

4.2 Morphological characterization of Microcrystalline cellulose

4.2.1 FT-IR spectra

The FT-IR spectra are shown in figure 4.3, together with the primary and minor consequent peaks in spectral pictures A and B. The discrete spectrum of polysaccharides was discovered in 1800-650 cm^{-1}, representing the structural confirmation of the presence or absence of pectin, hemicellulose, and cellulose. The intra and intermolecular interaction between cellulose resulted in a widened peak bending in all pre-treated MCC samples in the range of 3500-3000cm-1 measured O-H absorption. The modest peak stretching at 1740 cm^{-1} was displaced to 1735 and 1720 cm^{-1} in hot water pre-treated samples due to the partial elimination of hemicellulose amount by alkali and acid hydrolysis, whereas autoclave pre-treated samples did not show this significant peak.

Furthermore, pectin and its derivatives assigned 1615, 1264, 1043, 1093, and 1091 cm^{-1} peaks to mild asymmetric stretching of C-O and C-H_2 (methyl groups). The C-O vibration of pectic derivative acid was seen strongly in both WP and WS. However, this band disappeared in autoclave assisted pre-treated samples. Finally, following NaOH and chlorite hydrolysis eliminated, the aromatic ring structure with C=C stretch vibration of lignin in 1520-1510 cm-1 autoclave pre-treated samples. C=C stretching of lignin traces was discovered in WPT and WS MCC samples. The autoclave entirely stopped the absorption spectra of hemicellulose and lignin and helped pre-treatment, and chemical hydrolysis in autoclave assisted samples, 1734-1720, 1615, 1596, 1512, 1520,1344, 1269, 1244, 1094, 1091, and 1043cm^{-1}. In prior research (Kunusa et al., 2018), the absence of C=O in the region of 1510, 1523, and 1413 cm^{-1} peaks stretching was primarily due to the aromatic ring of lignin, which demonstrated the degradation of lignin. In several agro-industrial wastes, such as tea waste, the highest characteristic band discovered between 1430-1429, 1364, 1157, and

895cm-1 showed a strong peak bending symmetric CH2 and asymmetric C1-H denotes a pure crystalline cellulose band (Gao et al., 2017).

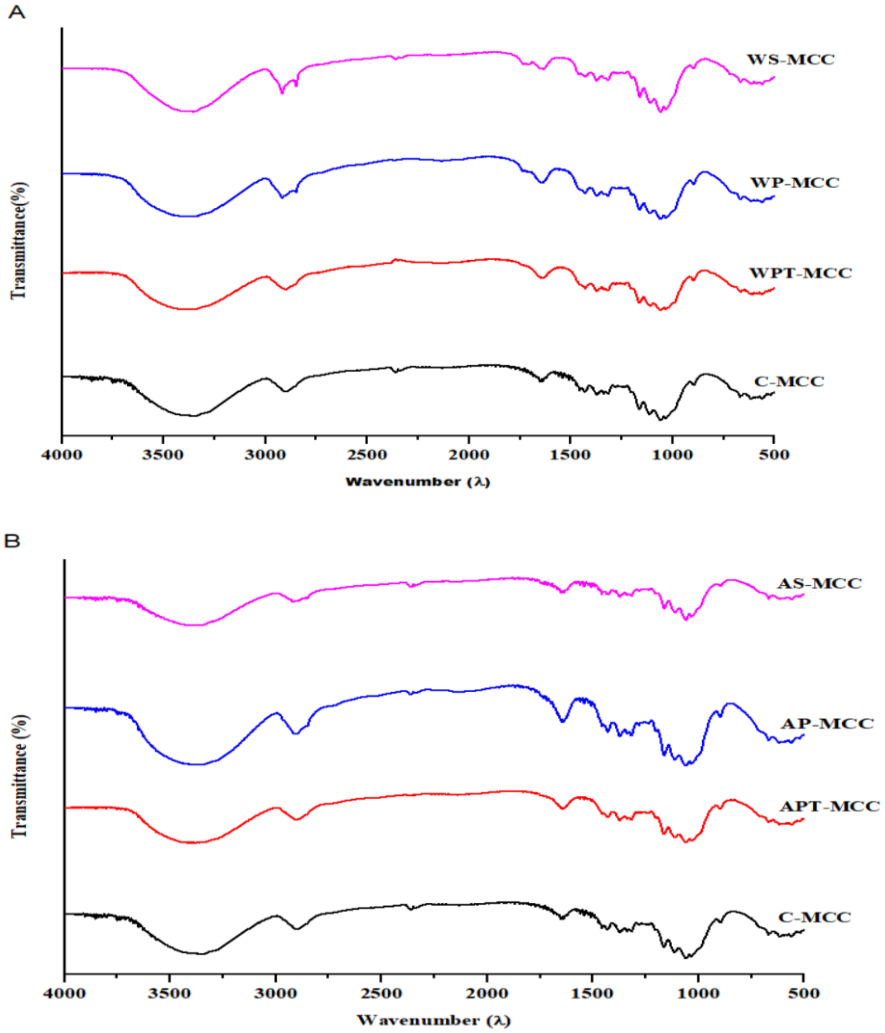


Figure 4.4 FTIR spectrum of MCC with different pre-treatments: A) hot water and B) autoclave assisted isolated MCC samples.

The previously indicated minute peak stretching at 900-895 cm^{-1} was observed in all MCC samples, with varied sharpness in peak strength dependent on the extent of process conditions. Overall, MCC samples isolated from autoclave assisted pre-treatments revealed a significant reduction in peak number with higher spectrum resolution when compared to MCC samples isolated from hot water aided pre-treatments (Bhandari, Adaval, et al., 2020).

Table 4.3 explains the shifting of peak bands and the presence or absence of functional groups in each MCC sample ranging from 4000-400cm^{-1}. Almost all MCC samples extracted using the autoclave aided approach had FTIR spectra that were similar to commercial MCC, with a modest shift in peak locations depending on the length of time, temperature, pre-treatments, and chemical hydrolysis. Following characterization techniques support to give deep insight on morphological alterations occurred in isolated MCC samples using scanning electron microscope.

Table 4.3 Spectral data of autoclave treated and water treated samples of SBW from FT-IR interpretation

Peaks information (Wavenumber in cm^{-1})							**Functional groups**
C-MCC	**WP**	**WS**	**WPT**	**AP**	**AS**	**APT**	
3853-3346	3354	3355	3384	3355	3853-3384	3853	O-H Alcoholic group wide broadened peak
2900	2917	2917-2849	2900	2915	2914	2901	C-H Saturated aliphatic ring in crystalline Phase
2400-2300	2400-2300	2400-2300	-	2400-2300	2400-2300	-	OH, C-H_2, Intermolecular hydrogen seen in uronic acid and ring linkage
-	1734	1720	1720	-	-	-	C=O, Carboxylic ester group or due to the ketone group in hemicellulose
-	1706	1706	1706	-	-	-	C=O, Derived pectin of ferulic or P-coumaric components

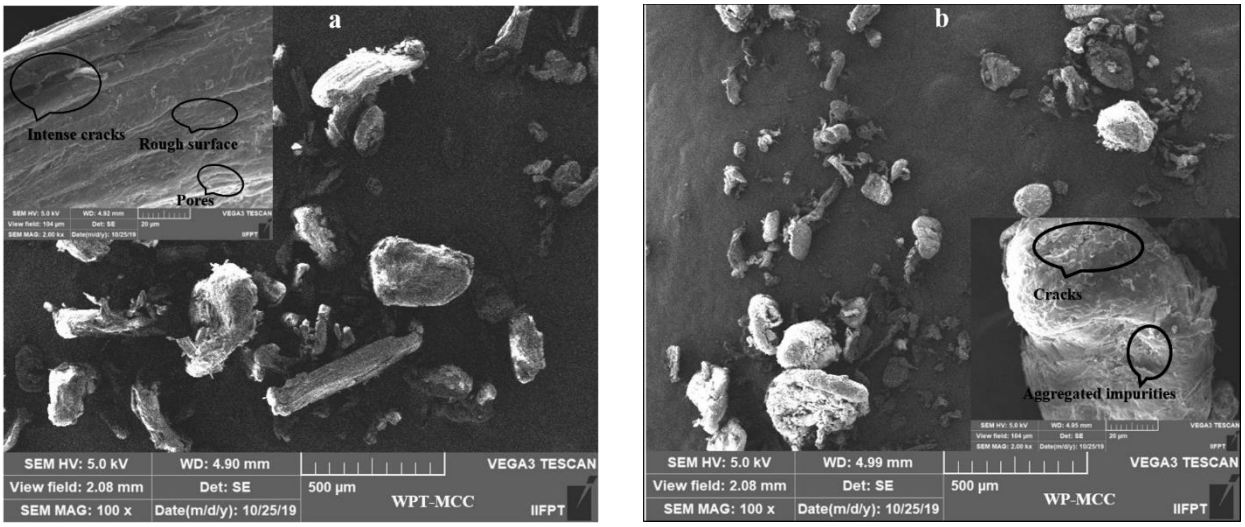
1647	1647	1636	1636	1636	1647	1647	O-H Interconnection between cellulose and adsorbed water molecules
1540	-	1540	1541	-	-	-	C-NH_2 Characterized band bending of protein especially amide bond formation
1430	1430	-	1429	1429	1430	1430	C-H_2, C-O-C Crystalline band of symmetric bending of MCC due to the intermolecular link formation in C6 and pyranose ring
-	1312	1312	1312	-	-	-	OH, C-O, C-O-C Minor bending of hemicellulose or aryl group of lignin
1374	1373	1373	1371	1373	1374	1373	C-H Asymmetrical stretching in cellulose
1164	1162	1161	1163	1162	1162	1163	C-O-C Characteristic asymmetrical vibration of bonding in or stretching of β-(1,4) glycosidic linkage
1113	1112	-	-	1113	1112	1112	C-O-C Pyranose ring skeleton stretching in cellulose structure
1059	1060	1061	1061	1060	1060	1061	C-O, C-H, C-OH, Side chain groups of native cellulose
-	1032	1032	1032	-	-	-	C-O Characteristic peaks of cellulose
-	975	975	975	-	-	-	C-OH Functional group stretching of pectin components

899	897	904	898	899	886	896	C-O-C, -C_1-H Sharp stretching of β-(1,4)-glycosidic linkage between sugar units of cellulose. (Distinctive peak displays the purity of the MCC)

*** Note: i) Hot water assisted extracted samples from peel, stalk and petiole were denoted as WP, WS and WPT. ii) Auto clave assisted extracted MCC samples from peel, stalk and petiole were denoted as AP, AS and APT. iii) commercial microcrystalline cellulose standard: C-MCC standard**

4.2.2 SEM and particle size distribution

Figure 4.5 depicts the structural features of extracted MCC after hot water and autoclave pre-treatments and C-MCC through SEM micrographs. The selected 100X micrographs and the magnified 2000X micrographs were depicted with black arrows (and forms) labelled as a, b, c, d, e, f, and g of each MCC sample.



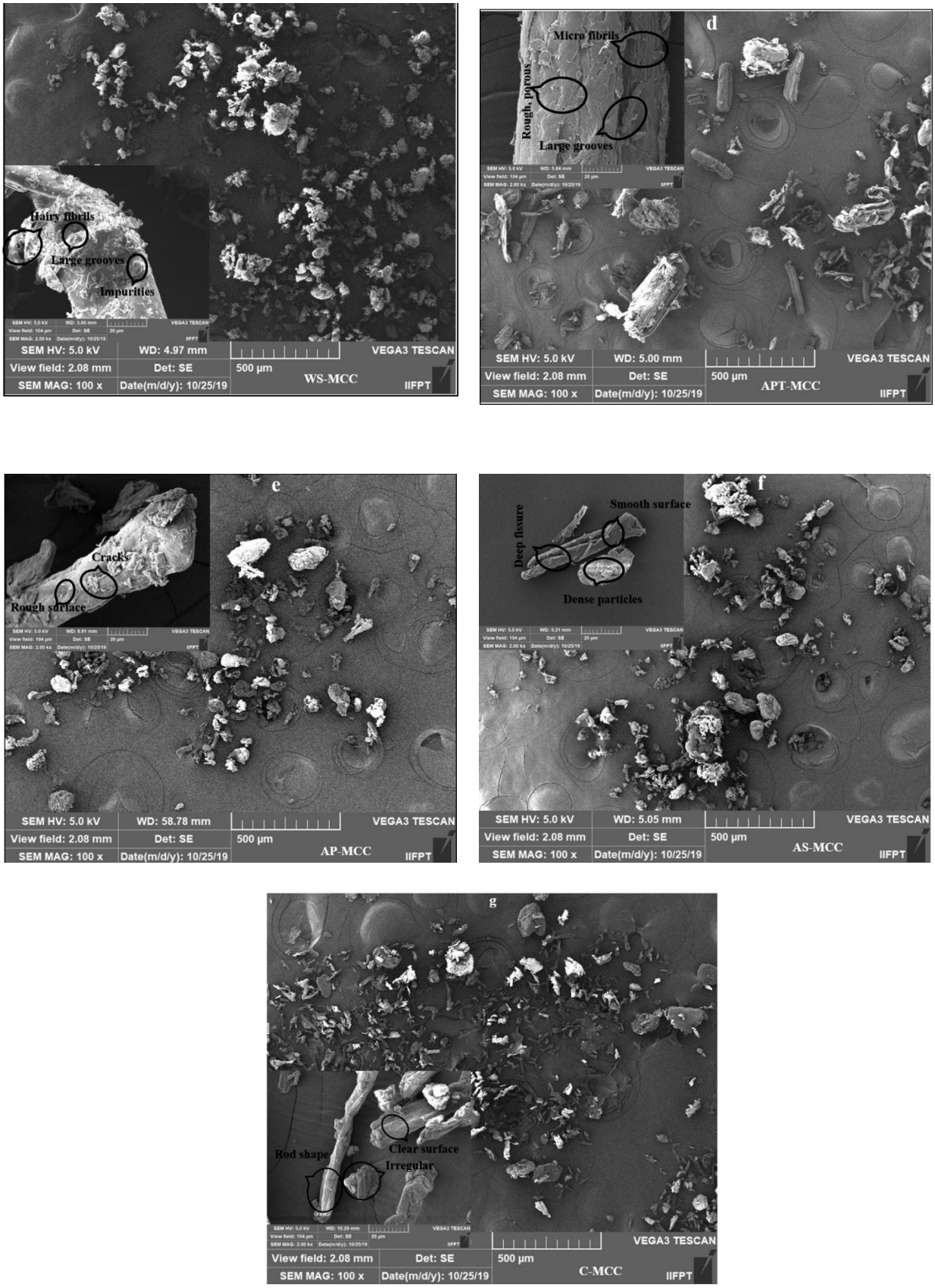


Fig. 4.5 SEM micrographs of extracted MCC from shallot biowastes a) WPT-MCC, b) WP-MCC, c) WS-MCC, d) APT-MCC, e) AP-MCC, f) AS-MCC and g) C-MCC

Induced pre-treatment and chemical hydrolysis are critical in converting cellulose to MCC. Compared to hot water treatment, autoclave-assisted pre-treated samples had well-defined cleavage, porous, and smooth surfaces (Luo et al., 2018). In both the MCC samples of WPT and APT, the petiole section exhibited in (a) and (d) micrographs has separate rod-shaped structures. The structural damages in MCC samples of WPT were discovered to be increased due to the prolonged reaction time, temperature, and chemical penetration. However, non-cellulosic components were eliminated and converted cellulose fibrils to individual short microfibrils of MCC by acid hydrolysis (Xu et al., 2019).

Because of the longer chemical hydrolysis, hot water pre-treated samples revealed more damage and irregularity. While the surface of the APT MCC samples grew more porous due to the elimination of amorphous regions by acid hydrolysis, the crystalline structures remained more robust. They maintained their slender, smooth, and rod-like characteristics in each particle. The autoclave-treated samples of e and f (AP & AS) grew more enlarged and smooth-surfaced, with transparent and fibrous irregular-shaped structures that resembled those of the g sample. The particle size and agglomerations are determined mainly by the composition of the raw material, pre-treatments, and hydrolysis methods. The longer the reaction period, the greater the agglomerates and, as a result, the particle's surface area and interior area in microfibrils (Lin et al., 2019).

Histograms of MCC samples displayed the particle size distribution in Annexure 5(a), (b), and (c). The mean particle size varies amongst samples, ranging from 16.14 to 36.34μm. Compared to the C-MCC (17.56), AS, APT, and WS had mean average particle diameters of 16.14, 21.82, and 24.84 μm, as shown in fig. 4.5. Similar research on rice husk and soya husk MCC revealed particle diameters of 255-342 and 396-615μm, respectively (Owolabi et al., 2017). The average L/D ratio of AP-MCC samples was 1.366, indicating that they are nearly spherical. The L/D ratio for APT and AS was determined to be 2.591

and 2.106µm, respectively. These ratios imply that the length is twice as long as the diameter, indicating that the particles are more longitudinal. This data is supported by the comparable pattern demonstrated by SEM images of the corresponding samples in fig 4.6.

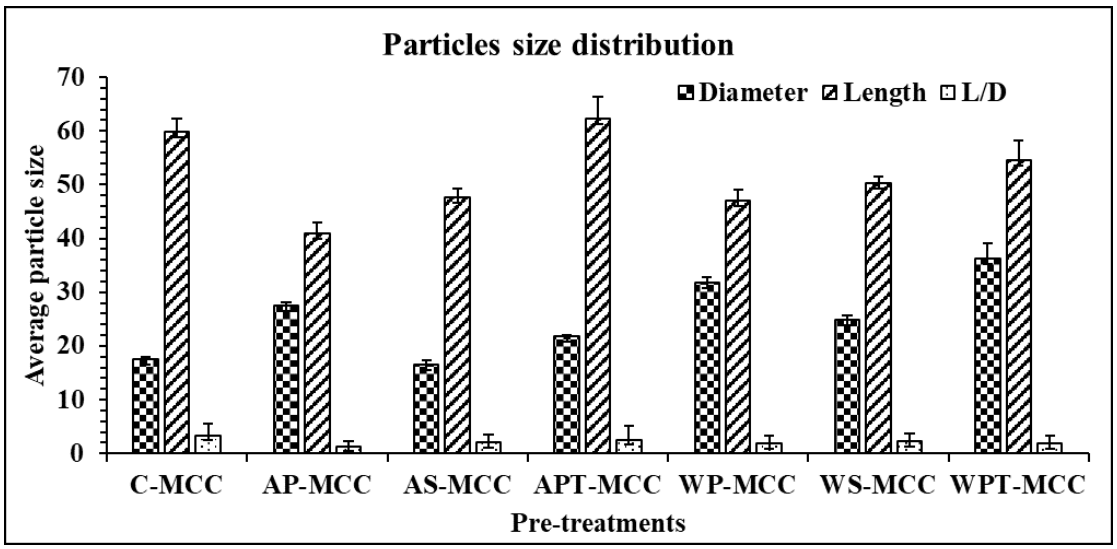


Fig. 4.6 Mean particle size distribution of extracted MCC from SBW (*Average mean calculated by the particle size (Diameter, Length and L/D ratio) measured at 100 particles from SEM image with standard error (n=100))

4.2.3 Crystalline characterization by X-ray diffraction (XRD) analysis

In extracted MCC samples, the increase or reduction in crystalline Index is primarily determined by variables such as process condition, structural alteration, and compositional deformation. The diffractograms of isolated MCC samples shown in Fig. 4.7 exhibit the patterns of the hot water assisted and autoclave assisted extraction processes, respectively.

The major three peaks of the C, WP, AP, and AS MCC samples that appeared at 2=15°, 22°, and 34° were connected to the crystallographic planes of 110, 200, and 040. Lattice displacement occurred primarily in planes of 200 where 2θ= 21.53° to 22.6° was reported in Alfa fibres. Previous studies on sago seed shell (Naduparambath &

Purushothaman, 2016)and sorghum stalks (Ren et al., 2019)revealed a comparable major plane in 110, 200, 040 with 2θ=16°, 22.8°, and 34.5°.

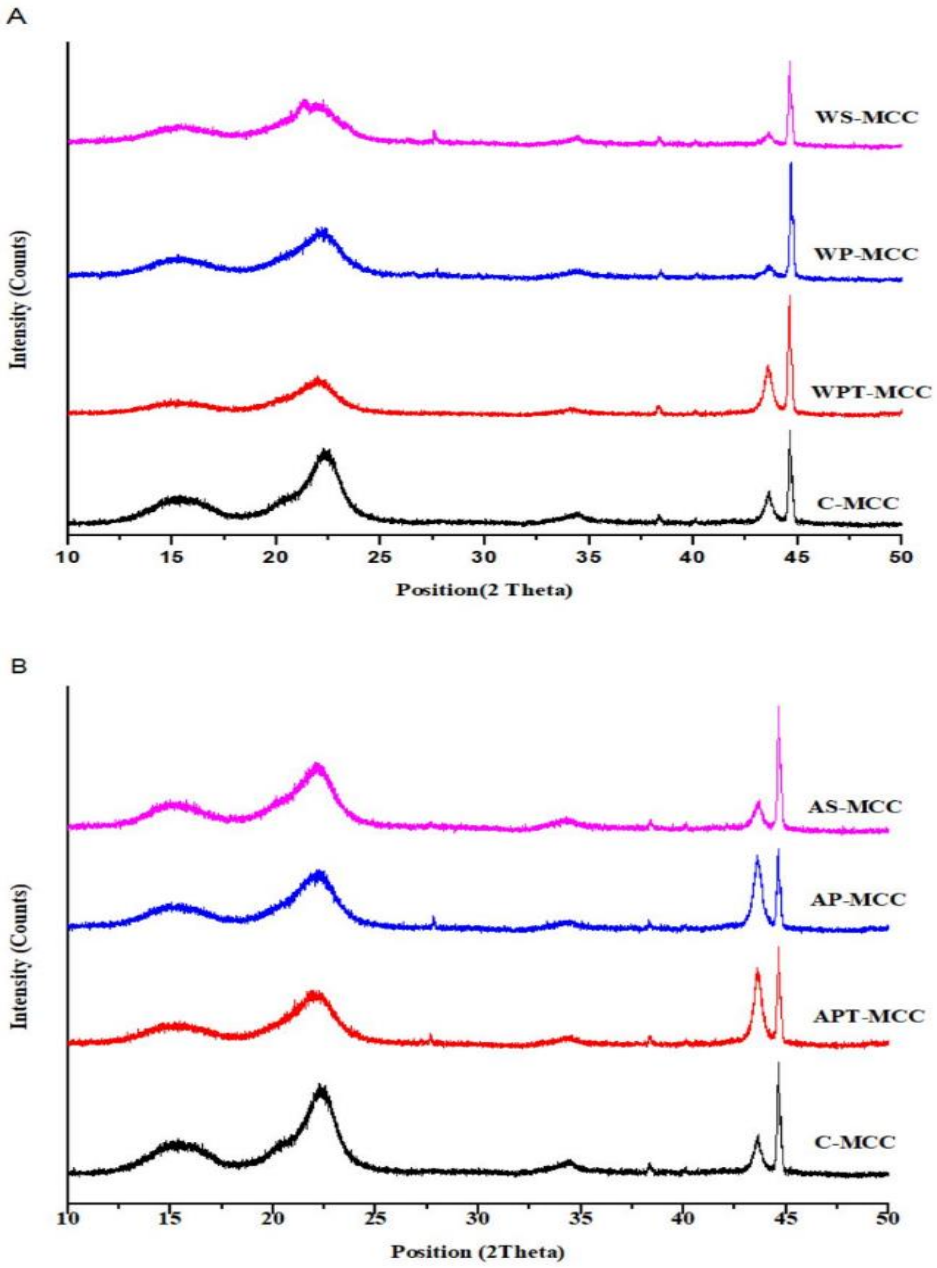


Fig. 6 X-ray diffraction patterns exhibited by MCC isolated from different pre-treatments i) hot water and ii) autoclave assisted samples.*Note: i) Hot water assisted extracted samples from the peel, stalk, and petiole were indicated as WP, WS, and WPT ii. Autoclave assisted extracted MCC samples from peel, stalk, and petiole were labelled as AP, AS, and APT. iii) commercial microcrystalline cellulose standard (C-MCC)

Pre-treatments and chemical hydrolysis during MCC extraction trigger hydrolytic disruption of the β-1,4-glucopyranose bond, releasing individual crystallites from cellulose

fibres and reducing amorphous components. This causes an increase for CI in isolated MCC samples. This had occurred in the MCC samples of WPT and APT, where the position of the same peak had moved to 2θ=14.9° and 21.9°, respectively. The amorphous peaks of monoclinic cellulose I are represented by the peaks seen in 2θ=15°, 14.9°. The presence of crystalline cellulose II, which is coupled by Vander Vaal force and can break owing to excess energy, is confirmed by the largest peak investigated at 2θ= 21, 22°.

The doublet peak in the plane of 200 and 200 at 2θ=21.3 and 22.2° was generated by cellulose I and cellulose II allomorphs in a WS MCC sample (Fawcett et al., 2013). These could possibly be the result of swelling converting cellulose I to cellulose II or alkali hydrolysis renewing cellulose. Researchers discovered the same similarity in oxy-MCC isolated from rice husk, which also discovered doublet peaks at 2θ=20°, 22°, which related to the co-occurrence of cellulose I and II, which transforms to a unique hybrid MCC structure with 84.8% CI (Ahmed-Haras et al., 2020).

Isolated MCC exhibits two distinct peaks at 2θ=43° and 44° that could indicate additional crystal lattices in all pre-treated samples. The formation of these peaks may be related to the formation of new crystalline planes atomic interfaces caused by the breakdown of cellulose I.

The higher degree of crystallinity of standard MCC was typically between 55 and 80 percent. Using the peak intensity technique in Eqn. (3.13**)**, CI was significantly higher in C, APT, AS, and WPT, with 78.28, 79.93, 76.75, and 83.74%, respectively. When compared to the C-MCC (77.55%), APT, AS and WP have excellent CI of 83.68, 82.50, and 81.53 %, respectively, using the peak area or convulsion approach via Eqn (3.14). Similarly, the CI of waste sources such as cotton, sugar bagasse, groundnut husk, corncob, rice husk, palm

stalk, and palm spikelet is 84.46, 87.4, 64, 78, 68.46, 76.2, and 81.3% (Bhandari, Roy Maulik, et al., 2020a; Shao et al., 2020).

The size of the crystallite is also an essential component that is related to the CI. It is determined using Scherrer's Eqn (3.15) and is shown in table 4.4 elucidates the average crystallite size in crystallographic planes of extracted MCC cellulose I and II. The CI and crystallite size of the autoclave assisted pre-treated samples performed better than C-MCC. Because of differences in source and nature, pre-treatments, the level of NaOH and bleaching agent interaction, HCl hydrolysis, reaction time and temperature, and extraction process, the crystallite size in extracted MCC samples was non-uniform (Kunusa et al., 2018).Thus the present study achieved higher crystalline cellulose can be extracted from the shallot bio-waste while comparing with other source of agro-industrial bio-waste. The thermal resistances of isolated MCC samples were inspected through DSC analysis.

Table 4.4 Crystallinity index and crystalline size of extracted MCC

Samples	CI (%)		Average Crystallite Size D (nm)
	Peak intensity (Segal formula)	Peak Area (Deconvolution method)	
C-MCC	78.28	77.55	24.93±10.75
WP-MCC	57.10	81.54	82.81±47.33
WS-MCC	54.67	72.39	31.05±8.88
WPT-MCC	83.74	77.98	46.28±18.64
AP-MCC	44.98	73.15	28.54±11
AS-MCC	76.75	82.50	57.43±25.24
APT-MCC	79.93	83.68	33.23±12.92

***Note: i) Hot water assisted extracted MCC sample from the peel, stalk, and petiole were indicated as WP, WS, and WPT. ii) Autoclave assisted extracted MCC samples from peel, stalk, and petiole were labelled as AP, AS, and APT. iii) commercial microcrystalline cellulose standard (C-MCC)**

Table 4.5 Crystallite size, d-spacing and micro strain in planes of major peaks (110, 200, 040) in X-ray diffraction of MCC extracted from shallot bio-waste

Crystalline properties	C-MCC			WP-MCC			WS-MCC				WPT-MCC			AP-MCC			AS-MCC			APT-MCC		
Planes	**110**	**200**	**040**	**110**	**200**	**040**	**110**	**$\bar{2}$00**	**200**	**040**	**110**	**200**	**040**	**110**	**200**	**040**	**110**	**200**	**040**	**110**	**200**	**040**
D (nm)	3.10	4.79	21.05	3.95	4.40	242.90	3.23	8.75	7.90	34.89	18.41	4.49	101.5	3.10	4.97	21.04	3.54	4.16	123.80	2.96	3.97	53.3
d-spacing (Å)	0.11	0.15	0.23	0.15	0.23	0.23	0.10	0.14	0.15	0.23	0.10	1.47	0.23	0.10	0.15	0.23	0.11	0.15	0.23	0.10	0.15	0.23
Dislocation density (δ in $nm^{-}2$)	33.56	9.11	0.10	64.20	51.67	0.02	95.96	13.07	16.02	0.82	2.85	49.71	0.10	32.27	20.89	4.75	80.02	62.25	0.07	11.38	6.83	0.01
Micro-strain (ε)	1.50	1.40	0.49	1.16	1.52	0.04	1.44	0.73	0.85	0.29	0.25	1.53	0.11	1.50	1.40	0.49	1.35	1.65	0.08	1.57	1.73	0.06

4.2.4 Thermal characteristics by DSC method

The DSC thermogram of the extracted MCC samples was shown in annexure 6(b) revealed two distinct endothermic peaks in various temperatures of differently pre-treated samples. The endothermic peaks were generated as a result of thermal decomposition's volatilization and charring (Amaro et al., 2019). The changes that occurred in MCC during heat breakdown were caused by moisture loss, intermolecular hydrogen bonding damage between the monomers, and melting of crystals present in the sample.(El-sayed et al., 2011; Trilokesh & Uppuluri, 2019; Yang et al., 2007).

The presence of crystals in current samples resists heat degradation depending on the nature of cellulose I and II. The DSC thermographs of the extracted samples shown in table 4.6 revealed that the first endothermic peak event occurred in extracted MCC from different samples between 80 and 145°. MCC samples of AS, C, and WS had the greatest temperatures in the first endothermic peak, with 144, 123.3, and 121°C, respectively. Following that, a second event observed at temperatures ranging from 200 to 300°C resulted in the extensive disintegration of crystalline cellulose.

The MCC samples of AS, AP, and WPT had the maximum thermal resistance in this event, with 263, 259, and 255, respectively. Prior research on biomass from wheat, soybean, banana, and rice revealed TM values of 296, 290, 301, and 268°C, respectively, corroborating the above findings (Chai et al., 2018; Cheng et al., 2017; Ibrahim et al., 2013; Jahan et al., 2011; Sukaimi et al., 2019). When MCC samples extracted from SBW were compared to control samples, the Hf was lower. Lower ΔHf was seen in the AP, WPT, and APT of 16.49, 25.82, and 47.47 Jg^{-2} respectively.

Thus, the result showed satisfactory thermal stability as well as the purity of extracted MCC samples as compared to the control (C) MCC sample. Also the source of the

raw material, hydrothermal pre-treatments and alkali-acid hydrolysis affect the thermal stability of the extracted MCC samples from shallot biowaste.

Table 4.6 DSC data tabulated from endotherm curves of MCC extracted from SBW

Sample	ΔH_f (Jg^{-2})	T_M (°C)			
		T_0	T_P	T_{M1}	T_{M2}
C-MCC	103.24	91.79	122.39	122.39	-
WP-MCC	62.44	56.03	81.46	82.6	-
WS-MCC	47.50	99.69	123.22	123.33	228
WPT-MCC	25.82	97.03	115.97	117	255.89
AP-MCC	16.49	67.82	88.46	87	259.3
AS-MCC	92.29	121	143.58	144	263
APT-MCC	47.47	97.09	121.87	150.48	251

*Terms denoted as ΔH_f= enthalpy of decomposition, T_M= melting temperature, T_0=onset temperature, TP= peak maximum temperature, T_{M1} & T_{M2}= endothermic melting curves, and Tg= glass transition temperature. *Note: i) WP, WS, and WPT denote as hot water assisted extracted samples from peel, stalk, and petiole, respectively. ii) Autoclave-assisted extracted MCC samples from peel, stalk, and petiole were denoted as AP, AS, and APT, respectively. C-MCC is a commercial microcrystalline cellulose standard

4.3 Techno functionality of extracted SBW's cellulose and microcrystalline cellulose

Throughout the extraction of SBW MCCs from different parts of the shallot, pre-treatments and chemical hydrolysis of parent cellulose cause massive structural modifications that impact its characteristic properties. The properties of modified cellulose can be found out by analysing the flowability, hydration and foaming capacities. It will give the requirements involved as an ingredient or additive for the development of food. The following studies were discussed in this section to help perceive the diverse applications of isolated SBWs MCC samples in various fields. The suitability of extracted MCCs as ingredients or additives in various fields of the food system can be validated by these techno functionalities.

4.3.1 Powder density, porosity and flow features

Bulk density is defined as the ratio of mass and volume due to the presence of inter-spatial voids between the particles. The powder densities of extracted SBW's cellulose was showed in Table 4.7. The BD of extracted SBW cellulose samples showed significant decrease as compared to the commercial cellulose samples. APTC and APC showed lowest values ($p \leq 0.05$) of bulk density as compared to commercial samples with 0.143±0.003 and 0.224±0.01 respectively.

When MCC samples were considered, bulk density got increased due to the morphological modification during the process of extraction pre-treatments, acid hydrolysis, size reduction and drying. As compared to CM, the least and highest bulk density was exhibited for WSM and APTM with values of 0.316±0.032 and 0.246±0.005 respectively exhibited in Table 4.8. In the case of extracted MCC from SBW cellulose, significantly higher bulk density was observed as compared CM and CC. Also, pre-treatment, extraction and milling processes exhibits more bulkiness and

flowability in all the extracted samples, especially in steam-treated samples. Increased bulk density implied increased particle size, void space and denser structure resulting out of increased compression force for the tableting process. However, lower values of bulk density resulted in better dilution potential reliant on a weight basis. As bulk density decreases, free flowability of powder increase and can have a profound effect on the tableting property. In general, conversion of MCC from cellulose showed incremental bulk density values due to the increase in density of powder particles and the spatial arrangement of particles in the powder bed.

True density of extracted SBW cellulose and MCCs showed no significant difference with CC and CM samples. In case of MCC samples, true density was slightly lower than CC of WPTC and APTC samples with 1.303 and 1.341g/cm^3 respectively. Moreover, in extracted MCCs, values were significantly higher than CC and CM. The WPTM and APM showed increased true density with values of 1.575 and 1.556 g/cm^3 respectively. Consequently, higher the true density, the more excellent will be the compressibility properties of MCC samples as compared to cellulose samples (Amin et al., 2014). Also, it is indirectly proportional to the porosity of powdered samples. For higher bulk density of the sample, lower will be porosity values and vice versa (Y. X. Wang et al., 2021).

Tapped density analyses the packing capability of a powder sample in a confined space throughout the transport process. As compared to CC, cellulose samples showed a slight increment in tapping density. In cellulose samples, WSC and ASC showed better tapping density with values of 0.539±0.001 and 0.525±0.01 respectively. While in pre-treated MCC samples, increased tapped density was observed due to the structural modification that resulted in increased particle size along with friction between particles (Kurek et al., 2018). Hot water treated samples

showed increase in tapping density than autoclaved pre-treated samples (Azubuike & Okhamafe, 2012; Jiang et al., 2020). Suitability of the powdered cellulose and MCC for food applications can be determined by measuring the densities to confirm its food applicability where lower densities make them suitable for usage in infant food, meat, bakery products, beverages and instant soups (Jiang et al., 2020). Similarly, increased values of bulk, true and tapped density are directly correlated to the excellent potential inflow properties and can be rearranged during compression force applied in the development of tablet preparation (Azubuike & Okhamafe, 2012).

4.3.1.1 Hausner's ratio and carr's index

Carr's index and Hausner's index were determined to find out the compressibility and cohesiveness to quantify the flowability of powder samples. The extracted cellulose from SBW streams showed a lower Carr's index than CC. The CC, APC and WPTC showed better Carr's index with 25.61, 23.91 and 27.37 respectively. On the other hand, transformation of cellulose to MCC from SBW samples cause morphological modification with reduced particle size than cellulose samples. Thus the SBW MCCs have reasonably improved flow properties than CM samples, specifically in autoclave assisted extracted MCC samples. The APTM, ASM and APM samples showed better compressibility than commercial MCC with values of 26.19, 26.85 and 26.85 respectively. The Carr's index can be improved by further milling and sieving of extracted SBW MCC samples (Amin et al., 2014; Strydom et al., 2011).

Hausner's ratio monitors the internal friction between particles as well as flowability of the powder samples. Hausner's ratio of ≤1.5 showed excellent to fair flowability. As compared to CC and CM samples, MCC samples showed better flowability with

1.36, 1.373 and 1.373 in APTM, APM and ASM respectively which indicate the fairly low cohesiveness in powder samples' flowability properties.

The overall flow properties of the extracted SBW's MCCs showed relatively improved better properties as compared to cellulose by the modification of particle size, shape and surface area due to depolymerisation of extracted cellulose to the shorter chains of MCC by chemical hydrolysis (B. Chen et al., 2020).

4.3.1.2 Porosity

Porosity property explicates the voids and pores within the sample due to intense pre-treatments and hydrolysis. The SBW's cellulose such as WPTM, WPM, APC and APTM showed significant difference from CC. As compared to CM, extracted cellulose from the shallot waste showed significantly higher porosity in ASC and WPTC. MCC samples also exhibited a similar trend as in the case of cellulose. ASM and WPTM showed intense porous powder with values of 82.58±1.55 and 88.38±1.37 respectively. After the transformation of MCC, reduced porosity was observed due to the extreme pre-treatments, chemical hydrolysis and milling process.

The SBW MCC's samples of ASM and APTM showed lower porosity of 78.69 and 78.78% as compared to CM. It is due to the uniform shapes and sizes as well as the reduced irregularity in structural modification of the extracted MCCs. Thus, porosity plays an important role during compression such as size reduction, fragmentation and drug discharge, especially in the field of pharmaceuticals in the applicability of tablet preparation (Amin et al., 2014).

Table 4.7. Flow behaviour of extracted cellulose from the shallot waste streams through hot water and steam-assisted pre-treatments.

Parameters	CC	WPC	WSC	WPTC	APC	ASC	APTC
Powder densities							
Bulk density (g/cm^3)	0.248±0.003^a	0.212±0.004^b	0.247±0.01^a	0.155±0.01^c	0.224±0.01^b	0.249±0.01^a	0.143±0.003^d
True density (g/cm^3)	1.339±0.01^a	1.424±0.03^a	1.452±0.04^a	1.303±0.08^a	1.393±0.04^a	1.42±0.02^a	1.341±0.01^a
Tapped density (g/cm^3)	0.502±0.02^{ab}	0.452±0.016^b	0.539±0.001^a	0.323±0.01^c	0.441±0.01^b	0.525±0.01^a	0.305±0.01^c
Flow properties							
Carr's index	25.61±4.17^a	29.59±3.45^a	31.11±1.57^a	27.37±10.4^a	23.91±2.41^a	28.7±5.45^a	29.54±1.49^a
Hausner's ratio	1.348±0.07^a	1.423±0.07^a	1.452±0.03^a	1.409±0.24^a	1.315±0.04^a	1.41±0.1^a	1.42±0.03^a
Porosity(%)	83.35±0.29^d	85.09±0.71^{bc}	82.95±0.74^{cd}	87.26±0.85^{ab}	81.99±2.08^{cd}	89.9±0.24^{cd}	81.62±0.56^a

***Note- Results were tabulated as mean ±standard deviation with triplicate values. Different Superscripts within the same table mean statistical difference ($p < 0.05$) with confidence level 95% interval.**

- **Autoclaved pre-treated cellulose samples from the peel(p), stalk(S) and petiole (PT) were denoted as APC, ASC and APTC.**
- **Hot water treated cellulose samples from the peel(p), stalk(S) and petiole (PT) were termed as WPC, WSC and WPTC.**
- **CC denotes commercial cellulose**

Table 4.9. Flow behaviour of extracted MCC from the shallot waste streams through hot water and steam-assisted pre-treatments

Parameters	CM	WPM	WSM	WPTM	APM	ASM	APTM
			Powder densities				
Bulk density (g/cm^3)	0.18±0.011^{b}	0.304±0.016^{ab}	0.316±0.032^{a}	0.273±0.007^{ab}	0.291±0.016^{c}	0.29±0.02^{b}	0.246±0.005^{a}
True density (g/cm^3)	1.36±0.08^{a}	1.439±0.11^{a}	1.411±0.07^{a}	1.575±0.11^{a}	1.556±0.09^{a}	1.373±0.09^{a}	1.373±0.091^{a}
Tapped density(g/cm^3)	0.278±0.0004^{a}	0.434±0.048^{a}	0.445±0.038^{a}	0.429±0.017^{a}	0.419±0.028^{b}	0.335±0.018^{a}	0.419±0.028^{a}
			Flow properties				
Carr's index	35.49±3.77^{a}	30.09±5.36^{b}	28.97±3.41^{b}	36.26±4.14^{ab}	26.85±4.72^{a}	26.85±4.72^{a}	26.19±4.42^{a}
Hausner's ratio	1.556±0.0911^{a}	1.439±0.116^{a}	1.411±0.068^{b}	1.576±0.107^{ab}	1.373±0.092^{a}	1.373±0.092^{a}	1.36±0.08^{a}
Porosity (%)	81.82±1.32^{a}	78.95±1.74^{a}	77.47±2.88^{a}	82.58±1.55^{a}	88.38±1.37^{a}	78.69±2.03^{b}	78.78±1.98^{b}

***Note- Results were tabulated as mean ±standard deviation with triplicate values. *Different Superscripts within the same table mean statistical difference (p≤0.05) with confidence level 95% interval.**

- **Autoclaved pre-treated microcrystalline cellulose samples from the peel(p), stalk(S) and petiole (PT) were denoted as APM, ASM and APTM.**
- **Hot water treated samples were termed as WPM, WSM and WPTM.**
- **CM termed as commercial Microcrystalline cellulose**

4.3.2 Hydration attributes of cellulose and MCC

Prediction of hydration properties such as WHC, SC, OHC, mainly depends on the source, structure, particle size, extraction treatments, conditions, and porosity of the cellulose and the MCC.

WHC measures the water holding capacity of the cellulose and MCC extracted from SBW streams. Fig 4.8 display that prepared cellulose samples exhibited excellent hydration properties than commercial cellulose (CC). WHC of APC and APTC showed significant incremental values as compared to CC with values of 19.78±0.81 and 18.85±0.05 respectively due to its porous structure induced by steam-assisted pre-treatment. Transformation of cellulose to MCC caused microstructural changes which attribute to the high expansion of microfibrils and milling into powder form which influence the hydration properties. The APM and WPTM showed excellent hydration capacity with 26.15±0.06 and 22.59±3.55 than other extracted MCC samples. Increment in WHC improves texture, mouth-feel and viscosity, thus can be termed as fat mimicker. It can also reduce the syneresis caused by fat separation from whey by removal of fat from milk and milk products which in turn enhances its marketability and nutritional properties. Also, point out the storage conditions and cost reduction for the development of food products.

Subsequently as WHC increases, an increase in SC was also observed in extracted MCC samples as compared to CC and CM. The MCC samples such as APM, APTM and ASM showed a significant increase in SC with 11.7±0.18, 8.908±1.68 and 5.050±0.37 respectively. This may be due to the pre-treatments, especially autoclaving that caused expansion of the surface area and induced greater swelling capacity. Also, cleavage of longer chain glucose to shorter chains by the depolymerisation reactions during acid hydrolysis successively destroyed the water

binding sites and cellulosic structure that influenced increase in hydration properties such as WHC and SC. Moreover, increase in WHC and SC exhibit therapeutic functions after being reached to the stomach. Cellulose and MCC starts expanding in the intestinal tract that causes intestinal peristalsis and enhance the level of satiety to inhibit the hunger sensation which subsequently prevents obesity and other related issues (Fang et al., 2020).

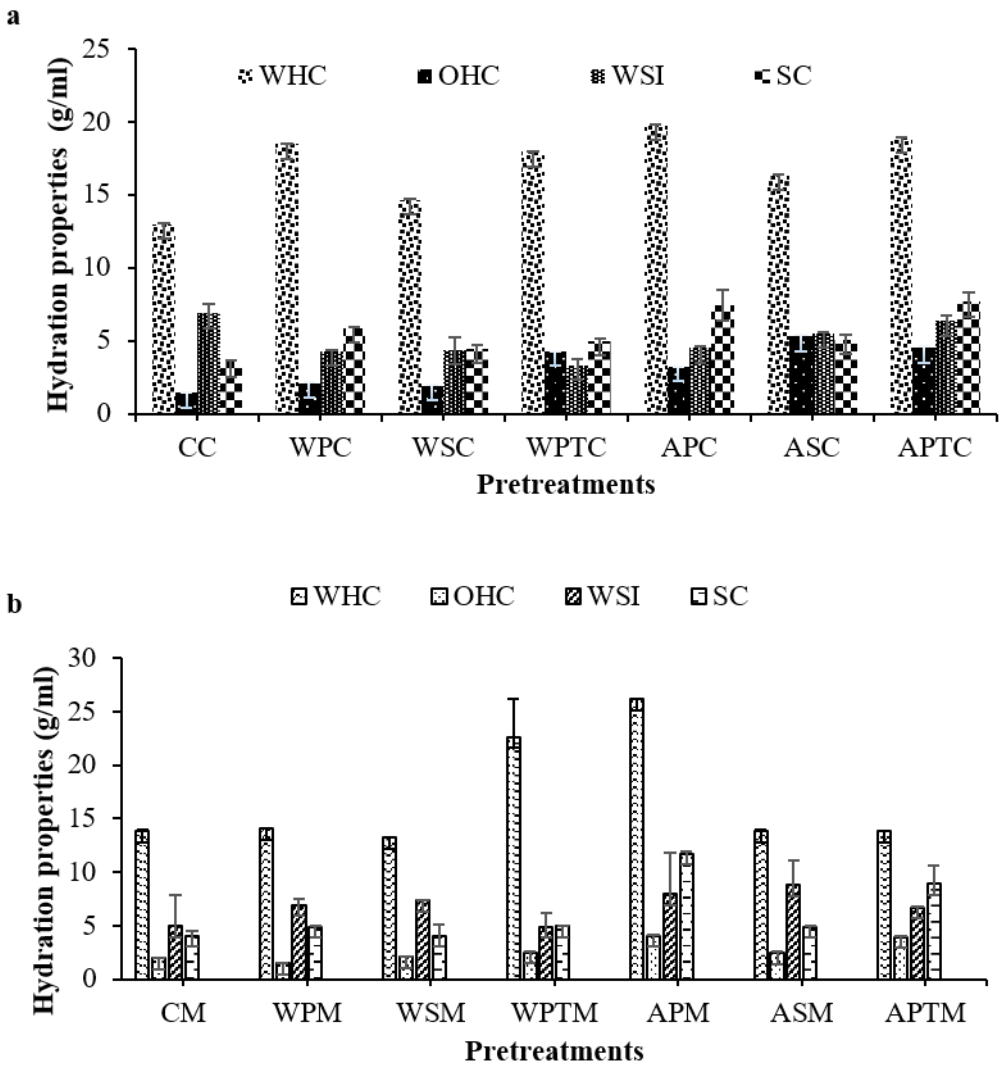


Figure 4.8. Effect of pre-treatments on Hydration properties of SBW's a) Cellulose and b) MCC samples.

*** Note: Results are represented as mean ± SD (n=3)**

- **Autoclaved pre-treated samples from the peel(p), stalk(S) and petiole (PT) were denoted as AP, AS and APT**
- **Hot water treated samples were termed as WP, WS and WPT**
- **CC & CM are commercial cellulose and MCC samples**

On the other hand, OHC of the extracted cellulose showed a significant increase in adsorption of oil than MCC samples. Interestingly, this is due to the loose and porous structure and high degree of polymerisation of SBW's cellulose. ASC and APTC samples showed increased absorption capacity of oil with 5.297 and 4.510 g respectively as compared to CC. While in MCC samples, APM and APTM with values of 4.084 and 3.903 g showed better OHC as compared to CM. OHC improves the taste and texture of the food and also exhibits better stability in the emulsification of food constituents (Tril et al., 2014). The decline of OHC as compared to other microfibrils or dietary fibre was due to the lower particle size. Also, inhibition of absorption of excess fat in the intestinal tract and stomach lining result in expulsion of the fat through defecation (Y. Zhao et al., 2018).

4.3.3 Foaming capacity and foaming stability

Fig 4.9 (a) and (b) illustrates the values of FC and FS of extracted cellulose and MCCs from SBW streams. The foaming properties help to evaluate the stability of the ingredients in various areas of food applications, especially for production of dough, batter and cake mixes etc., The FC showed a slightly significant difference in all cellulose samples as compared to CC (Mokhtar et al., 2018). While in FS, no significant difference ($P \leq 0.05$) was observed in extracted SBW cellulose samples. However, after the modification of cellulose through acid hydrolysis, MCC samples become more porous leading to significant differences in properties as compared to CM. FC and FS values were high in APC with values of 24.68 and 83.33% respectively. While in MCC samples, APTM and APM showed a significant increase in FC as compared to CM with values 32.33 and 27.99 % respectively. It happens

due to the increased air entrapment in the porous region by autoclave pre-treatment whereas, APTM showed increased FS with 76.97% as compared to CM. The factors such as extraction process, viscosity, and pH are the major factors that are significant for the foam capacity and stability (Ahmad et al., 2009).

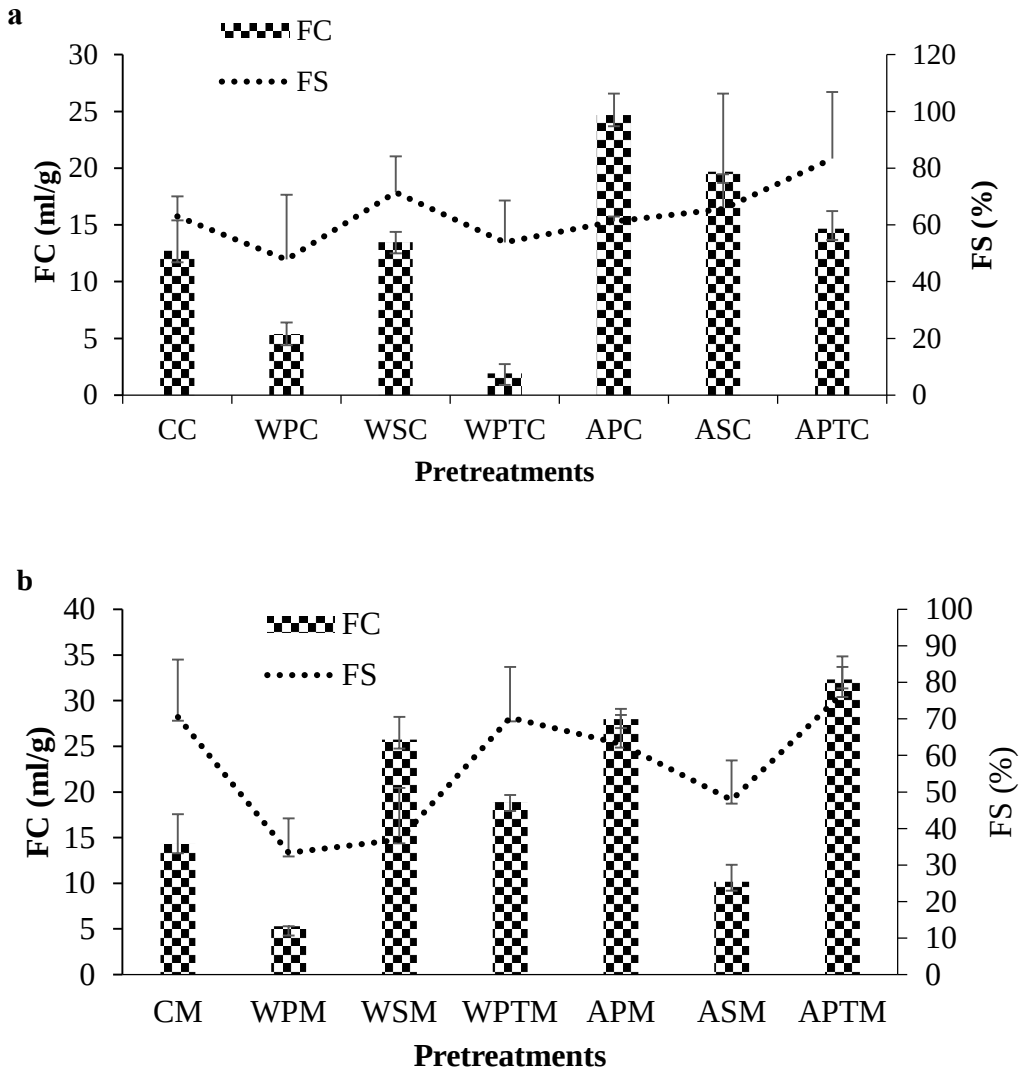


Figure 4.9. Foaming capacity and stability of extracted (a)cellulose and (b) MCC samples from shallot waste streams. *Note: Results are represented as mean ± SD (n=3)

- **Autoclaved pre-treated samples from the peel(p), stalk(S) and petiole (PT) were denoted as AP, AS and APT**
- **Hot water treated samples were termed as WP, WS and WPT**
- **CC & CM are commercial cellulose and MCC samples. Triplicate values with SD values**

4.6 Rheological flow characteristics

Rheological properties of extracted cellulose and MCC are demonstrated as in Table 4.11. Suspended aqueous samples of SBW MCCs displayed rheological characteristics related to its surface charge, swelling capacity and intramolecular interaction which caused increased shear rate with increasing shear stress. Table 4.9 displays the shear stress v/s shear rate parameters of isolated cellulose and MCC samples. Herschel-Bulkley and power-law models were fitted on the stress-shear rate curve to assess the flow index(n), consistency (K) and coefficient ratio (R^2). The characteristics of the extracted cellulose and MCC samples from SBW streams mainly depend on the extraction process, particle size and source. In general, these samples exhibited flow index n >1 which indicate shear thickening behaviour of the liquid samples (Jia et al., 2014). Extracted cellulose samples showed better flow index and consistency as compared to MCC. Among the extracted samples, different sources of shallot waste stream showed significant fluctuation in consistency and flow index in both the cellulose and MCC samples. the flow index was higher in APTC (2.4258±0.75, 0.7479±0.150) APC (0.7778±0.108, 1.1943±0.0) and WSC (1.4340±0.19) $Pa.s^n$ respectively which help to produced good viscous food products. These samples showed the nature of increased values as the shear rate increases which exhibit as dilatant liquid with increased shear-thickening. As compared to CC, APTC samples showed better K values with consistency index values of 2.4258 and 0.7479 along with a coefficient ratio of 0.903.

The transformation of cellulose to MCC using autoclave pre-treatment has improved its flow index (Orasugh et al., 2018).The autoclave pre-treatments made the intact structure of cellulose to porous matrix by the formation of polymeric chain

through H-bonding which in turn increased the water holding capacity of the food matrix. Owing to that, an increase in shearing rate with shear stress increases the flow index that result in thickening of liquid by the addition of MCC samples. As compared to CM, ASM and APTM showed better flow of 1.66405 and 1.127 along with flow index values of 0.9531 and 0.9258 $Pa.s^n$ respectively. Remarkably, the correlation coefficient (R^2) of the ASM and APTM samples showed higher values of 0.945 and 0.973 as compared to CC and CM that proved good model fitting. Also consistency depends on the nature, porosity, concentration, solvent type and temperature. All the extracted MCC samples showed significant increment in flow properties compared to cellulose samples.

Table 4.9 Rheological measurement of extracted cellulose and MCC from shallot biowaste by hot water and steam-assisted pre-treatments.

Sample	Consistency index K	Flow index n ($Pa.s^n$)	R^2
Cellulose			
CC	0.4087±0.01	1.2044±0.75	0.903±0.127
WPC	0.0058±0.008	1.2729±0.03	0.969±0.005
WSC	0.3149±0.048	1.4340±0.19	0.983±0.022
WPTC	0.1259±0.089	1.0536±0.03	0.999±0.0004
APC	0.7778±0.108	1.1943±0.01	0.990±0.01
ASC	0.411±0.028	1.0674±0.05	0.998±0.003
APTC	0.7479±0.150	2.4258±0.75	0.903±0.127
MCC			
CM	0.4378±0.335	1.1386±0.198	0.979±0.028
WPM	0.941±0.058	1.01245±0.012	0.989±0.02

WSM	0.6826±0.42	1.1112±0.150	0.991±0.012
WPTM	0.6685±0.46	1.01505±0.001	0.981±0.027
APM	0.9531±0.012	1.01245±0.012	0.997±0.003
ASM	0.4025±0.57	1.66405±0.88	0.935±0.093
APTM	0.9258±0.001	1.127±0.178	0.973±0.013

***Note- Results were tabulated as mean ±standard deviation with triplicate values. Autoclaved pre-treated microcrystalline cellulose samples from the peel(p), stalk(S) and petiole (PT) were denoted as APM, ASM and APTM. whereas hot water treated samples were termed as WPM, WSM and WPTM. CM denotes commercial MCC**

4.4 Particle size distribution

Fig 4.10 and 4.11 illustrates the particle size distribution of cellulose extracted from SBW streams with autoclave assisted pre-treatments along with chemical hydrolysis. It was observed that particle size distribution was varying from each source due to the difference in morphological confirmation (Varghese et al., 2022).

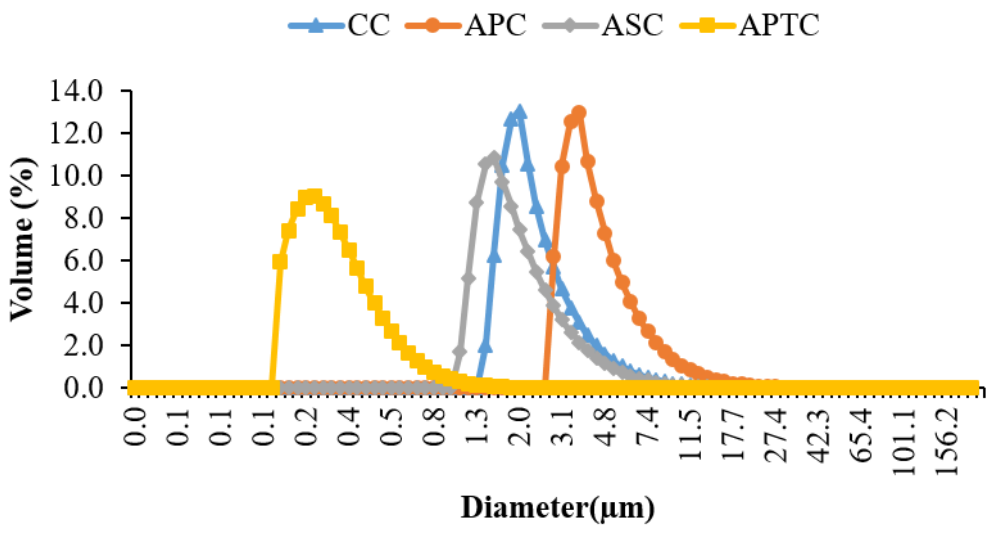


Figure 4.10 Particle size distribution of cellulose extracted from SBW's. *Note– Autoclaved pre-treated samples from the peel(p), stalk(S) and petiole (PT) were denoted as AP, AS and APT. CC termed as commercial cellulose

The dynamic laser scattering technique is a tool to enumerate the distribution between micro and Nano-sized particles. As compared to CC, APTC and ASC showed the lowest particle size distribution with a Z average of 0.1667 and 2.2728 µm. D_{90} percentile of the particle size average was in the range of 0.2555 and 3.5008 µm in APTC and ASC. The polydispersity index was observed lower with values of 1.317 and 1.47 in APTC and ASC respectively.

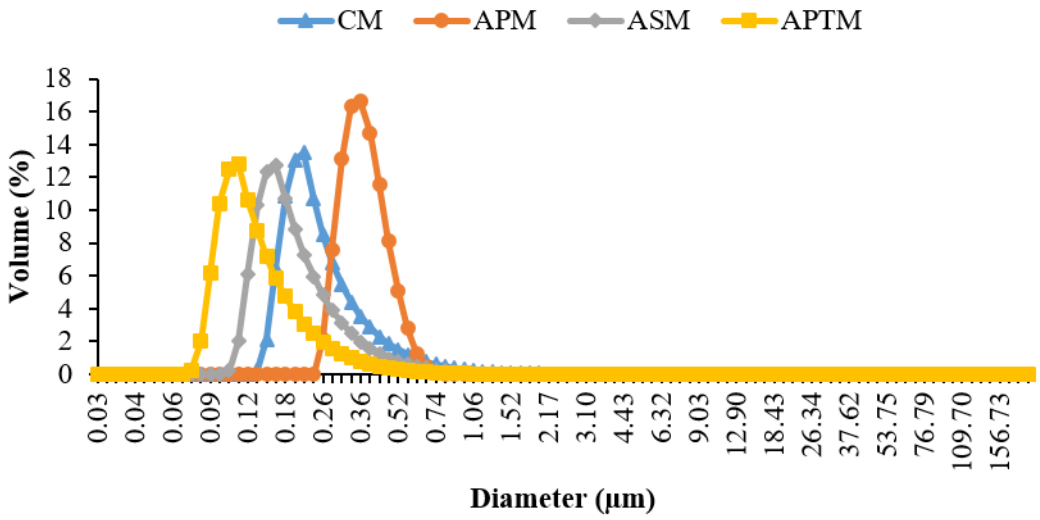


Figure 4.11. Particle size distribution of MCC extracted from SBW's. *Note: Autoclaved pre-treated samples from the peel(p), stalk(S) and petiole (PT) were denoted as AP, AS and APT. CM is the commercial MCC

While in case of SBW's MCC, reduced size was observed as compared to cellulose samples. Identical fine particle distribution was found out to be 0.2349 and 0.3717 µm in APTM and ASM respectively in the D_{90} percentile (90%). It indicates that finer particles show excellent dispersion and solubility properties that could be exploited in food processing and development (Akatan, 2022). However, the polydispersity index (PDI) was increased for MCC as compared to the cellulose samples with a value of 2.028 in APTM. The polydispersity index is directly proportional to the uniform particle size distribution of the given samples. The increase in polydispersity index will make more board particle size distribution and

vice versa (Zhao et., al. 2018). These differences are mainly by extraction conditions like pre-treatments, grinding techniques and source of materials (Varghese et al., 2022). The span is mainly dependent upon the width of the particles between broader to wider sizes in finely powdered samples. In this experiment, extraction to modification of cellulose to MCC, span size was decreased gradually in APM, APTM and ASM of 0.578, 1.224 and 1.179 µm respectively as compared to CC and CM. Z average was reduced with 0.1585 and 0.2481 µm in APTM and ASM respectively. Thus, particle size of the MCC samples was reduced after the depolymerisation of cellulose during effective acid hydrolysis which was proved through a volume basis. A detailed tabulation of particle size and zeta potential of MCC samples was given in table 4.10

4.5 Zeta potential

Zeta potential gives an account of the electrical charges on the surface and dispersion stability of the particles in MCC samples (Mandal & Chakrabarty, 2011). The Zeta potential was lower in APC and ASC compared to CC with values of -17.4 and 24.8±8.03 respectively. Moreover, MCC samples showed in table 10 with values -28.6±4.46 and -29.5±5.3 in APM and APTM respectively. Removal of impurities and better hydrolysis result in smaller particle size with high surface area and mean surface charge favours the particle to particle interaction. The negative values of zeta potential indicate the stability and the possible agglomerating tendency between the particles. Also, increase in negative potential more anionic crystalline cellulose (purity) content in extracted samples.

Table 4.10 DLS and Zeta potential of the particle distribution of extracted SBW's cellulose and MCC.

Samples	Z average (µm)	D10 (µm)	D50 (µm)	D90 (µm)	Span	Polydispersity Index (PI)	Cumulative average (%)	Zeta potential (mV)	Conductivity (mS/cm)
CC	2.904±2.03	1.6093	2.2087	4.4019	1.264	2.78	5.752	-26.9±7.77	0.0155
APC	7.117±0.61	3.9956	5.4697	10.756	1.236	3.073	6.4862	-17.4±5.71	0.0311
ASC	2.273±1.56	1.2299	1.7442	3.5008	1.3021	1.47	5.0694	-24.8±8.03	0.0714
APTC	0.167±0.09	0.0904	0.1347	0.2555	1.226	1.317	2.4109	-36.7±5.08	0.009
CM	0.593±0.42	0.3279	0.4554	0.8919	1.238	3.352	7.2819	-33.5±4.87	0.0607
APM	0.159±0.11	0.0902	0.1227	0.2349	0.579	2.028	4.5739	-29.5±5.3	0.0185
ASM	0.430±0.10	0.3085	0.394	0.5366	1.224	2.678	5.1508	-28.6±4.46	0.0201
APTM	0.248±0.18	0.1381	0.1908	0.3717	1.179	2.727	5.8146	-32.3±5.98	0.0977

***Note – autoclaved pre-treated samples from the peel(p), stalk(S) and petiole (PT) were denoted as AP, AS and APT. CC & CM are commercial cellulose and MCC. Triplicate values with SD values. Span denotes the total with of the PDI**

4.7 Optimization and Replacement Efficiency of MCC as Fat Replacer and Stabilizer in Low Fat Stirred Plain Yogurt

4.7.1 Experimental setup

The samples were prepared according to table 4.11 and the pre-fermentation process was carried out at T_1 and T_2 until it reaches pH 4.6. The concentration of PMCC, SMCC, and PTMCC was in the range of 0.25, 0.5, 0.75 and 1g per 100g of yogurt samples. The control samples were also prepared without the addition of MCC. The finished products were homogenized at 9500rpm for 2min for the uniform distribution of the MCC. The yoghurt samples were immediately cooled at 4˚C and stored for 48h. The quality parameters were inspected throughout the processing and storage of developed stirred yogurt.

Table 4.11 Experimental setup for the development SBW MCCs incorporated stirred yoghurt

Extracted MCC's	Treatments	Temperature (°C)	Milk (ml)	MCC (g)	Starter Culture (ml)
	WMC	37(T1) 43(T2)	98	0	2
	SMC		98	0	
PMCC	PY1		97.75	0.25	
	PY2		97.5	0.5	
	PY3		97.25	0.75	
	PY4		97	1	
SMCC	SY1		97.75	0.25	
	SY2		97.5	0.5	
	SY3		97.25	0.75	
	SY4		97	1	
PTMCC	PTY1		97.75	0.25	
	PTY2		97.5	0.5	
	PTY3		97.25	0.75	
	PTY4		97	1	
CMCC	C1		97.75	0.25	
	C2		97.5	0.5	
	C3		97.25	0.75	
	C4		97	1	

*Terms displayed above designates as follows; PMCC, SMCC, PTMCC- Extraction of MCC autoclave assisted pre-treatment of SBWs. Treatments contains in stirred yogurt preparation were WMC (control-whole milk without addition), SMC (control-skim milk without addition), P1(0.25%), P2(0.5%), P3(0.75%) and P4(1.0%) and vice versa. T_1 and T_2 denote the two different incubation temperatures used for fermentation (37°C and 43°C).

4.7.2 Effect of pH, Titratable acidity, TSS on fermentation of yoghurt in addition to PMCC, SMCC and PTMCC

The fermentation was carried out at two different temperatures of T_1 and T_2 in varying concentrations of P-MCC, S-MCC and PT-MCC were illustrated in table 4.12. The pH plays an important role in the development of yoghurt. There were significant changes($P \leq 0.05$) in pH shown in different temperatures as compared to SMC and WMC in the presence of probiotic microorganisms such as *Lactobacillus bulgaricus* and *L. cremoris* along with *S. thermophilus*. As the temperature increases, the time to attain pH 4.6-4.5 was extremely varying at T_1 and T_2 (Turgut & Cakmakci, 2018). The pH of yoghurt samples was significantly decreased during the fermentation process which significantly decreased to 4.58 which slightly varies in P4, S4 and PT4 within 5 hours at T_2 as compared to T_1. The pH changes drastically in all samples treated with T_2. While in T_1, the time taken to attain pH 4.6 was 9-10h in all combinations of stirred yogurt samples were observed in fig 4.12. Thus, fermentation was not interfering with the sample combinations as compared to WMC and SM (Vénica et al., 2020). As the pH decreases, products are more prone to other pathogenic contamination. So immediately stop the fermentation by storing at 4-5℃ to ease the further degradation and vice versa in the case of TA (Hasani et al., 2017; Vanegas-Azuero & Gutiérrez, 2018).

Titratable acidity (TA) which is inversely proportional to pH, TA values also showed a significant increase ($P \leq 0.05$) while fermentation in all samples treated at T1 and T2. In WMC samples, TA values showed increase in the 5th hour and 10th hour at T1 and T2 with 0.165 and 0.207 % of lactic acid, because of the increased availability of nutrients as well the increased multiplication of probiotic microorganisms as compared to treated samples. Whereas in PMCC, SMCC, PTMCC, TA decreases suddenly at T2 with 0.186 and 0.222% of lactic acid respectively in P4, S4, PT4 within the 6^{th} hour were showed fig 4.13.

All the treated samples also showed an increase in TA as compared to an increase in temperatures but no drastic increments with the increment of MCC. Thus the addition of MCC was not altering the fermentation with respect to TA (Senaka Ranadheera et al., 2012).

Initially, the addition of SBW MCCs into the Skim milk samples in varying % was not significant affected the TSS quantity along with control samples. TSS values were significantly decreased during the lactic acid fermentation due to the consumption of nutrients by the rapidly growing lactic acid bacteria in the sample. At T_1 and T_2, TSS drastically changed from 11.2 to 7 in WMC and SMC. At T1, a sudden decrease of TSS in the P4, sample from 13.6 to 6.3 in the 6th hour of the fermentation was observed. In case of SMCC and PTMCC, TSS was drastically reduced ($P \leq 0.05$) from 13.6 to 7.4 (S_4) and 13.6 to 7.4 (PT_4) respectively at 6^{th} hour of fermentation process showed in fig 4.15. L. bulgaricus is known to produce proteolytic enzymes by degrading the protein to peptides and amino acids, thus stimulating the growth of S. thermophilus which further produces formic acid and CO_2 that accelerates the growth of L. bulgaricus and L. cremoris. This mixture inoculum will ferment the available lactose sugar to lactic acid (Kieserling et al., 2019).

Concluding the results, all the samples showed same trend in fermentation of low fat yogurt in addition of SBWs MCC samples has no effect on pH, TA and TSS was observed same in prior literature. The addition of 1% of PMCC, SMCC AND PTMCC (P4, S4 and PT4) showed significant deviation during the fermentation. Also, starter culture, temperature, milk source and time was significantly plays a critical role in fermentation process and yogurt quality.

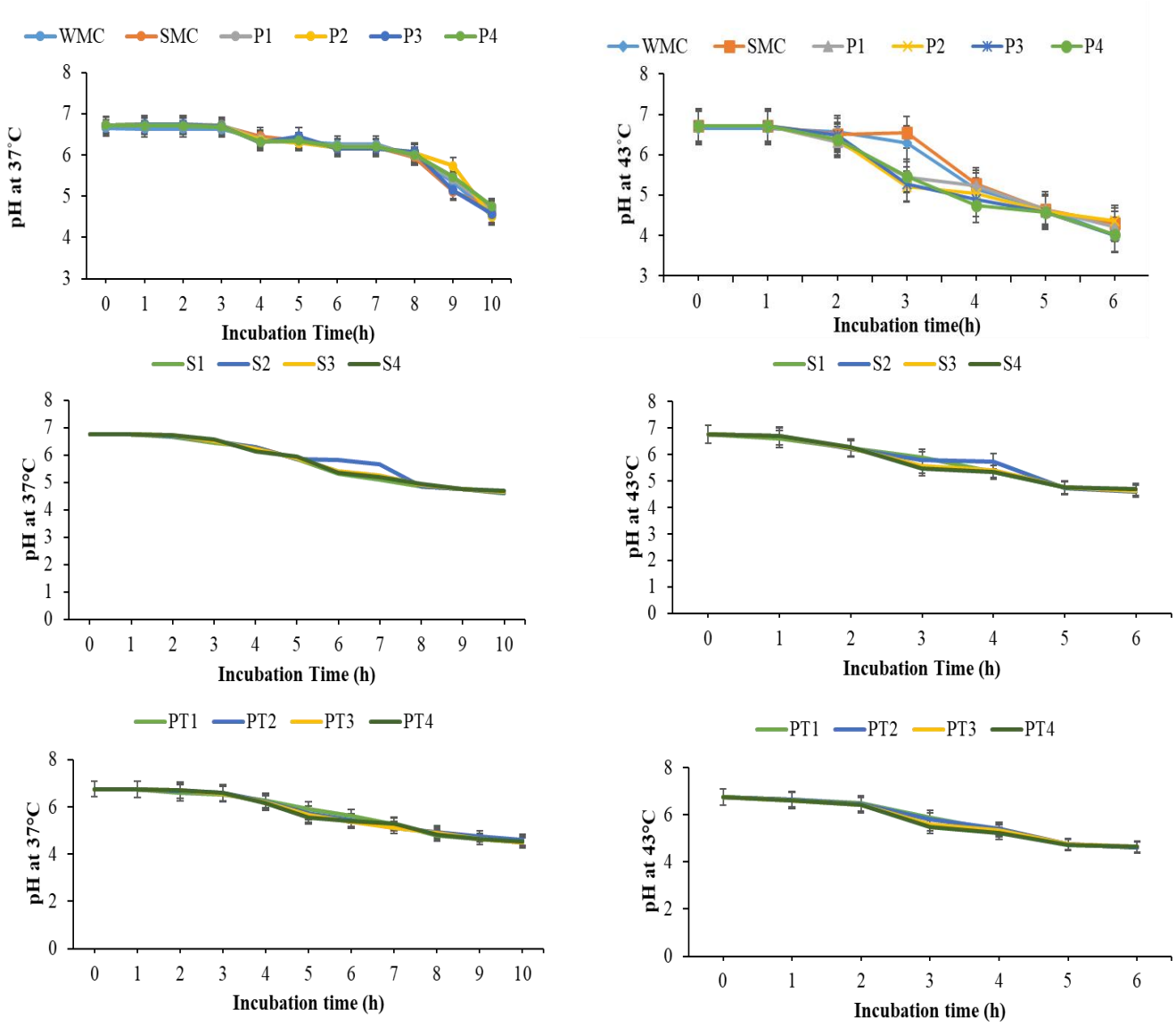


Figure 4.12 Effect of pH on pre-fermentation of yoghurt samples in addition to P-MCC, S-MCC, PT-MCC as compared to control samples (WMC, SMC) at T_1 and T_2 (37°C and 43°C)

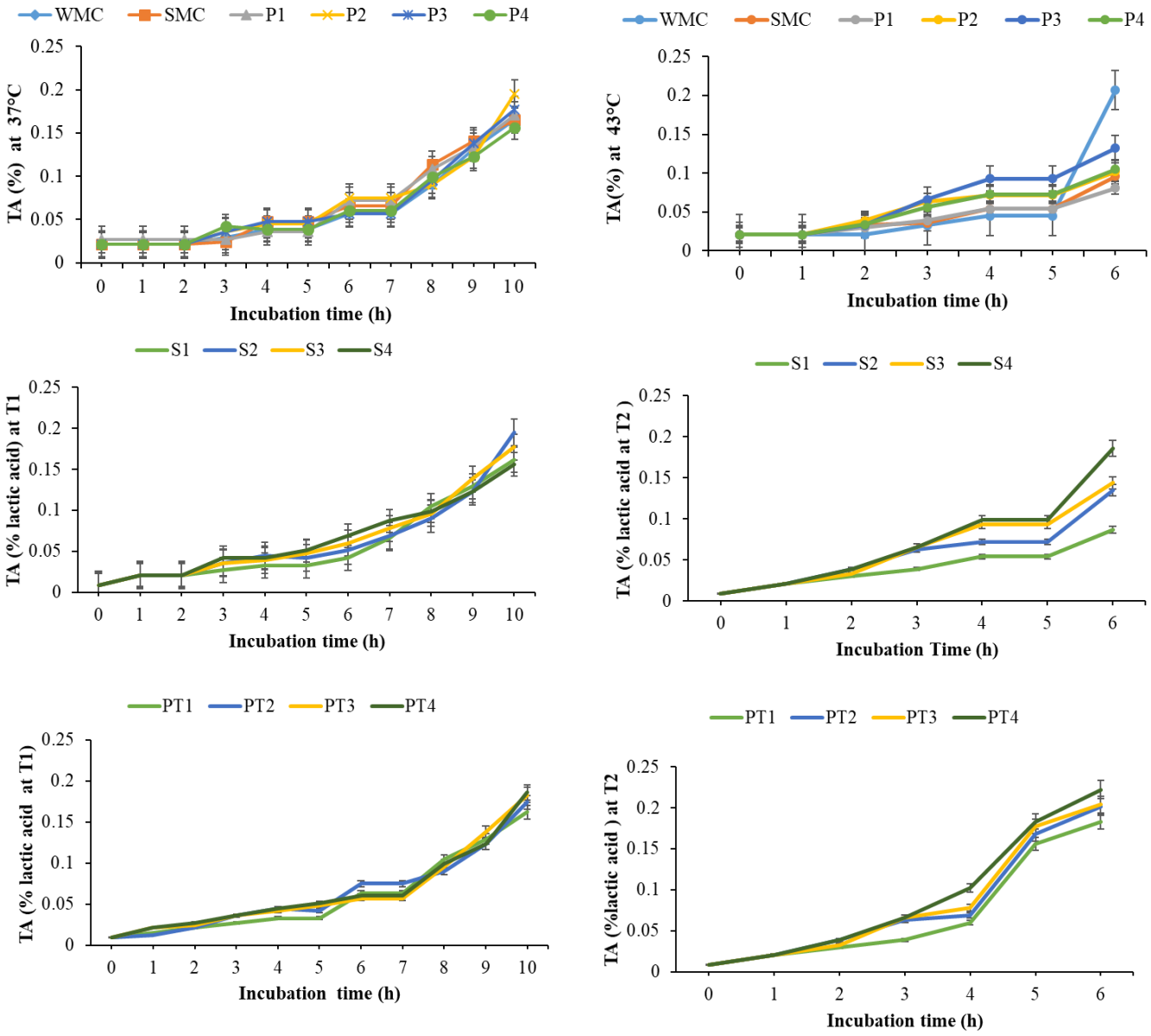


Figure 4.13 Effect of Titratable acidity (TA) on pre-fermentation of yoghurt in addition to P-MCC, S-MCC, PT-MCC as compared to control samples (WMC, SMC) at T_1 and T_2 (37°C and 43°C). *Note-Results are represented in the form of mean ± Standard deviation(n=3)

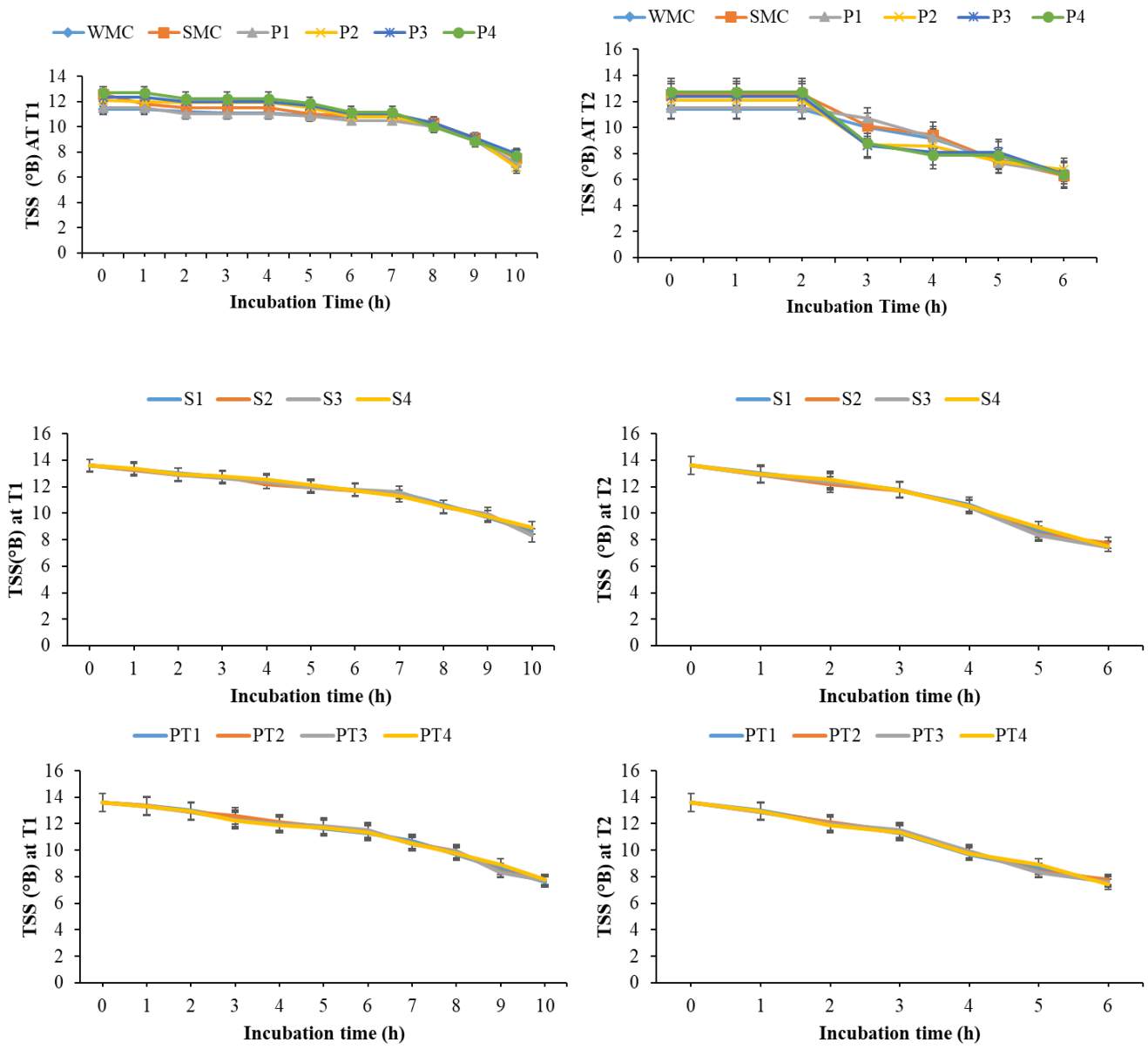


Figure 4.14 Effect of Total soluble solids (TSS) on pre-fermentation of yoghurt in addition to P-MCC, S-MCC, PT-MCC as compared to control samples (WMC, SMC) at T_1 and T_2 (37°C and 43°C). *Note-Results are represented in the form of mean ± Standard deviation(n=3)

4.7.3 Effect of microbial growth during fermentation of yogurt in addition to MCC

The lactic acid bacterial count is mainly dependent on the survival of bacterial culture, media and suitable temperature. While the pH reaches 4.6, the highest microbial count was showed in WMC, SMC, with 5.246 and 4.998 CFU/ml respectively. In case of SBWs MCC added samples also showed slightly increased microbial load at T2 with the values of 4.405, 5.412 and 5.686 CFU/ml in P4, S4 and PT4 respectively within the 6^{th} hour. As the temperature increases, an increase in the bacterial count was showed in all samples as in fig 4.16. As compared to T2, T1 showed uniform increase of LAB count with time. Within the short period, it attained a significant viable count as compared to T2. Within the treated sample, a significant difference was ($P \leq 0.05$) shown in the samples treated at T1. Thus, T1, the extent of increment of viable cell count reaches a maximum at the 10th hour due to the lag phase and an uneven growth rate was showed in each yogurt samples, the cause factor being the difference in incubation temperature (Kieserling et al., 2019). The addition of MCC had not significantly affected the whole fermentation process as compared to control samples. However, 1% added SBW MCCs (P4, S4 and PT 4) showed gradual increment in concentration in lactic acid bacteria along with control samples (Zanhi & Jideani, 2012).

Thus, conclude that, survival of probiotic bacterial was significantly higher in addition of increasing concentration of SBW MCCs samples (P4, S4 and PT4) compared to WMC and SMC was proved according to prior studies (Sobhay et al., 2019).

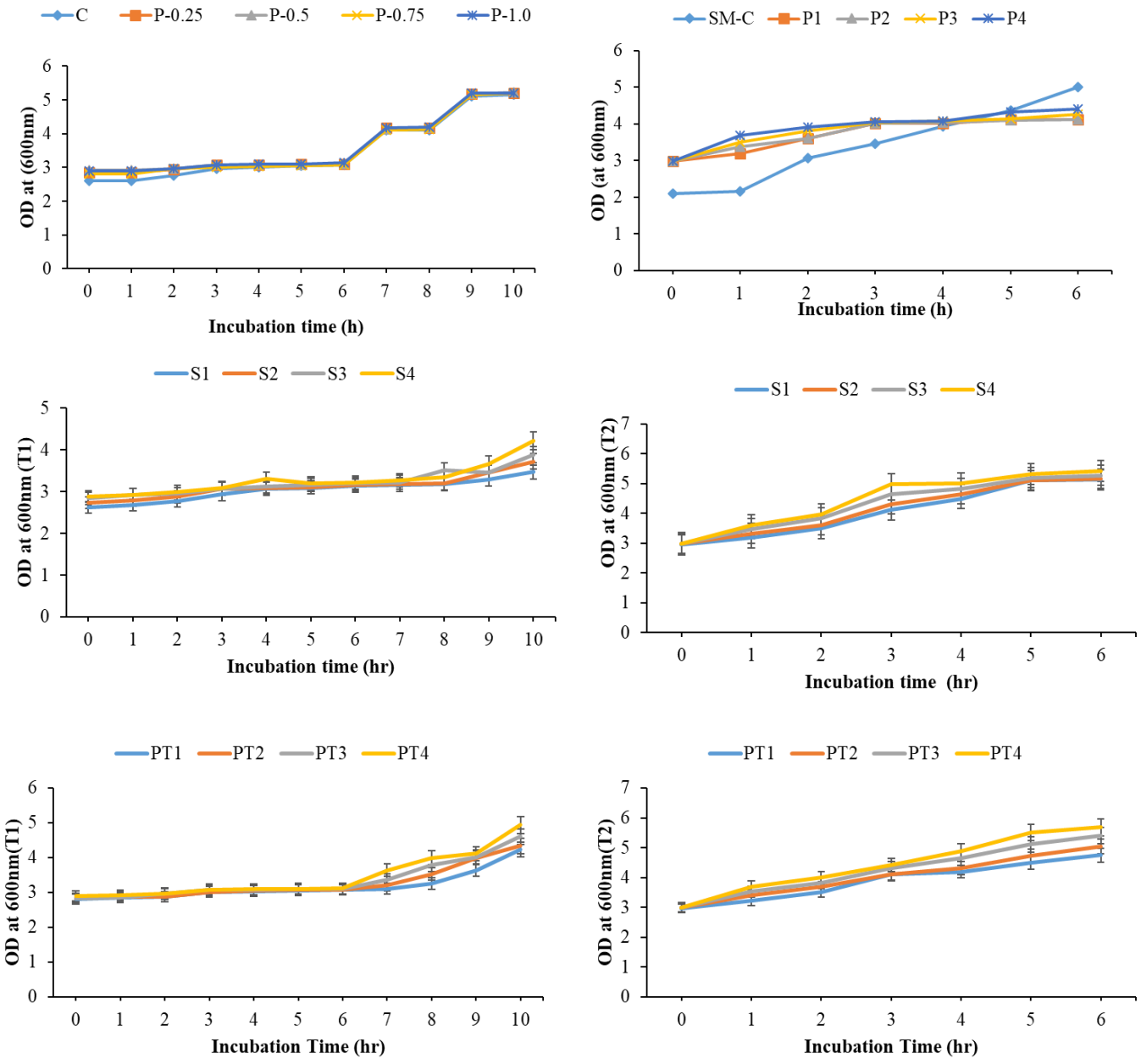


Figure 4.15. Effect of lactic acid bacterial growth (LAB) on pre-fermentation of yoghurt in addition to P-MCC, S-MCC, PT-MCC as compared to control samples (WMC, SMC) at (37°C and 43°C). *Note-Results are represented in the form of mean ± Standard deviation(n=3)

4.7.4 Physiochemical properties of formulated products of low fat stirred yogurt with the addition of SBWs MCC

The physicochemical parameters mainly observed in the finished products were colour index, moisture, total ash and protein as shown in table 4.12. The addition of SBW MCCs showed an influence on the moisture content in yogurt samples. The results showed that, concentration of PMCC % increases, an increased moisture content was shown in P_4 (1%) with 92.05%, owing to the porous nature of MCC that holds the water resulting in high hydrophilicity during storage. While, inaddition of SMCC and PTMCC showed A slight decrease in moisture content as the concentration increases. In SMCC and PTMCC, lowest moisture content was 86.11±0.25 and 84.21±0.12(%) respectively. Due to the temperature and MCC variations was significantly affect the quality of low fat stirred yogurt. Thus, compared to control samples, in addition to MCCs cause significant increase in moisture content. Also, due to the increment of bulk density of the MCCs samples. As the temperature, a slight evaporation because of moisture loses in control and CC samples.

Longer the time taken for fermentation, more absorption of water will take by the MCCs samples as compared to control samples. Also, highest protein content of 4.463g at T_1 and 4.463g at T_2 was observed in WMC. T_1 and T_2 treatment was not significantly affected the protein content during fermentation process. Also, as the increase the concentration of MCCs (0.25-1.0%), quantity of the milk samples was decreased which leads to the slight decrease in the protein content ($P \leq 0.05$) to 3.238g at T_1 and 2.275g at T_2 was observed in P4. Also, PT4 and S4 also showed lower protein content with 3.5 -3.56 and 3.35- 3.33% at T_1 and T_2. The slight reduction of protein can weaken the bonding between the water molecules which cause syneresis during the storage period. Thus, results conclude that there was no significant difference in varying temperature, time and concentration MCCs.

Although, skim milk which was purchased contains less than 1% of total fat content. Inaddition to MCCs in milk, quantity of milk content will reduce very slightly that cause very slight decrease in fat content in samples such as $P_1<P_2<P_3<P_4$, these order whereas, P_4 contains lowest fat content as mentioned in table 4.13 and vice versa for PTMCC and SMCC. The least and highest fat content was found in control samples (3.829%) as compared to treated samples. Also, no fat content was present in SBWs MCC samples which also a reason for the no increase or decrease in stirred yogurt samples. Also temperature and time was not significantly affected fat content which proved in results shown. Total ash content slightly varied with increase in concentration of MCC in the yogurt samples at both fermentation temperatures.

Table 4.13 shows the, an increase in the concentration of SBWs MCC in yogurt resulted in an increase in total ash content. Increased ash content was found in P_4, S_4 and PT_4 contains highest concentration of fibre content in yogurt samples. As compared to control samples, no significant difference($P\leq0.05$) was showed in MCCs incorporated yogurt samples depending upon the temperature and time. It is mainly detecting the increase of mineral content in developed stirred yogurt due to addition of MCCs (Dietary fibre) which results increases the nutritional quality. As shown in the table 4.13 shows significantly difference ($P\leq0.05$) inaddition of SBWs MCC as compared to control samples. There was no degradation happened to fibre during the fermentation and temperatures (T_1 and T_2). Increased concentration was found in P_4, S_4 and PT_4.

Also it proved that, microbial interaction will slightly destruct the insoluble SBWs MCC while milk fermentation. It also binds with nutrient to give a good structural integrity to the final products. Addition of MCCs varies from 0.25 to 1% in each samples, the finished products contains fiber concentration at T_1 and T_2 was found similar in quantity with 0.983, 0.985 and 1.065% in P_4, S_4 and PT_4 respectively. The presence of these MCCs helps to

improve the texture, rheological factors, hydration properties and health benefits of yogurt products.

Due to addition of MCC, there was no significant ($P \leq 0.05$) change in colour parameters as compared to control samples. Increase in the MCC concentration resulted in only a very slight decrease in the L* a* and b* values. In prior studies, some of the hydrocolloids had affected the colour attributes due to the extent of processing and storage conditions. The SMC, P_1, S_1 and PT_1 showed the highest whiteness of 86.17, 86.17, 85.35 and 86.41 at T2 respectively. As the temperature and time cause the color value due to component reaction at higher temperature and longer exposure.

Moreover, fermentation temperature and time did not interfere with the quality of MCC fortified yogurt samples after 48 h refrigerated conditions. Appearance and colour also plays an important indicator for the acceptance of products acceptance. Thus, SBWs MCC was not highly impart the colour aspects of developed stirred yogurt as the results showed in table 4.12. Additionally, the lack of whiteness in yoghurt can be rectified by adding juice or puree, which increases the nutrient value and also fruit attributes such as colour and flavour to the yogurt foodstuffs (X. Wang et al., 2020; Y. X. Wang et al., 2021).

In conclusion, decrease in the proximal content of yogurt after the fermentation was varying according to the concentration of SBW MCCs addition, incubation temperature and time. No significant changes ($P \leq 0.05$) in protein, fat, ash and fibre content at T_1 and T_2. However, moisture and colour index showed significant changes at T_1 in all yogurt samples due to the prolonged time taken for the yogurt development. At T_2, extent of reduction of moisture, protein, fat, ash and fibre level was higher in P_4, S_4 and PT_4 with gradual lowering within 5h without disturbing the yogurt texture, colour and aroma.

Table 4.12. Compositional analysis of stirred yogurts samples after the fermentation process at T_1 and T_2. (*Note-Results are represented in the form of mean ± Standard deviation(n=3))

Samples	Moisture (%)	Protein (%)	Fat (%)	Total Ash (%)	Total Fibre (%)	Colour Index
			T_1			
WM-C	88.83a±1.66	4.463a±0.071	3.829±0.005	0.82a±0.046	-	84.98±0.01
SM-C	85.33a±3.17	3.558b±0.041	0.46±0.011	0.741a±0.109	-	86.41±0.03
P_1	90.67a±0.94	4.463a±0.071	0.46±0.023	0.795b±0.007	0.244±0.041	84.51±0.11
P_2	90.85a±1.85	3.558b±0.041	0.441±0.002	0.941b±0.055	0.495±0.147	85.58±0.016
P_3	91.5a±0.92	3.471a±0.041	0.43±0.024	0.967b±0.579	0.74±0.051	84.14±0.18
P_4	92.05a±0.074	3.238a±0.071	0.465±0.02	1.242ab±0.092	0.983±0.027	84.60±0.05
S_1	88.27±0.23	3.529±0.04	0.472±0.017	0.999±0.16	0.243±0.0006	87.13±0.019
S_2	87.13±0.1	3.471±0.04	0.46±0.023	1.0±0.65	0.529±0.133	86.73±0.061

S_3	85.99±0	3.383±0.04	0.441±0.002	1.067±0.09	0.738±0.0236	87.27±0
S_4	86.11±0.25	3.325±0.07	0.43±0.024	1.378±0.002	0.985±0.021	86.22±0.005
PT_1	88.65±0.13	3.675±0.14	0.465±0.02	0.961±0.01	0.246±0.002	85.27±0.005
PT_2	87.29±0.25	3.558±0.08	0.456±0.04	0.909±0.03	0.501±0.0012	84.62±0.005
PT_3	86.49±0.122	3.558±0.082	0.446±0.001	0.99±0.009	0.748±0.0036	82.3±0.005
PT_4	84.99±0.139	3.5±0.285	0.436±0.009	1.067±0.05	1.065±0.049	80.55±0.005
T_2						
WM-C	88.25±1.49	4.463±0.071	3.951±0.11	0.796^{a}±0.014	-	84.98±0.01
SM-C	86.27±0.54	3.558±0.041	0.391	0.773^{a}±0.025	-	86.17±0.17
P_1	82.45^{a}±0.51	3.471±0.041	0.462±0.063	0.729^{c}±0.05	0.244±0.041	86.17±0.04
P_2	84.5^{a}±5.55	3.238±0.071	0.459±0.03	0.863^{b}±0.043	0.495±0.147	83.54±0.033

P_3	87.05[a]±3.07	2.917±0.109	0.45±0.023	0.934[ab]±0.024	0.74±0.051	82.95±0.36
P_4	87.22[a]±1.79	2.275±0.071	0.475±0.022	1.045[a]±0.036	0.983±0.027	84.67±0.031
S_1	87.29±0.25	3.529±0.04	0.479±0.023	0.980±0.02	0.245±0.0006	85.35±1.42
S_2	86.59±1.09	3.5±0.07	0.462±0.063	1.092±0.043	0.529±0.132	83.79±0.0047
S_3	85.46±0.499	3.471±0.08	0.459±0.03	1.101±0.043	0.738±0.024	82.26±0.004
S_4	83.99±0.27	3.383±0.04	0.45±0.023	1.195±0.003	0.985±0.021	80.65±0.005
PT_1	87.29±0.254	3.616±0.082	0.475±0.022	0.943±0.02	0.245±0	86.41±0.0045
PT_2	87.02±0.25	3.558±0.082	0.47±0.047	1.0±0.039	0.529±0.133	86.11±0.005
PT_3	85.82±0.49	3.5±0.14	0.458±0.04	1.078±0.03	0.738±0.0236	86.39±0.005
PT_4	84.21±0.12	3.5±0.24	0.447±0.04	1.165±0.005	0.818±0.257	85.26±0.03

4.8 Effect of incorporation of SBW's MCC in production of low fat stirred yogurt samples on serum separation, water holding capacity, rheology and texture properties

4.8.1 Syneresis properties

Syneresis seems to be the discharge or excavation of liquid out of a gel as either a consequence of gel deformation. Such a criterion is unpleasant throughout yogurt storage and quality has a significant impact on customers' acceptance of the product. The inclusion of by-product fibre in the formulation of novel foods might performance and scalability, technical, and functional characteristics such as OHC, viscosity, consistency, sensory attributes, and syneresis (Vénica et al., 2020).

Serum separation rate predicts the resistance of certain products toward the forced separation and to find out the initial rate of whey separation in yogurt samples during the storage period as demonstrated in fig 4.16. In each interval of time, serum separation was significantly increased ($P \leq 0.05$) in SMC with 68% at T_1. While at T_2, the same phenomenon was observed with 66% at 15min. During the time interval, 6 minute onwards showed a gradual whey release in serum separation which give insight into the holding capacity of the SBW MCCs in yogurts samples.

In addition to concentration of SBW MCCs, a significant decrease ($P \leq 0.05$) in serum separation rate for a particular period as compared to control samples. The least serum separation was found in PT_4, P_4 and S_4 of, 42.7%, 43% and 52% was observed in 15 min at T_2. Lower the syneresis rate, the more stable will be the milk protein linkage under the acidic condition of yogurt samples.

Fig. 4.17 revealed that in MCC added samples, P_4 was more stable than other concentrations with 31.34 (T_1) and 34.67% (T_2) respectively which is significantly superior than SMC and WMC (Dhingra et al., 2012). As it can be observed, an increased level of fibres addition leads to a decrease in the syneresis values, due to the holding capacity of fibres that absorb the whey released by the gel matrix (Espírito-Santo et al., 2013).

Thus, syneresis was improved significantly as compared to the control sample, since the SBW MCCs samples more anionic in nature that can easily interact with hydrogen bridges with charges moieties on the surface of the protein (cationic nature), that stabilize the water molecules to form a three dimensional network controlling syneresis (Hashemi & Hosseini, 2021).

Thus 1% SBW MCC samples supplementation in low fat stirred yogurt help to reduce the syneresis rate with values of 65.33, 57.3 and 48% in P4, PT4 and S4 at T_2 as compared to control samples (32%). Thus, SBW MCCs can be recommend as stabilizer for controlling the syneresis in all type of yogurt drinks without altering the its consistency, aroma and taste to improve the consumer acceptability along with health benefits.

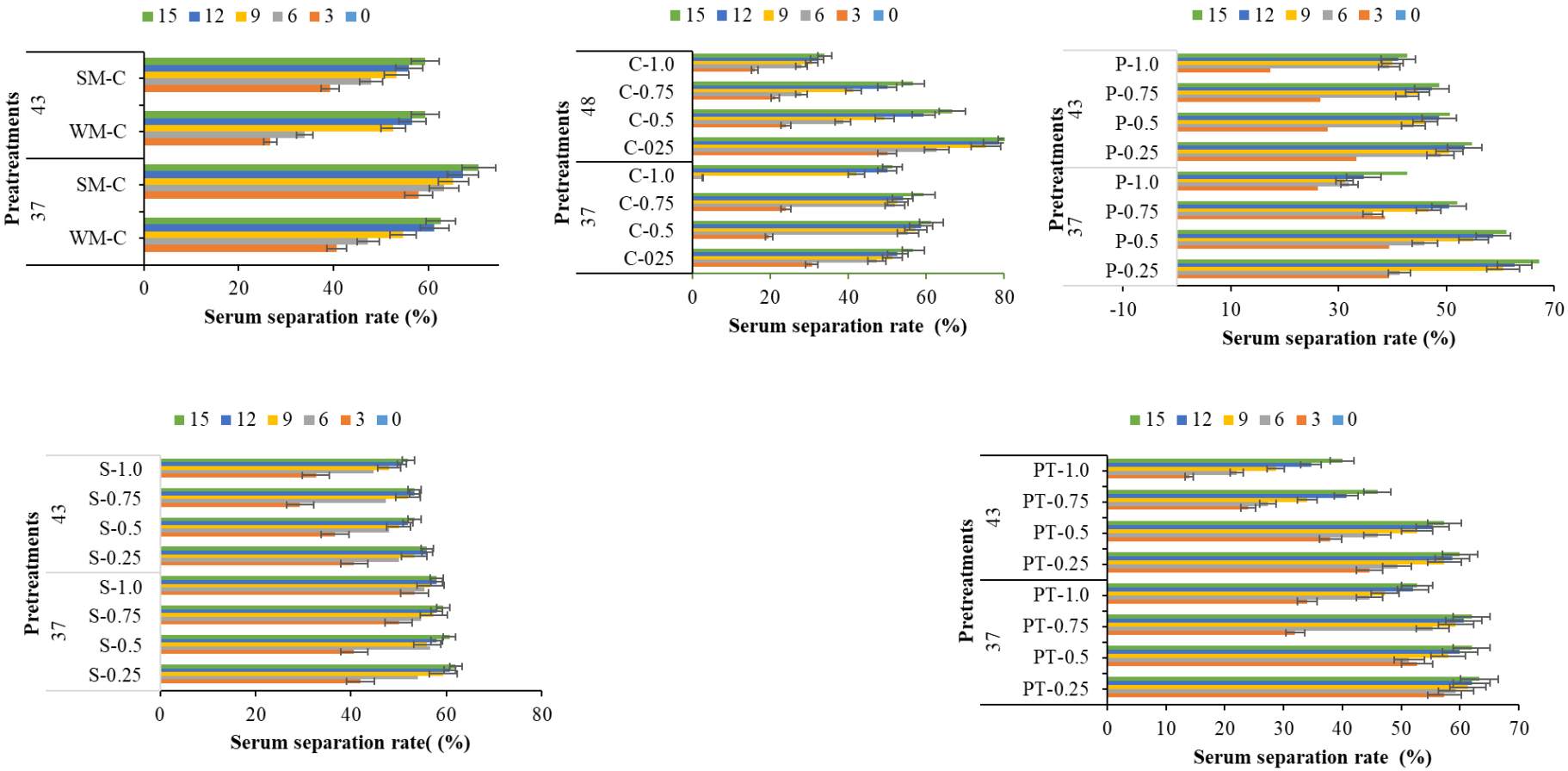


Figure 4.16 Effect of incorporation of SBW's MCC in serum separation rate production of low fat stirred yogurt samples at T_1 and T_2 (37°C and 43°C). *Note-Results are represented in the form of mean ± Standard deviation(n=3)

4.8.2 Water holding capacity(WHC)

The water holding capacity of the stirred yogurt product give an account on the consistency and texture strength of the gel formation in the yogurt preparation. The WHC values were significantly varies from time, temperature and concentration of SBWs MCC samples. As the time increases in fermentation process, more whey will start to expel due to the weakened bonding between the protein-fibre and water molecules (M. Mousavi et al.2019).

As increase in the fibre concentration along with reduced fermentation process with increased temperature showed better results throughout the process. Significantly Increased WHC ($P \leq 0.05$) was found out in P_4 and PT_4 with 66.6 and 64.45% respectively at T_2 as compared to control samples was demonstrated in fig 4.17. while in case SMCC and CMCC, WHC was increased at T_1 with 60.93 and 60.17%.

Thus the WHC is mainly depended on the hydration capacity as well as the retention of the water molecules by the protein and fibre to form three dimensional interactions within the gel configuration in the curdling of milk (Mahsa Ardabilchi12019). Increased addition of SBWs MCC (1%) facilitated to strengthen protein bonding between free state water molecules and minus level of water discharge during the storage period.

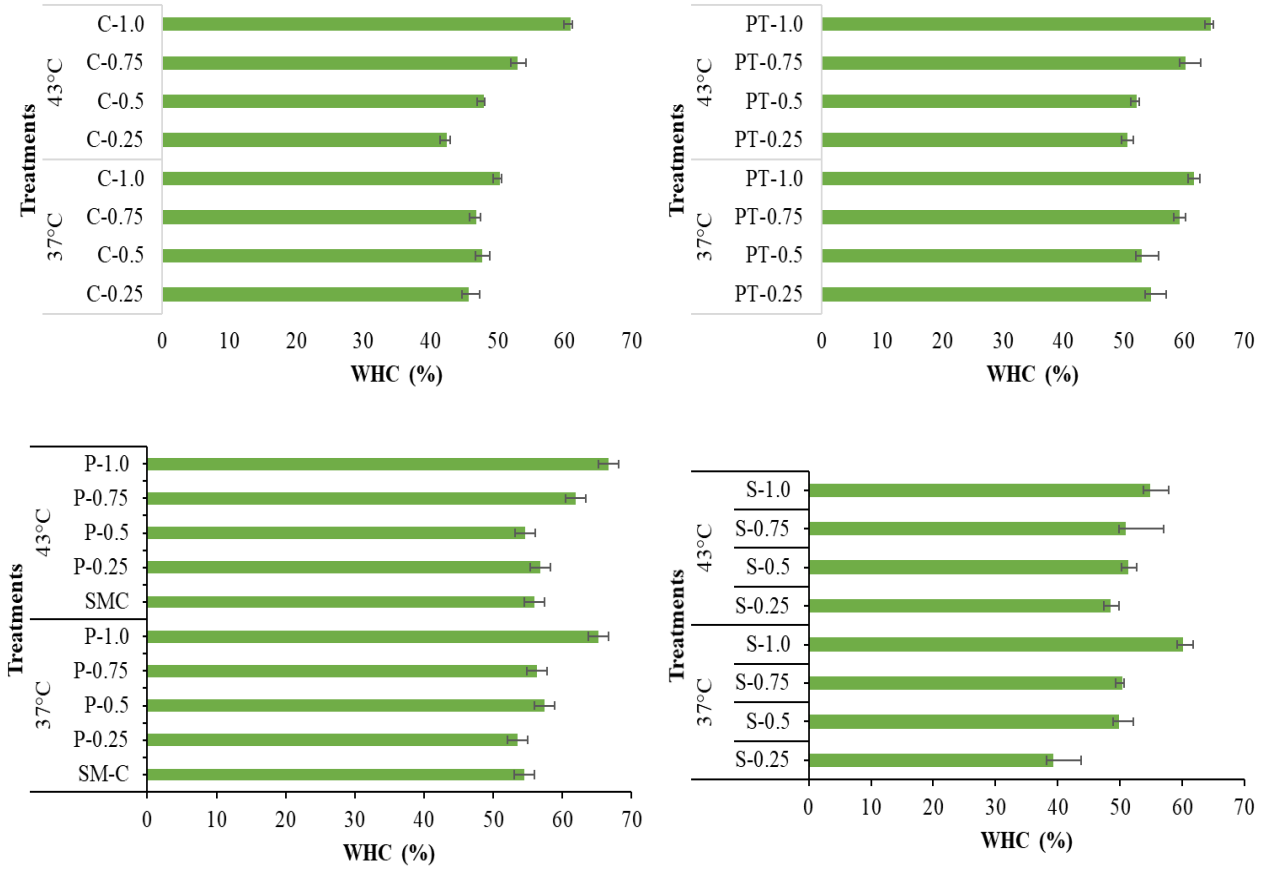


Figure 4.17. Effect of Water holding capacity (WHC) of SBW's MCC incorporated low fat stirred yogurt samples. *Note-Results are represented in the form of mean ± Standard deviation(n=3)

4.8.3 Textural parameters

The textural parameters showed significant increases ($P \leq 0.05$) with addition of SBWs MCC into a low fat stirred yogurt. As shown in table 4.13. During the post fermentation, P_4, S_4 and PT_4 with increased SBWs MCC concentration caused significant ($p \leq 0.05$) impact on textural parameters such as firmness, consistencey, cohesiveness,work of cohesion and intrinsic viscosity. Textural attributes such as firmness and consistency was improved in 43°C with concentration of 1% MCCs addition as compared to WMC and SMC. Firmness and consistency was more modified in PT_1 and P_1 with 29.54g & 684.51g.s. than control.

Table 4.13 Textural attributes of SBW's extracted MCC incorporated to the low fat stirred yogurt at T_1 and T_2

Samples	Firmness (g)		Consistency(g.s)		Cohesiveness(g)		Work of Cohesion(g.s)		Intrinsic viscosity(g.s)	
	T_1	T_2	T_1	T_2	T_1	T_2	T_1	T_2	T_1	T_2
WMC	20.12^{b}±0.08	22.39^{a}±1.65	418.8^{b}±7.53	676.4^{a}±6.54	-12.14^{b}±0.31	-9.53^{a}±0.925	-3.061^{a}±1.72	-1.085^{a}±0.34	-19.12^{a}±7.29	-2.98^{a}±0.74
SMC	19.74^{a}±0.77	20.47^{abc}±0.42	383.7^{a}±5.88	453.4^{a}±23.4	-11.22^{a}±2.31	-9.18^{a}±0.038	-3.155^{a}±2.34	-8.897^{a}±2.01	-2.642^{a}±0.25	-9.178^{a}±0.038
P_1	16.86^{b}±0.19	19.01^{bc}±1.5	355.6^{e}±34.0	491.45^{cd}±8.9	-9.830^{a}±0.92	-10.96^{ab}±0.41	-0.82^{a}±0	-13.7^{a}±3.82	-3.258^{a}±0.87	-8.948^{b}±0.19
P_2	19.74^{bc}±0.15	20.59^{ab}±1.08	419.5^{cd}±23.8	614^{ab}±23.65	-9.57^{a}±0.35	-9.415^{a}±0.27	-1.30^{a}±0.23	-10.44^{b}±0.37	-2.659^{a}±0.33	-9.102^{b}±0.12
P_3	20.32^{abc}±0.12	24.75^{ab}±0.74	460.8^{cde}±5.47	663.48^{a}±6.41	-12.00^{ab}±0.44	-9.105^{a}±0.12	-1.18^{a}±0.16	-9.98^{b}±0.92	-4.071^{a}±0.5	-9.764^{b}±0.087
P_4	21.66^{a}±1.08	26.45^{a}±2.41	547.8^{bc}±71.5	684.51^{a}±1.52	-14.17^{b}±1.73	-8.95^{a}±0.19	-1.28^{a}±0.062	-7.00^{ab}±0.11	-3.295^{a}±1.28	-10.69^{b}±0.68
PT_1	24.63±0.36	25.07±0.038	481.8±15.59	470.1±0.005	-13.31±0.94	-14.66±0.0081	-1.66±2.58	-3.11±0.08	-12.63±0.3	-35.51±0.43
PT_2	26.14±0.038	26.19±0.108	505.03±2.83	501.37±5.76	-13±2.47	-16.1±0.58	-3.92±2.02	-8.09±1.86	-15.58±2.77	-31.59±5.96
PT_3	26.44±0.179	28.67±0.829	514.69±9.66	555.9±12.21	-14.39±3.44	-11.72±0.36	-9.51±5.97	1.96±0.49	-23.65±8.48	-37.09±0.36
PT_4	28.9±1.12	29.54±0.038	568.23±11.66	576.73±17.71	-11.34±0.28	-12.01±0.25	-1.04±0.62	-0.16±0.4	-11.18±4.8	-29.11±6.06
S_1	19.12±0.76	21.71±0.61	356.64±18.9	430.4±7.17	-11.58±0.79	-14.21±0.6	-1.41±0.39	-0.86±1.99	-26.41±0.37	-9.92±2.94

S_2	21.07±0.62	23.73±1.08	400.5±29.61	409.3±23.34	-8.83±0.32	-11.96±2.46	-0.69±0.09	-2.01±1.31	-20.74±4.308	-18.9±1.78
S_3	21.91±0.62	25.4±0.2	430.33±11.22	480.3±42.29	-12.62±2.14	-10.57±1.62	-1.24±0.22	-1.61±0.33	-19.26±1.27	-15.83±4.27
S_4	23.37±1.56	26.44±0.18	422.32±11.21	531.1±2.45	-9.523±0.87	-11.06±0.43	-1.02±0.14	-1.83±0.29	-25.47±3.18	-15.23±4.34

***Note- the codes termed as SMY- Skim milk control (without addition of MCC), CBY-commericial MCC , PBY-peel MCC, TBY-petiole MCC, SBY-stalk MCC incorporated in low fat stirred yogurt samples. *Note-Results are represented in the form of mean ± Standard deviation(n=3)**

The index of viscosity and the consistency are two related metrics. The thickness of the sample is indicated by consistency, whereas the index of viscosity determines the resistance of the product sample that flow through the disc after backward extrusion. Thus, textural qualities of low fat stirred yoghurt were altered by the incorporation of SBWs MCC during prefermentation. The protein content and total solids in the skim milk influences the consistency of the yogurt by the MCC and protein content as well as the water molecule complex bind strongly to from stable structure to stirred yogurt (Saleh et al., 2020; X. Wang et al., 2019). As compared contrrol samples, consistency was increased significantly high ($P \leq 0.05$) in P_4 and PT_4 with 684.51 and 576.73 g.s respectively.

It supports the idea that 1% SBWs MCC is always the best dose for a reasonable effect on water binding or absorption of water molecules by MCC and linked to milk protein cause the gel structure reinforcement. Similar explanations for the enhanced firmness were suggested in trials involving the incorporation of various byproducts from fruit waste into yogurt, such as passion fruit peel powder and carrot cell wall particles.

Cohesiveness is essential for stirred yogurt due to its semi-firm texture. Cohesiveness assesses the feature of yogurt by requiring a force to remove yogurt that has hung to the spoon/mouth while eating the sample. The yogurt with P_4 and PT_4 samples having the higher cohesiveness values than the control samples. The effect of AP concentration on the viscosity index produced comparable findings. samples. Cohesiveness and intrinsic viscosity also increased by up to 1% concentration of SBWs MCC. From the conslusion, P_4 and PT_4 showed excellent performance in functionality effects such as syneresis, WHC, and textural properties compared to control samples with effective supplementation of 1% powder samples in low fat stirred yogurt. This contribute, economical impact in utilization of SBWs as food additive as fat replacer and stabilizer in yogurt products without altering the its attributes with nutraceutical efficacy.

4.9 Sensory analysis of optizimed low fat plain yogurt samples inaddition of SBWs MCC

A group of 30 semi-trained and untrained sensory assessors used the hedonic scale to conduct the sensory evaluation. The following sensory aspects were assessed for the yogurt samples; appearance, colour, taste, texture, scent, aftertaste, syneresis, and overall acceptability. The texture of yogurt is one of the primary qualities that determines its sensory quality and appearance, mouthfeel, and overall consumer acceptability (Mousavi, Heshmati, Daraei Garmakhany, et al., 2019; Mousavi, Heshmati, Garmakhany, et al., 2019; Vénica et al., 2020). The eight sensory parameters play an important role in sensory evaluation of low fat stirred plain yogurt to find out the effectiveness of added SBW MCC.

The sensory scores give an enlighten on significant difference between the each SBW MCCs and it sensory qualities as compared to control samples. PBY and SBY scored significantly better appearance in yogurt samples with values of 7.8 and 7.45 respectively as compared TBY and control samples. this may due to the better homogenization and network formation between PMCC and SMCC with casein micelle and water molecules without any lump formation (Gonzalez et al., 2011). In case of colour, SBY (7.8) scored highest point as compared to other samples. The aroma in terms of its sourness or yeasty were found significant difference in SBW MCCs and control samples. the control samples (SMY, CMY) scored highest score along with PBY with 7.36, 7.32 and 7.36 respectively. this may due to the off flavour was coming from the SBY and TBY due to the increased sourness as compared PBY and control samples.

According to texture parameter, SBY and CBY showed same scores with 7.2 as compared other samples due to its proper mixing with yogurt sample during stirring and also uniform distribution of MCC among the yogurt composition without imparting the gel structure and also smoothness attracts the consumer's perception too. But one of the

drawback was the after taste of the SBW MCCs and commercial MCC showed least score as compared to SMY (7.08). This may due to improper aggregates cause graininess feeling in the tongue make unfavourable perception to panellist. This is mainly related to particle size distribution of the SBW MCCs and Control MCC. this can be imparted by proper size reduction, uniform stirring, aggregate formation (hygroscopic nature) and sieving of MCC samples (Gonzalez et al., 2011; Kim et al., 2020; Saleh et al., 2020; TELES & FLÔRES, 2007; Vanegas-Azuero & Gutiérrez, 2018). Syneresis is an important criterion for the selection of effective stabilizer, thickening and fat replacing agent. The TBY, SBY and CBY showed significant effect on syneresis parameter with 7.96 and 7.92 points as compared control samples respectively.

Thus proves that, PTMCC and SMCC (petiole and stalk MCC) can improves the microstructure of stirred yogurt with better reduction of the whey separation defect by effective holding capacity of serum and protein molecules with strong interaction helps the stability of the yogurt during the storage. The panellists gave the PBY (Peel MCC) sample the highest ratings for appearance, taste, texture, and overall acceptability was shown in fig 4.18. The selected SBY sample received the greatest scores for colour, texture, and syneresis observance. The panellists found the reduced fat swirled yoghurt to be moderately acceptable. According to the findings, PBY and SBY are the most preferred.

4.9.1 Acceptability index

A product must have an acceptability index more than 70% in order to be called sensory wise acceptable (Lucas et al., 2018a). Based on the results of this index was showed Table 4.14. it was decided that, as compared to control formulation, the yogurts containing 1% SBWs MCC were more acceptable. The formulation with the highest index of acceptance was SBY along with PBY and SMY with 86%, 85.2%and 85.4% respectively. overall acceptability score was highest in PBY and CBY with 7.36 points from the sensory panellist along SBY (7.12) respectively.

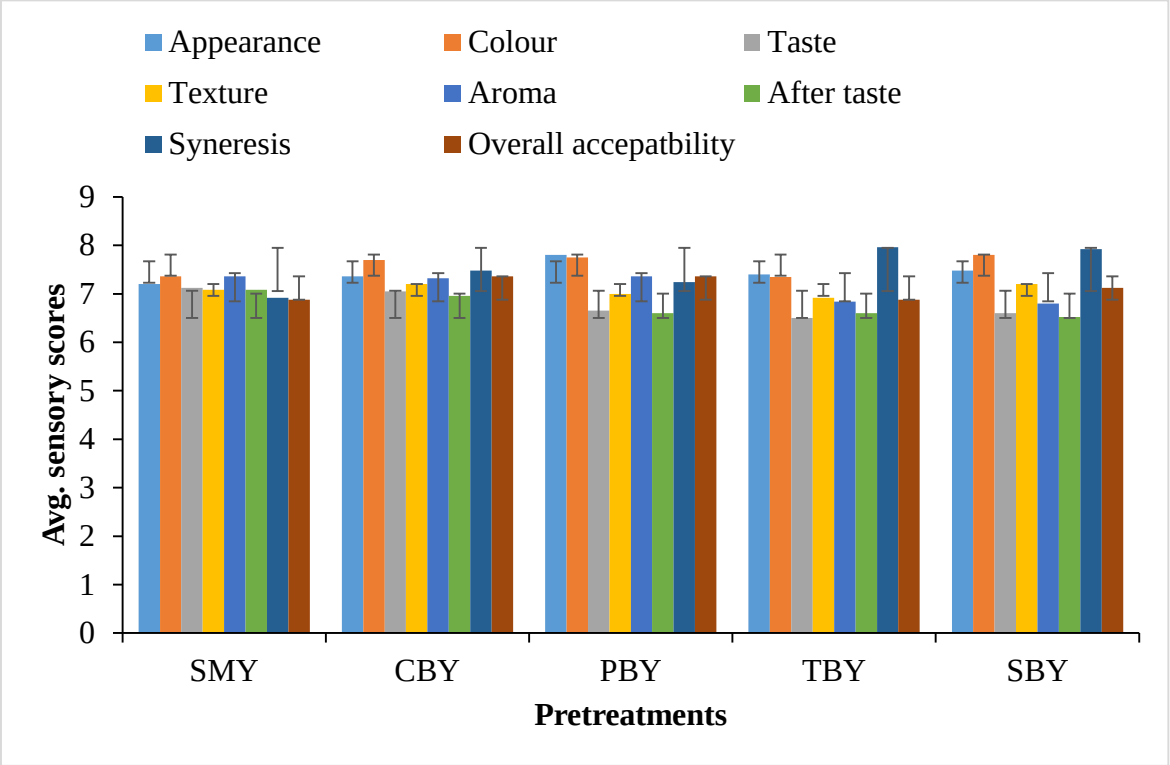


Figure 4.18. Sensorial scores of low fat stirred yougurt incorporated by SBWs MCC.

***Note- the codes termed as SMY- Skim milk control (without addition of MCC), CBY-commericial MCC , PBY-peel MCC, TBY-petiole MCC, SBY-stalk MCC incorporated in low fat stirred yogurt samples. *Note-Results are represented in the form of mean ± Standard deviation(n=3)**

In conclusion, sensory performance of developed low fat stirred yogurt samples along with control samples exposed the consumer perception and acceptance toward the yogurt drink. Most preferred stirred yogurt sample was PBY with average satisfactory scores for appearance, colour and aroma along with SBY. Whereas, TBY showed satisfactory performances in texture and syneresis free yogurt samples but with lowest score in taste due to its aggregate formation (Hasani et al., 2017). Further, panel members give suggestions to improve the sensory attributes such as aroma, taste and after taste by addition of any fruit puree or crush to attract all age groups and improve the score itself.

Table 4.14. Acceptability index (AI) optizimed low fat plain yogurt samples inaddition of SBWs MCC

Samples	Appearance	Colour	Taste	Texture	Aroma	After taste	Syneresis	Overall acceptability	Mean average
SMY	90	92	89	78.7	92	78.7	76.9	86	85.4
CBY	86.7	85.6	83.1	77.8	91.5	87	83.1	81.8	84.9
PBY	92	86.1	88.1	90	92	73.3	80.4	81.8	85.2
TBY	93.5	81.7	72.2	76.9	85.5	82.5	88.4	86	83.3
SBY	93.5	97.5	82.5	90	85	72.4	88	79.1	86.0

***Note- the codes termed as SMY- Skim milk control (without addition of MCC), CBY-commericial MCC , PBY-peel MCC, TBY-petiole MCC, SBY-stalk MCC incorporated in low fat stirred yogurt samples.**

4.10 Effectiveness of SBW MCCs on quality attributes of optimized low fat plain stirred yogurt flavoured with fruit puree and its shelf life

The optimized plain stirred yogurt sample with 1% of SBW MCCs were further modified to improve the sensory attributes by the addition of fruit puree with the recommendation of sensory panellist to increase the consumer preference to all age group with beneficial inputs. Along with that, prepared drinkable LFFSY (low fat fruit flavoured stirred yogurt further stored for the shelf life evaluation to prove the effectiveness of added MCCs to prove its as stabilizer, thickening and fat replacing agent in drinkable low fat yogurt with noble dietary qualities for all age groups.

4.10.1 Selection of fruit concentration for the consumer suitability to promote the SBW's enriched low fat stirred yogurt

Overall acceptability depends upon the flavour, taste and texture and colour is the vital trait which stimuli customer's acceptability. Plain Yogurt is predominantly sour and bland taste due to the lactic acid formed by fermentation process. For better acceptance of product, fruits, flavouring agents and sweeteners are added to yogurt to improve flavour balance and mask partially acetaldehyde flavour of yoghurt and to improve colour. The fruit pulp was extracted from ripened jamun fruit which was converted to fruit crush with TSS of 62°B.

The proximate analysis showed significant changes for fruit pulp to puree was showed in table 4.15. Reduced the moisture content from 84.89% to 14.48% in fruit puree due to the processing method. Also protein, crude fibre were decreased in puree due to the filtration and removal of the peel and seed portions to avoid interference during development of LFFSY. Additionally, the carbohydrate was increased in fruit puree as compared to pulp because of sugar addition with values of 85% (Lubbers et al., 2004; Ścibisz et al., 2019; Turgut & Cakmakci, 2018).

The sensory evaluation was carried out by a group of 30 semi trained and untrained sensory assessors. Samples were coded and panellists were evaluated each sample according to the questionnaire mentioned in the chapter 3 under the section 3.11.3. The overall acceptability of fruit crush concentration was 10% (JBY5) with highest score with values of 7.77 point respectively. Additionally, JBY5 also got highest scores in colour, taste, texture and aroma with 8.03, 7.63, 7.82 and 7.53 points respectively as compared to other concentration as well as the control sample (0% fruit puree)). Also, after taste was preferred with the addition of 10% fruit puree (Kavaya et al., 2019). Whereas, increased concentration of fruit puree (JBY4) gives good appearance to yogurt samples which significantly affected

as compared to other samples were illustrated in fig 4.19. Thus, JBY5 with 10% of fruit was used for the preparation of fruit stirred yogurt along with the addition 1% SBW MCC samples. After the incorporation of the fruit crush along with optimized SBW's MCC (1%) in low fat stirred plain yogurt were further stored in refrigerated condition for 28days. The quality evaluation was done at the intervals of 0, 7, 14 and 28 days (Torrico et al., 2020).

Table 4.15 Physio-chemical properties of standardised fruit pulp and fruit puree

Parameters	Pulp(%)	Fruit puree(25%)
Moisture	84.89±2.2	14.48±1.86
Protein	0.259±0.0	0.129±0.09
Fat	0	0
Total ash	0.6±0.42	0.34±0.47
Fibre	1.22±0.24	0.53±0.02
Carbohydrate	13.161	85
TSS	8.2	62
TA (%)	2.05±0.2	0.106±0.0

***Note- Results were tabulated as Mean±SD with triplicates**

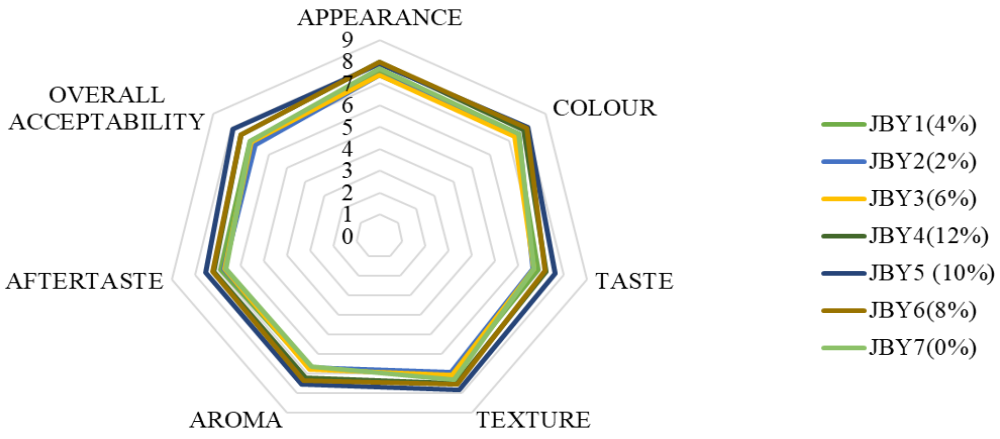


Figure 4.19 Sensory parameters for selection of fruit puree concentration for the consumer suitability to promote the SBW's enriched low fat stirred yogurt. *Note- Results were tabulated as Mean±SD.

4.10.1.1 Acceptability index

The different concentration of the fruit puree was evaluated to find out the AI based on the panel testing using hedonic values. Based on the results of this index was showed in Table 4.16. it was decided that, as compared to control formulation, the yogurts containing 10% of fruit puree (JBY5) were more acceptable. As the average mean, also showed, highest score with 86.30% as compared to other samples. Also other sensory attributes also supported the JBY5 for giving highest scores in colour taste, texture aroma, after taste and also overall acceptability index with 89.3, 84.8, 86.9,83.7, 83.9 and 87.8% respectively. Thus, JBY showed better acceptability index as compared other samples (Lafarga et al., 2019).

Table 4.16. Acceptability index (AI) optizimed low fat fruit yogurt samples inaddition of SBWs MCC

Attributes	JBY 1(4%)	JBY2 (2%)	JBY3 (6%)	JBY4 (12%)	JBY5 (10%)	JBY6 (8%)	JBY7 (0%)
Appearance	83.5	81.9	82.0	88.7	87.8	88.5	85.6
Colour	81.8	83.7	81.5	87.0	89.3	88.3	83.9
Taste	76.2	74.1	83.5	80.3	84.8	79.6	74.4
Texture	79.1	77.0	78.5	83.3	86.9	83.7	81.1
Aroma	74.6	74.1	75.2	80.0	83.7	81.5	73.7
Aftertaste	76.6	74.8	74.4	79.9	83.9	80.4	74.1
Overall Acceptability	77.4	74.4	77.0	82.9	87.8	83.0	77.8
Average mean	78.46	77.14	78.89	83.16	86.30	83.57	78.65

4.10.2 Physio-chemical evaluation of developed low fat plain stirred yogurt flavoured with fruit puree (LFSFY) during the storage period

The physiochemical analysis of developed low fat plain stirred yogurt flavoured with fruit puree provides the information of storage studies on pH, Titratable acidity, TSS and colour

stability were analysed during the storage period. The methods followed for the estimation of physiochemical analysis were prescribed in previous chapter and the results are reported in table 4.17 with graphical representation in figure 4.10 along with scientific reasons behind it.

4.10.2.1 pH and Titratable Acidity

The pH and acidity of low yoghurt are crucial elements in determining its quality. The pH of fruit flavoured low fat stirred yogurt samples with SBWs MCC after 1, 7, 14, and 28 days in the refrigerator conditions were observed in this section. The pH was suddenly dropped in control sample due to the increase in the acidity(Gad et al., 2010; Pontonio et al., 2020; Sobhay et al., 2019). The concentration of SBWs MCC was no significant effect on pH and TA in developed sample.

But, the addition of the fruit puree into the low fat yogurt will increase the pH also the level of acidity has a proportionate connection, as indicated in Table 1. L. acidophilus was able to use flaxseed and make lactic acid. As a result, post-acidification rose. This conclusion was consistent with the findings of, who discovered that the acidity of yoghurt rose due to the addition of fibre and the stimulation of LAB development.

The pH of all samples significantly reduced ($p \leq 0.05$) to 4.5 until it reach 28-day storage period showed in fig 4.20 with the greatest pH decline occurred in control samples, during the first 7 days of cold storage. A gradual decrease of pH was seen in the SBWs MCC and fruit puree added stirred yogurt after the 7^{th} day. Yogurt's titratable acidity increased due to the addition of fruit puree caused lockstep with the drop in pH was showed in Fig. 4.21. The TA reduced significantly ($P \leq 0.05$) and the pH gradually lowered for all treatments at the close to the storage period (day 28).

Within the same storage time, SBWs MCC addition had no influence on TA of yoghurt as compared to the control sample ($p > 0.05$). thus, SBWs MCC and fruit puree added samples were showed better stability during the storage period due to the strong interaction between MCC, casein micelle and water molecules. Also, deterioration can also due to the acidification rate, which must be balanced between gel strength and commercially viable fermentation duration. As the increase in the TA, causes rapid protein aggregation, leading in the production of a modest number of protein-protein linkages and substantial particle/cluster rearranging, due to a weak milk gel with big pores and increased repulsion of whey (Yekta & Ansari, 2019).

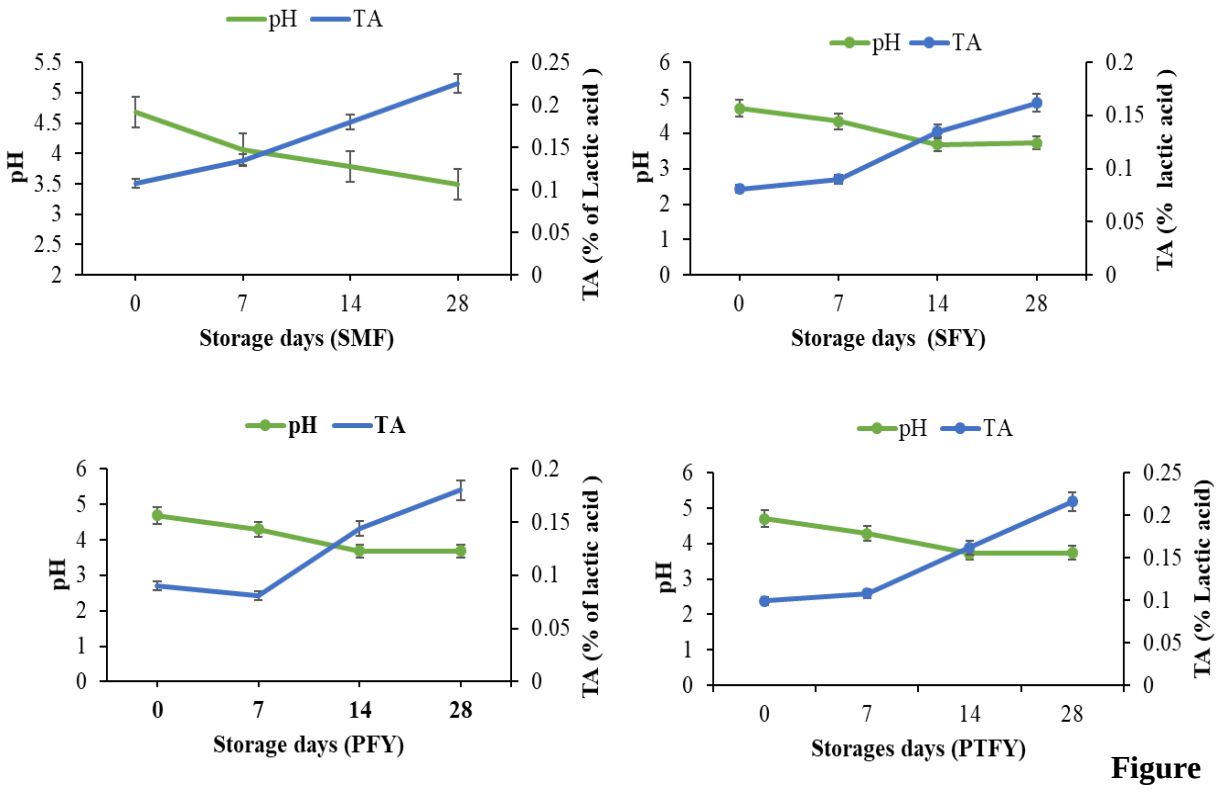


Figure 4.20 Effect of pH v/s Total titratable acidity (TA) of low fat fruit flavoured yogurt samples during storage period. *Note- Results were tabulated as Mean±SD. Treated samples were denoted as SMF-control sample without MCC, SFY-SMCC (1%) with addition of 10% fruit puree, PFY-Peel MCC (1%) with addition of 10% fruit puree, PTFY- petiole MCC (1%) with addition of 10% fruit puree

As in the fig 4.20, a sudden increment in TA and decrease of pH was shown in SMF (control) during storage period 0^{th} to 7^{th} day. Interestingly, TA and pH was showed sudden peak at 14^{th} day of storage period in PFY and SFY with significant stability shown as compared to SMF and PTFY

4.10.2.2 Total soluble solids

After addition of the fruit puree, increase in TSS was found out in all SBWs MCC incorporated samples due to increase in sugar content. As its storage periods starts, TSS in all samples were 17°B was dropped to 12.63, 13. 96, 14.44 and 15.26°B respectively in SMF, PFY, SFY and PTFY at end of the storage period (28^{th} day). Fig 4.21 shows the fluctuation happening in the TSS during the storage period (Mousavi, Heshmati, Daraei Garmakhany, et al., 2019)

4.10.2.3 Colour index

Color is a significant attribute in food since it is the first characteristic observed by customers and hence frequently determines their preference (Jafari et al., 2019; Vénica et al., 2020). The given fig 4.21 illustrate the lightness (L*) of supplemented with or without SBWs MCC of low fat stirred fruit yogurt stored at 4°C for 28 days' storage period. Due to the addition of fruit puree, slight wine red shades was seen in all the samples. the color index was slightly ($P \leq 0.05$) shifted with its addition and slightly reduced with no significant effect on storage as compared to control samples. The highest lightness index was find out in SMF with 82.59 and lowest was in PTFY with 80.5 is detected at the 1^{st} day of the storage condition.

At the end of the storage period, decreased lightness (L*) was in SFY along with PFY as compared with SMF with 77.53 and 78.57% respectively. while in PTFY was more stable throughout the storage period as compared to other samples including control (Yu et al., 2021).

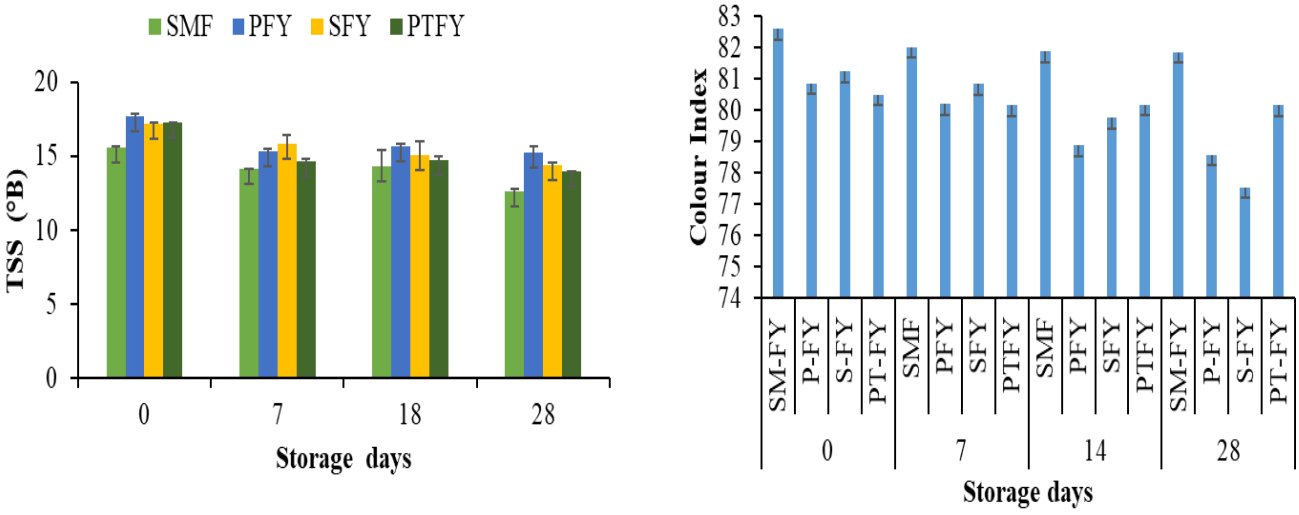


Figure 4.21 Effect of TSS and colour index of LFFSY samples on storage period. ***Note-Results were tabulated as Mean±SD. Treated samples were denoted as SMF-control sample without MCC, SFY-SMCC (1%) with addition of 10% fruit puree, PFY-Peel MCC (1%) with addition of 10% fruit puree, PTFY- petiole MCC (1%) with addition of 10% fruit puree**

4.10.3 Compositional analysis of the low fat stirred fruit yogurt during storage period.

The proximate composition of prepared SBWs MCC optimized low fat stirred fruit yogurt samples during the storage period was shown in fig 4.22. It provides the overall increase/decrease in constituent presence in LFFSY samples. The methods followed for the estimation of proximate analysis were prescribed.

During storage period, moisture content was significant decreased in the SBWs MCC incorporated samples as compared to control sample without MCC addition. Especially, in SFY and PTFY was significantly decreased ($P \leq 0.05$) from 86-74.33% and 85-78.61% during the storage period (28 days). In case of protein content, no significant changes ($P \leq 0.05$) in between samples as compared to control sample. An increases in protein was found in SMF and PFY of 2.92 and 2.75g/100g at the end of the storage period.

Also in case of fibre content, no significant changes among the SBWs MCC samples due to stable structural configuration and no interference between microorganisms (Xu et al., 2019). While the ash content in all samples also maintain stable throughout the storage period. Whereas, carbohydrate content was significantly increased due to the destruction of sugars in to reducing form from the addition of fruit puree in overall samples. Reduction of fat was also visible in all samples due to the increment in microbial load (Srimali et al., 2019).

In conclusion, as the storage period increases, a significant decrease in moisture content as compared to SMF. But in case of protein, no significant difference was found in protein and ash content in yogurt samples. Also, no significant degradation was observed in MCC concentration.

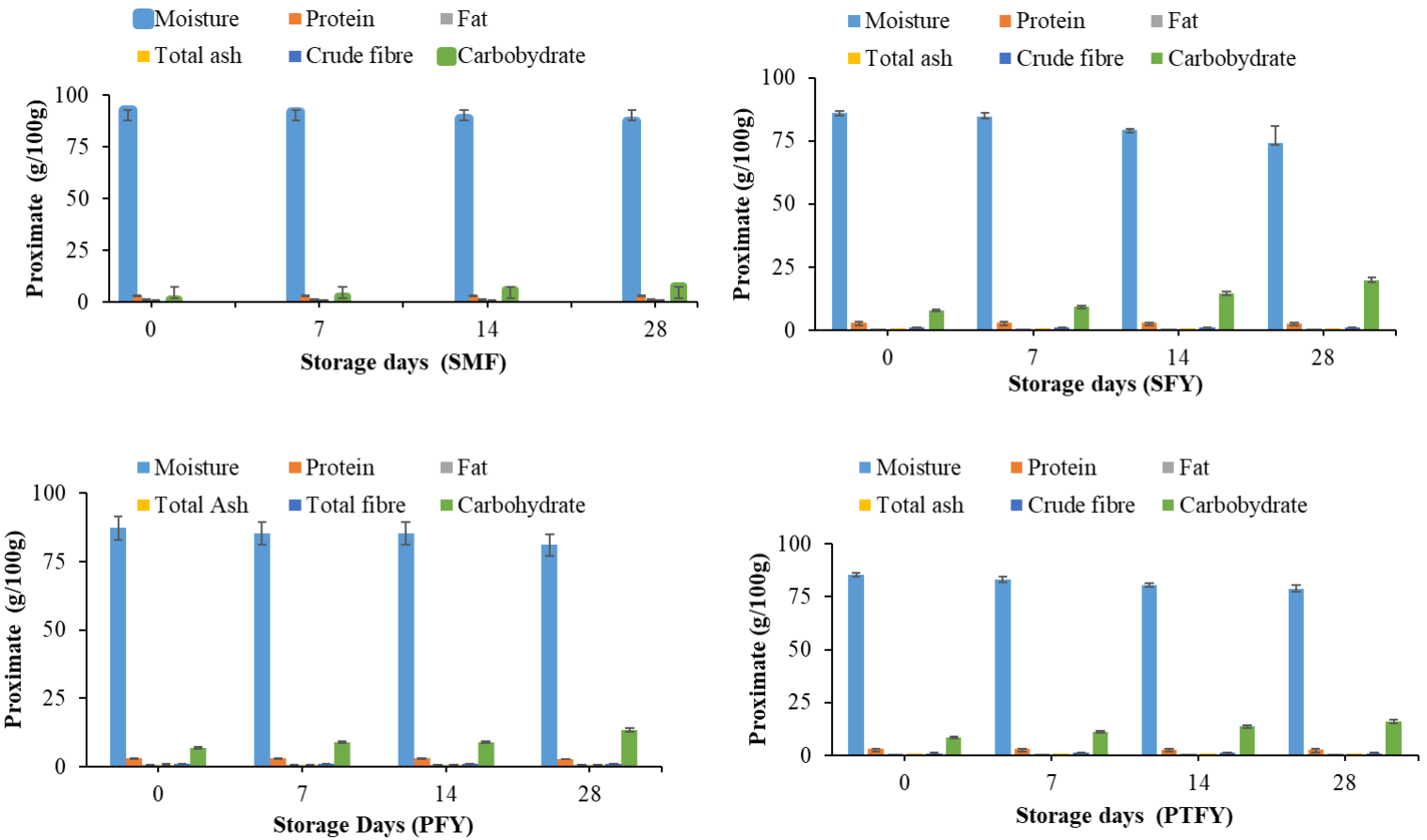


Figure 4.22 Effect on proximal content of LFFSY samples during the storage period. ***Note- Results were tabulated as Mean±SD.**

Treated samples were denoted as SMF-control sample without MCC, SFY-SMCC (1%) with addition of 10% fruit puree, PFY-Peel MCC (1%) with addition of 10% fruit puree, PTFY- petiole MCC (1%) with addition of 10% fruit puree

4.10.4 Microbial quality of low fat stirred fruit yogurt incorporated by SBWs MCC

4.10.4.1 Probiotic efficiency of Lactobacillus Survival

Until the duration of refrigerated storage, the vitality of probiotic lactobacillus is the most essential quality criterion in the finished product. As prior literature, revealed that the LAB count was greater in samples containing increased concentration of flaxseed and kept for a shorter period of time. In other words, the Lactobacillus bulgaricus and Lactobacillus cremoris count was proportional to the nutrient content, temperature and time was shown in Fig.4.23.

Even during days of storage, the Lactobacillus microorganism count drops gradually. To have best potential therapeutic benefits, meals containing probiotic bacteria should have a minimum value of >6 log CFU/g. Moreover, the rate of growth was reduced in SBWs MCC incorporated in low fat stirred yogurt samples than in the control sample. Until the 14^{th} day, increased survival was more seen during the storage period.

The significant increase ($P \leq 0.05$) in the growth count of Lactobacillus count was found in SMF, SFY and PTFY of 8.76, 7.6 and 7.46 CFU/ml respectively in 14^{th} day of the storage period. The end of the storage period, a slight growth reduction of the LAB viable count due to the lowering of pH was observed after 28 days of storage. Fiber, as a stimulant agent, was shown to selectively boost the growth of probiotics such as lactobacillus species during the storage period. To have best potential therapeutic benefits, meals containing probiotic bacteria should have a minimum value of >6 log CFU/g.

It also showed comparable findings regarding the influence of fibre addition on probiotic bacteria survival in probiotic yoghurt supplemented with other sources of fibre extracted from agro-industrial waste source. Previous research has shown that prebiotic fibres such as inulin, oat bran, unripe banana flour, and apple fibre can increase L. casei

development (Mousavi, Heshmati, Daraei Garmakhany, et al., 2019; Mousavi, Heshmati, Garmakhany, et al., 2019).

Past research has shown that product formulation can affected by probiotic bacteria strains, microbial interactions, pH and titratable acidity, inducers and additives, salt, encapsulation, ripening factors, growing conditions, inoculation types, and operating temperature and packing conditions, whole have an impact on the vitality of probiotic bacteria (Karimi et al., 2018).

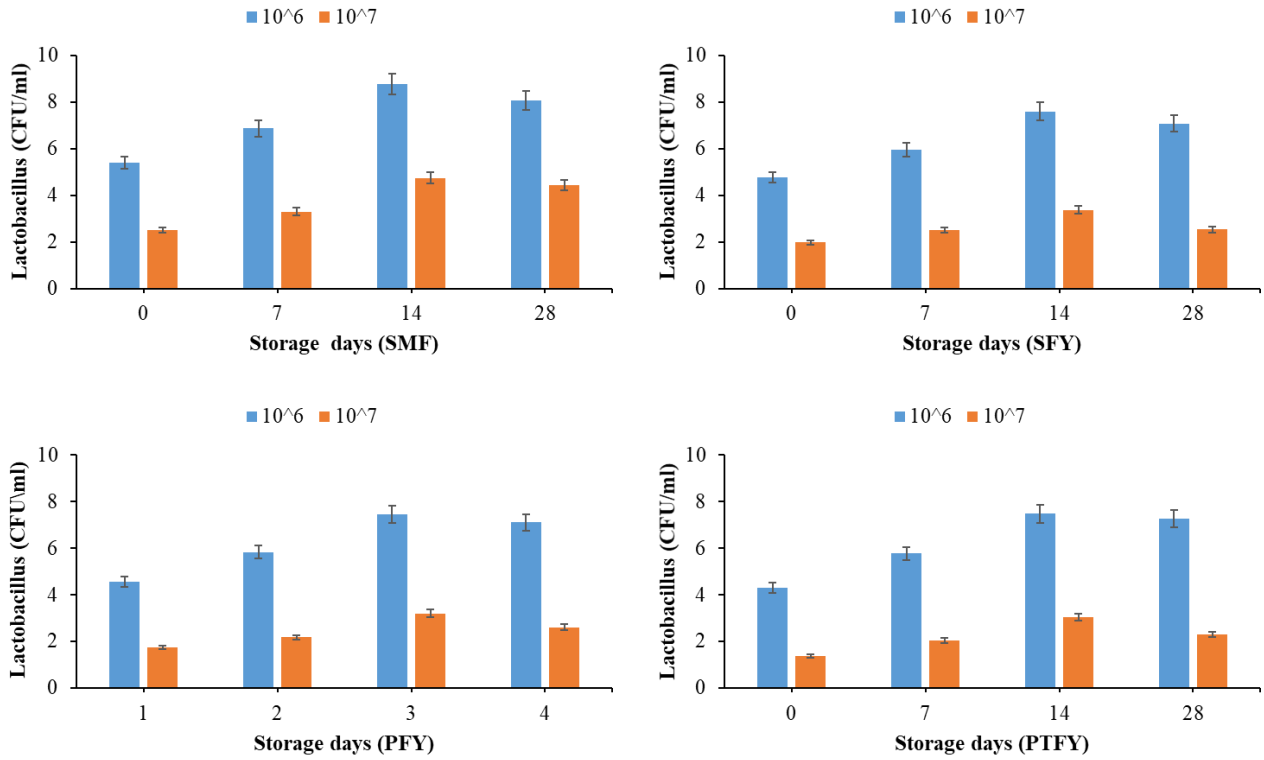


Figure 4.23 Probiotic efficiency of Lactobacillus Survival by incorporation of SBW's MCC in low fat stirred fruit yogurt. ***Note- Results were tabulated as Mean±SD. Treated samples were denoted as SMF-control sample without MCC, SFY-SMCC (1%) with addition of 10% fruit puree, PFY-Peel MCC (1%) with addition of 10% fruit puree, PTFY- petiole MCC (1%) with addition of 10% fruit puree**

4.10.4.2 Total viable count

The total bacterial load will give an account of the overall viable cell count present in the low fat stirred fruit yogurt samples (Turgut & Cakmakci, 2018). However, bacterial load was found out to be higher in control and SFY of 4.43 and 4 CFU/ml respectively at of 10^{-7} dilution of samples. In, SBWs MCC incorporated fruit yogurt samples showed significantly ($p \leq 0.05$) minimal growth load of bacterial count which was illustrated in fig 4.24.

The bacterial count was found to be limited level (10CFU/g) under the FSSAI regulation. Also, SBWs MCC incorporated samples showed significant decrease ($p \leq 0.05$) in bacterial count especially PFY and PTFY as compared to control sample.

4.10.4.3 Mold and yeast count

Total mold and yeast count was varying from sample to sample due to the sample nature and composition difference in the SBWs MCC samples. The mold concentration of prepared yoghurt is determined by the conditions of sample preparation, treatment and packing (HadiNezhad et al., 2013; Kiros et al., 2016).

Highest fungal growth was found in SMF and PTFY with 2.10 sand 1.63 CFU/ml respectively of 10^5 dilution factor was observed in fig 4.25. Least amount of fungal growth was seen in 0.33 and 1.23 in SFY and PFY. The yeast and mold growth increased in storage period can be due to the addition fruit puree which contain sugar molecule. As a result, prepared yoghurt samples were deemed safe for ingestion (limited level of 50CFU/ml).

From the results, it was confirmed that, the presence of mold and yeast in all the samples were maintained as safe level in all the treated samples stored at 4°C for 28 days of storage period. Also, proved that, due to the presence of SBW MCCs, growth of yeast and

mold was delayed during the storage period which approves the addition of SBW MCC have significant impact on yeast growth especially in SFY and PFY (Lee et al., 2017; Slavin, 2013; Turgut & Cakmakci, 2018). Also confirms that samples were prepared and stored in safe condition along with good hygienic practices.

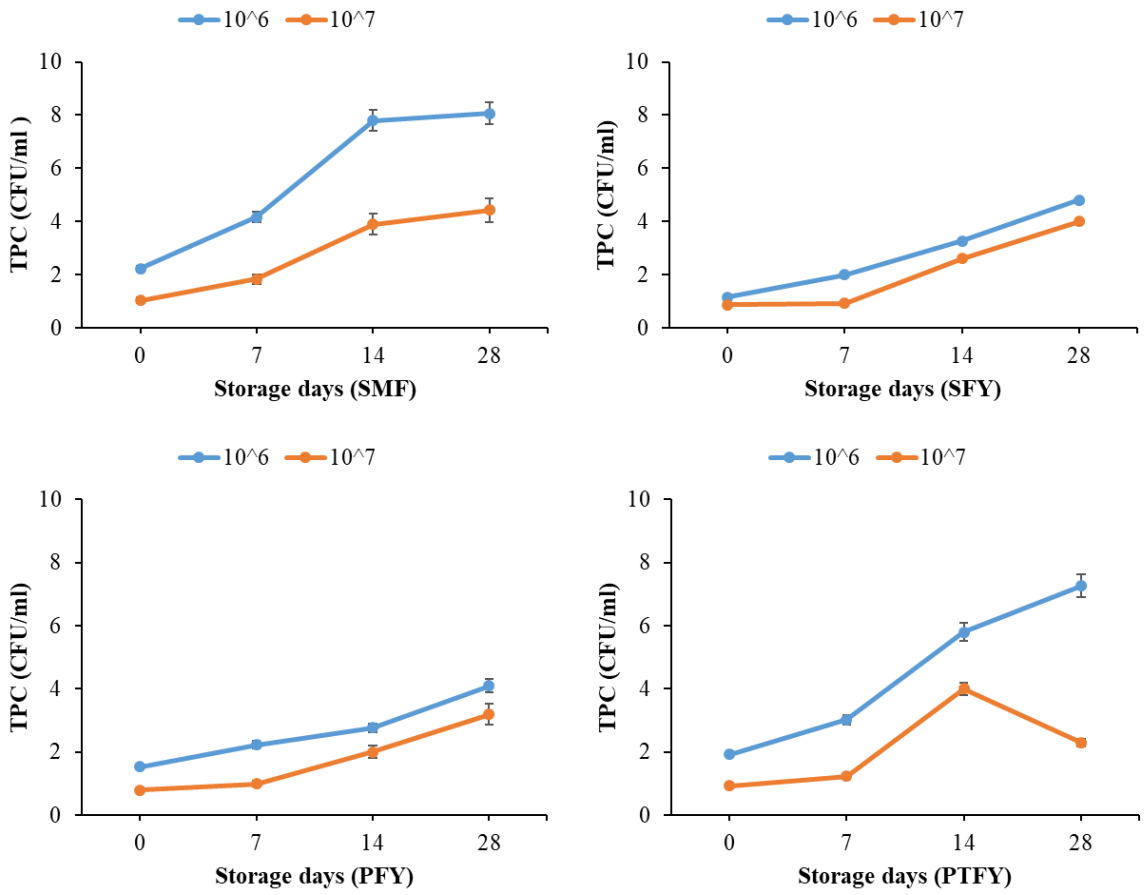


Figure 4.24 Total plate count quantification of low fat stirred fruit yogurt incorporated by SBWs MCC samples. ***Note- Results were tabulated as Mean±SD. Treated samples were denoted as SMF-control sample without MCC, SFY-SMCC (1%) with addition of 10% fruit puree, PFY-Peel MCC (1%) with addition of 10% fruit puree, PTFY- petiole MCC (1%) with addition of 10% fruit puree**

4.10.4.3 Coliform count

During the storage period, no coliforms were found in the prepared samples including control sample. The fecal coliform count of produced samples indicated the overall amount of harmful bacteria present in the yogurt. Coliform is a hazardous bacterium that is mostly found in nature and can cause diarrhea. According to FSSAI, the completed products ought to have coliform less than 10cfu/ml (BSTI) and for international standard, zero coliforms in all food products.

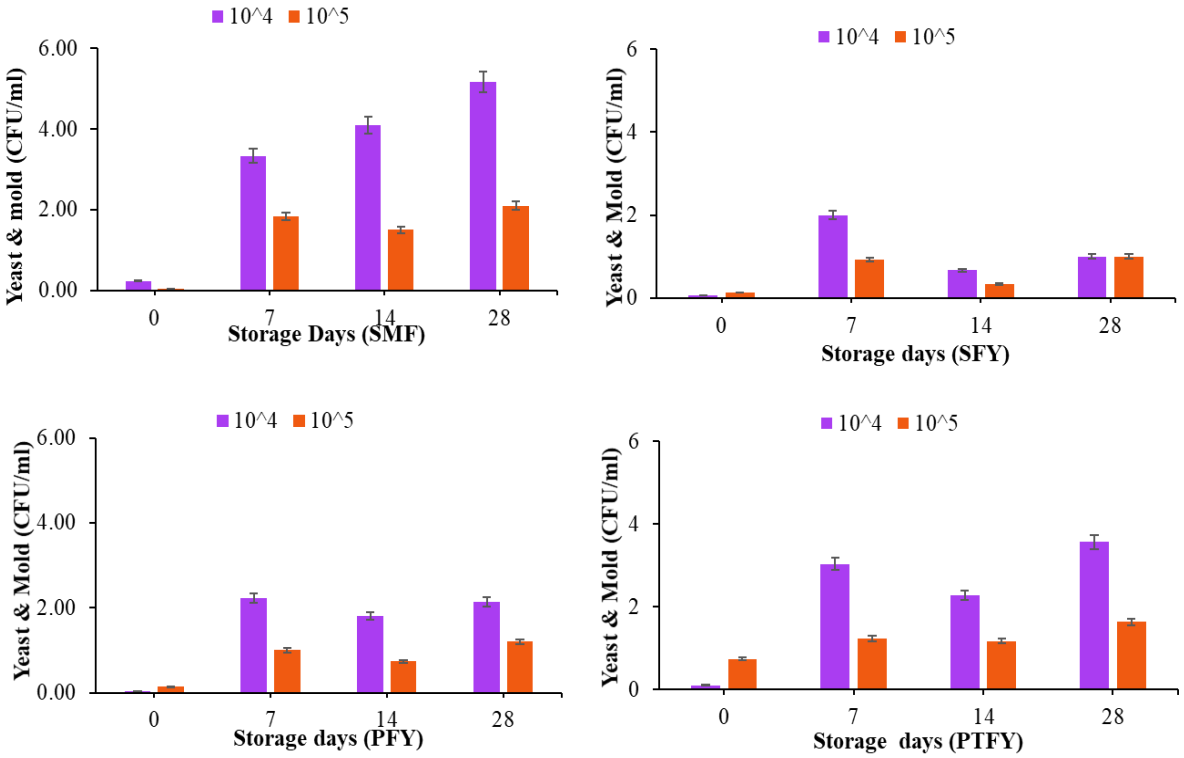


Figure 4.25 Yeast and mold quantification of low fat stirred fruit yogurt incorporated by SBWs MCC samples. ***Note- Results were tabulated as Mean±SD. Treated samples were denoted as SMF-control sample without MCC, SFY-SMCC (1%) with addition of 10% fruit puree, PFY-Peel MCC (1%) with addition of 10% fruit puree, PTFY- petiole MCC (1%) with addition of 10% fruit puree**

4.10.5 Functionality of SBW's MCC as stabilizing and fat replacing agent in low fat stirred yogurt

4.10.5.1 Syneresis index

Syneresis is an unfavourable phenomena that manifests itself as a layer of whey repulsed on the top of yogurt. It is caused by protein aggregation rearrangements that result in thick aggregates that release whey liquid and can be intensified by elevated incubation time, temperature and force. Yogurt mixing will cause disrupts the gel is formed and allows the casein network to rearrange, leading in whey repulsion.

Incorporating the SBWs MCC pre and post fermentation process will decrease yogurt phase separation under refrigerated conditions depending upon the concentration of MCC (Nair et al., 2021; Yousefi & Jafari, 2019). At all storage periods studied, the addition of 1% SBW's MCC diminish the serum separation of yogurt as compared to control sample. The treated samples were poured separated stored in measured centrifuge under refrigerated condition during the storage period. Due to the fruit puree addition cause the sudden reduction of pH within the storage period cause increases the whey repulsion due to the gel shrinkage from the products (Kavaya et al., 2019).

Each treatment's syneresis increased as expected over the 28^{th} day storage period. As a result, SMF (control), showed significant increment ($P \leq 0.05$) of 74.6% in 28^{th} day of the storage of yogurt was illustrated in fig 4.26. While, in addition of SBWs MCC, whey release was significantly controlled by absorption of excess of serum by the MCC samples and made more rigid bonding between casein and water molecules(Gilbert et al., 2020).

Thus, PFY, SFY and SFY showed significant reduction of syneresis by 30.67, 34 and 38.33% respectively observed in 28th day of storage.

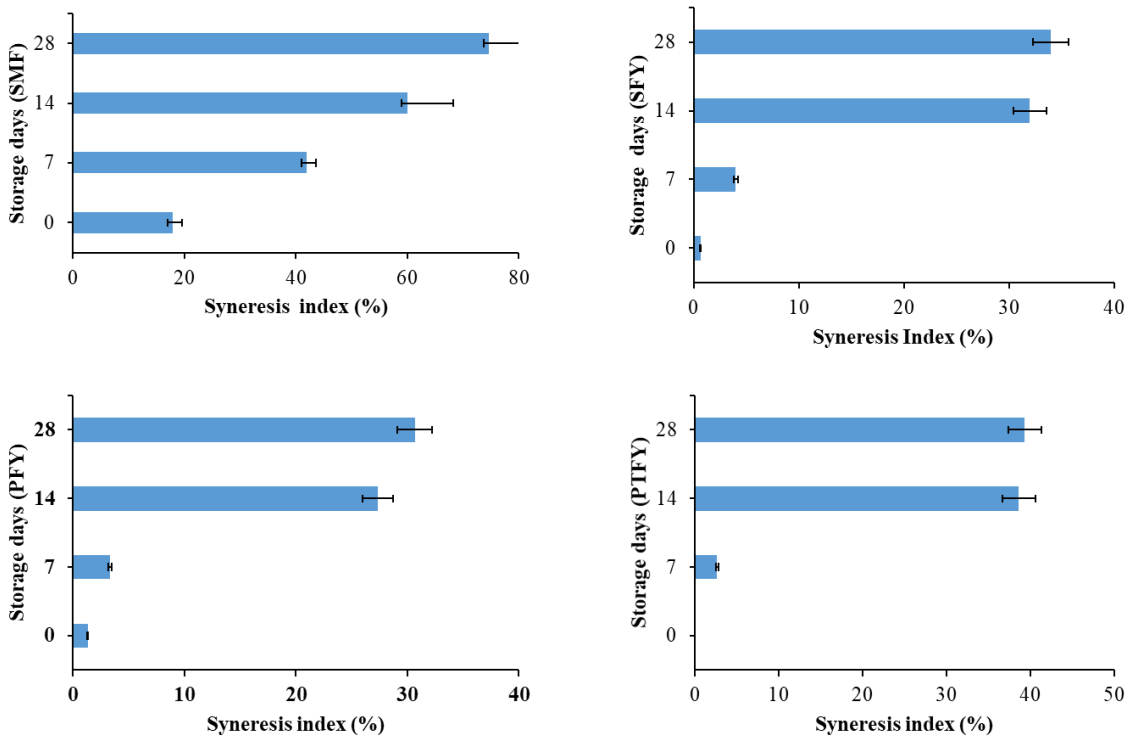


Figure 4.26 syneresis effect on low fat fruit flavoured stirred yogurt by incorporation of SBW's MCC during storage period. *Note- Results were tabulated as Mean±SD. Treated samples were denoted as SMF-control sample without MCC, SFY-SMCC (1%) with addition of 10% fruit puree, PFY-Peel MCC (1%) with addition of 10% fruit puree, PTFY- petiole MCC (1%) with addition of 10% fruit puree

4.10.5.2 Rheological characteristics

Table 4.17 summarises the rheological parameters in terms of flow index (n) and shear stress. The inclusion of SBWs MCC in stirred yogurt will increases viscosity. The Herschel Bulkley model was used to determine that all samples had a flow index n <1 in the initial stage of storage period indicating that the nature of the stirred yogurt with addition of fruit puree and SBWs MCC caused shear thinning (termed as pseudo plastic fluid) due to the increased shear rate during the storage period (X. Wang et al., 2019; L. L. Zhao et al., 2016).

As the end of the storage period, SFY and PTFY showed n>1 which means, shear thickening was happened in the stirred yogurt samples.

While, PFY showed increase during the period of time but showed n>1, ends with shear thinning of the yogurt samples along with control samples. Furthermore, all samples with a higher consistency index demonstrated better gel strength and consistency of the samples as compared to the control sample. Increased K constant values ($P \leq 0.05$) was observed on 14^{th} day of the storage period with 10.643 along with 9.106, and 8.760 respectively in PTFY, SFY and PFY as compared to SMF.

Thus, results were proved that, SBW MCCs showed that a strong hydrogen bonding was formed between the casein molecules to form a network between the protein (Osorio-Arias et al., 2020), MCC polymer and water molecules at end of the storage period under cool condition. PTFY and SFY (1% MCC) samples showed excellent flow behaviour as compared to PFY due to the MCC concentration, stable cellulosic structure and chain length as well the increased zeta potential efficacy as compared to control samples. Also excess firmness also leads to the repulsion of whey which will increase the serum separation

. This can be avoided by the optimized concentration of MCCs in to the yogurt samples. Thus, SBW MCCs can be utilized to performed as a better food additive can improve significantly the viscosity of the yogurt samples.

Table 4.17 Changes in rheological characteristics of low fat fruit flavoured stirred yogurt by incorporation of SBW's MCC during storage period (28days).

Samples	k	n (Pa.s^n)	R^2
		0th	
SMF	4.058±2.57	0.573±0.27	0.905±0.036
PFY	5.271±0.47	0.298±0.047	0.86±0.003

SFY	4.416±0.17	0.463±0.10	0.929±0.018
PTFY	8.778±0.096	0.368±0.093	0.915±0.016
		7^{th}	
SMF	0.281±0.0003	0.9380±0.009	0.998±0.00004
PFY	10.81±1.58	0.356±0.054	0.998±0.0015
SFY	8.983±3.13	0.301±0.08	0.8114±0.12
PTFY	5.626±3.31	0.734±0.14	0.949±0.009
		14^{th}	
SMF	2.221±0.34	0.549±0.063	0.970±0.04
PFY	8.7603±0.0943	0.454±0.05	0.959±0.028
SFY	9.106±0.72	0.383±0.12	0.980±0.009
PTFY	10.64±5.45	0.454±0.304	0.9224±0.06
		28^{th}	
SMF	0.277±0.047	0.853±0.005	0.971±0.005
PFY	0.415±0.08	0.8536±0.037	0.974±0.031
SFY	0.2012±0.027	1.171±0.138	0.988±0.002
PTFY	0.3361±0.21	1.359±0.271	0.998±0.002

***Note- Results were tabulated as Mean±SD. Treated samples were denoted as SMF-control sample without MCC, SFY-SMCC (1%) with addition of 10% fruit puree, PFY-Peel MCC (1%) with addition of 10% fruit puree, PTFY- petiole MCC (1%) with addition of 10% fruit puree**

4.10.5.3 Texture evaluation of optimized stirred fruit yogurt samples in stored conditions

The SBWs MCC and fruit puree addition into the low fat stirred yogurt will significantly affect the textural parameters. Also variation in addition of MCC will also improve the textural characteristics was already discussed in the section 4.8.3. During the storage period, caused a significant fluctuation to the parameters was showed in the table 4.18. All the optimized sample with 1% (PMCC, SMCC, PTMCC) SBWs MCC in the stirred yogurt showed better increased texture and consistency as compared to SMF (without MCC). Increased TSS will improve the firmness and consistency throughout the stored

condition. Thus the incorporation of SBWs MCC will improves the syneresis, textural parameters as well WHC of the yogurt samples.

The amount of force necessary to achieve a particular degree of deformation, known as firmness, is a regularly studied criterion in defining yoghurt smoothness. The firmness values of the samples like SMF, PFY, SFY and PTFY varies from19.51- 17.92, 18.51 - 24.96, 20.22- 23.66 and 20.61- 23.81 respectively during the storage period. As it shown in table 4.19, the control samples showed no significant increment in firmness, but at the end of the storage period, firmness got reduced as compared to samples.

The addition of flaxseed to yoghurt samples increased its firmness substantially, whereas changes in storage time had no effect on its firmness and no significant changes in the hardness of the stirred yogurt samples during the storage period(Mousavi, Heshmati, Daraei Garmakhany, et al., 2019). But, the stability of the SMF was become weaken without the addition of MCC samples. While in case of SBW MCC samples incorporated stirred yogurt along with fruit puree showed significant increase at 14^{th} day of storage period in PFY and PTFY with 25.63±0.033 and 25.19±0.11 respectively.

At end of the storage period, SFY showed slight decrease in firmness by instability in gel matrix due to the increase in TA and pH in stirred yogurt samples. The consistency of the SBWs MCC samples showed significant increase($P \leq 0.05$) during storage mainly PFY and SFY with values of 395.9 - 556.91, 427.9-592.2and 423.9-580.3 g.s respectively.

The intrinsic viscosity results agree with the apparent viscosity results, confirming the hardening effect of SBWs MCC. A significant increase in the cohesiveness also find in all treated samples as compared to control. There was no significant influence on cohesiveness in all treated samples during the storage period at refrigerated conditions. It may be attributed to the high viscosity given by SBWs MCC samples and optimized conditions (both soluble and insoluble components), which reinforces the internal gel

structure. Interestingly, the separated whey and trapped casein clusters formed by shearing were absorbed by SBWs MCC samples, which also reinforced the loose and opened protein structure(Mousavi, Heshmati, Daraei Garmakhany, et al., 2019). Prior literatures showed yogurt containing partially hydrolysed guar gum showed a similar rise in cohesion and viscosity at the same time (Xinya Wang 2019).

From resultants from textural attributes proved that, SBW MCCs showed better stability as compared to control samples due to its excellent absorption capacity along with strong network between the casein molecules to avoid the whey repulsion and give strength to casein matrix formation with smooth texture upto 14^{th} day of storage period. Afterwards, an increment of microbial load, an increase in TA leads to sudden lowering of pH cause weakening of hydrogen boding between the MCC and casein which cause whey separation more.

Thus, overall textural parameters, PFY and PTFY plays a critical role to improve the textural parameters such as firmness, consistency, cohesiveness and intrinsic viscosity as good stabilizing agent to improve the storage stability of low fat fruit flavoured stirred yogurt (LFFSY).

Thus, SBW MCCs samples can be promoted as stabilizer and fat replacing agent and functional ingredient in the field of dairy processing.

Table 4.18 Textural traits of the low fat stirred fruit flavoured yogurt samples inaddition of SBWs MCC during storage period.

Samples	Firmness (g)	Consistency (g.s)	Cohesiveness (g)	Work of Cohesion (g.s)	Intrinsic Viscosity (g.s)
1 week					
SMC	19.51±1.41	395.9±41.4	-10.16±1.45	-1.56±0.56	-40.9±6.67
PFY	18.51±0.32	375.2±17.2	-11.86±0.69	3.56±6.98	-29.91±2.44
SFY	20.22±1.7	427.9±40.4	-9.983±1.31	-1.28±0.71	-34.39±0.47
PTFY	20.61±1.81	423.89±53.3	-9.91±1.52	4.14±7.2	-31.61±0.32
2 week					
SMC	19.31±1.34	383.2±44.35	-11.57±1.56	-1.64±0.33	-58.63±0.61
PFY	23.55±0.074	490.6±12.2	2.29±16.06	-1.71±0.86	-53.04±0.031
SFY	25.22±0.28	529.23±2.57	-9.68±0.33	-1.24±0.18	-55.84±0.08
PTFY	25.5±0.22	537.25±1.61	-9.35±0.47	-1.39±0.104	-49.16±4.02
3 week					
SMC	20.25±1.34	414.6±44.4	-10.47±1.56	-1.4±0.33	-59.06±0.61
PFY	25.63±0.033	544.38±6.82	-9.447±0.43	-1.05±0.21	-42.64±0.004
SFY	21.51±1.96	443.87±55.28	-9.907±1.52	3.15±5.71	-63.9±1.1

PTFY	25.19±0.11	526.89±5.19	-10.23±0.22	-1.37±0.17	-45.63±0.139
4 week					
SMC	17.92±0.58	354.1±23.53	-11.93±0.39	-2.027±0.46	-30.42±1.59
PFY	24.96±4.24	556.91±88.2	-12.85±1.34	9.843±9.16	-64.77±3.64
SFY	23.66±0.54	592.2±3.26	-9.35±1.27	-1.48±0.12	-60.41±0.35
PTFY	23.81±0.94	580.25±20.67	-9.33±0.033	-1.38±0.176	-61.56±3.29

***Note- Results were tabulated as Mean±SD. Treated samples were denoted as SMF-control sample without MCC, SFY-SMCC (1%) with addition of 10% fruit puree, PFY-Peel MCC (1%) with addition of 10% fruit puree, PTFY- petiole MCC (1%) with addition of 10% fruit puree**

4.10.6 Sensory evaluation of low fat stirred fruit flavoured yogurt samples with addition of SBWs MCC

High-quality yogurt should hold substantial aroma and taste as well as retain its curd stability without any defects of shrinkage, lump formation, and serum separation. The scores collected for appearance, flavour, mouthfeel, body and texture, syneresis, and overall acceptability of different yogurt formulations are displayed in the fig 4.27. The sample were coded in three number alphabet were termed as JBY, JFY, JCY, JPY, JSY and JTY (without MCC control, with addition control, with Commercial MCC, PMCC, SMCC and PTMCC sample provide suitable appearance, colour, texture and aroma to the yogurt product.

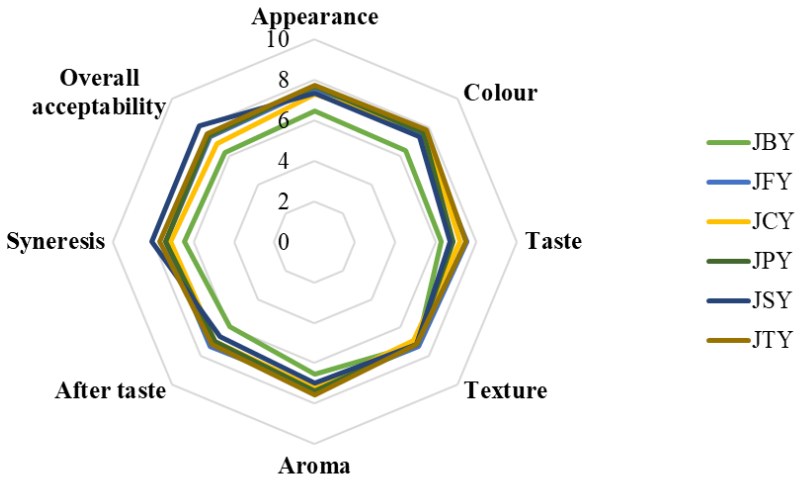


Fig 4.27 Consumer acceptance estimation of low fat fruit flavoured stirred yogurt samples with addition of SBWs MCC using descriptive sensory parameters.

JSY and JPY also showed highest score in texture attributes along JTY. Syneresis separation was not seen the JSY and JPT and most liked by panellists. Overall acceptability is based on complex and multidimensional factors that consists of diverse sensory traits including flavour, texture, and appearance perceptions (DRAKE et al., 2001; Kim et al., 2020; Oliveira et al., 2021; Pérez et al., 2021). Panellists evaluated the overall acceptability of yogurt formulations with scores of 8.1and 7.55 which indicating that they liked JSY and

JPT samples. The following results were proved by the determining the acceptability Index (AI) were showed in table 4.19. The results showed better AI in JSY and JPY of 85.75 and 84.91% respectively.

Table 4.19 Acceptability index (AI) of fruit flavoured low fat stirred yogurt with incorporation of SBWs MCC

Samples	JBY	JFY	JCY	JPY	JSY	JTY
Appearance	80.6	84.4	81.1	85.6	81.7	86.1
Colour	79.4	83.9	86.1	85	81.7	86.7
Taste	78.1	83.9	80	76.1	83.8	83.3
Texture	80	81.1	86.3	89.4	89.4	79.4
Aroma	81.9	80.6	90.6	91.9	87.5	83.9
After taste	74.4	81.1	87.5	86.9	82.5	80
Syneresis	80.6	82.2	79.4	82.2	89.4	85
Overall acceptability	78.1	81.7	76.1	82.2	90	83.9
Mean average	79.1	82.4	83.4	84.9	85.7	83.5

CHAPTER 5

SUMMARY AND CONCLUSIONS

Overall, the dissertation discusses about the types, sources and exploitation of agro-industrial waste from shallot cultivation and processing. Moreover, utilization of lignocellulose extracted from the shallot waste, structure and its modification as well as the application in diverse fields are also discussed. In present work, extraction and purification of cellulose and transformation to microcrystalline cellulose was discussed in depth and also to focus on its application in yogurt products and its characteristics. Efforts were also made to find out to find out its functional excellence as a stabilizer, thickening and fat replacing properties in yogurt samples and give insight for further modification as an additive in other areas. thus, increase the effective utilization of shallot waste to a cost-effective raw material for the extraction of MCC which is economical for producers and processors.

Because of its distinctive taste and strong flavour, *Allium cepa* is widely farmed and consumed as a culinary ingredient around the world. China and India are the two major onion exporters in Asia, accounting for the first (26MMT) and second (22MMT) positions in the global production scenario respectively. One of the most significant variations of *Allium cepa var. aggregatum* is a small-sized onion variety that is generally known as "shallot" or "small onion" and is more famous in southern India, where 75 percent of shallots are farmed, with a production rate of 12 T/ha.

According to recent research, polysaccharides isolated from allium species contain phytonutrients that are useful against a variety of ailments such as diabetes, obesity, cardiovascular disease, cholesterol level inhibition, and allergy infection. During production and processing, 25-30% of the biomass sections are wasted, which are highly perishable and cause significant environmental contamination. Commercial exploitation of these products

will lead to higher revenue as well as jobs, thereby enhancing people's livelihoods. Shallot biowaste is a rich source of dietary fibres, both soluble and insoluble. It can also be a consistent supply of high-value functional compounds that are intended to promote human well-being. Cellulose and microcrystalline or crystalline cellulose, which have gained appeal in many applications, is one of the principal dietary fibres approved by as GRAS. The availability of raw materials for the production of cellulose and microcrystalline cellulose is a major issue.

The main sources of cellulose and its derivatives are hardwood and cotton linters, which increase the risk of deforestation. This can be solved by using biomass from agricultural sources and processing byproducts from plant sources, such as husk, peel, stem, stalk, flower, and so on. According to several studies, agricultural biomass can be a good source of crystalline cellulose. With consistent viscosity, gelling power, emulsification capacity, textural modifier, fat replacer or mimicker, heat resistance, and water binder, it demonstrates multifarious functionality.

Microcrystalline cellulose has a higher water holding capacity than modified cellulose, forming a gel-like structure in yoghurt products that improve consistency, texture, and syneresis. Stirred yoghurt is a drink that is created by homogenising flavoured or unflavoured yoghurt to a smooth consistency and then drinking it. Given that stirred yoghurt is often created with whole milk to ensure smoothness and uniformity, the current study hypothesises that adding MCC to low-fat milk will aid in the development of stirred yoghurt. The produced yogurt's storage stability was also tested in a refrigerated environment.

Salient features of present research

- Shallot biowaste streams (SBWs) are rich source of crude cellulose content of all samples. The crude fibre content of Peel, stalk and petiole were found to be relatively high, with 54.76, 49.51, and 40.73%, respectively
- In untreated SBW fibre, the peel(P) fibre had the highest cellulose content (31.41%) and the lowest lignin content (6.047%) when compared to the stalk(S) and petiole (PT) samples. The soluble fibre content was comparatively high in the Petiole, Peel and Stalk (9.09, 8.11, and 4.29%) respectively.
- MCC extraction is mostly determined by the source, pretreatments, time, temperature, chemical hydrolysis, and cellulose yield percent. In comparison to the ash content of 0.54, 3.32, and 0.77%, autoclave pre-treated α-cellulose samples treated at 120°C for 30 min at 15psi and 2.0N NaOH exhibited good quality of cellulose yield of 29.31, 27.94, and 25.70 % in APM, ASM, and APTM
- Throughout the pre-treatments, 21.98, 17.36, and 16.92 percent MCC were recovered from AS, AP, and WPT, with lower ash contents of 0.065, 0.185, and 1.195 percent, respectively. While the lightness index (L*) in selected MCC samples of C (commercial MCC), APT (autoclave pretreated petiole), AP (autoclave pretreated petiole), and AS (autoclave pretreated petiole) were substantially better than hot water pre-treatment.
- The observation revealed that, overall, MCC recovery was found to be relatively high in AS, AP, and WPT with the values of 79.39, 59.23, and 55.63% respectively
- The FTIR spectra revealed the shifting of peaks and functional groups presence in SBWs MCC samples in the range of 4000-400cm^{-1}. The most prominent, characteristic band detected between 1430-1429, 1364, 1157, and 895cm^{-1} showed a

sharp peak bending symmetric CH_2 and asymmetric C1-H representing a pure crystalline cellulose band.

- The structural damages in MCC samples of WPT were discovered to be larger due to the reaction time, temperature along with chemical reaction. Moreover, non-cellulosic components were removed and converted cellulose microfibrils to short microfibrils of MCC by acid treatments. The autoclave pre-treated MCC samples had distinct cleavage, porous and even surfaces. Whereas, the surface of the APT MCC samples grew more porous due to the destruction of amorphous phase by acid hydrolysis. Crystalline phases persisted more stable and maintained their structural integrity in all particles. The autoclave-treated samples of APMCC & ASMCC more enlarged particle and fibrous irregular-shaped structures that resembled to commercial MCC.
- The mean particle size varies amongst samples, ranging from 16.14 to 36.34μm. As compared to the C-MCC (17.56), AS, APT, and WS had average particle size of 16.14, 21.82 and 24.84μm,
- The major peaks of the C, WP, AP, and AS MCC samples were observed at 2θ=15°, 22°, and 34° were connected to the crystallographic planes of 110, 200, and 040.
- Using the peak intensity technique, Crystallinity index was significantly higher in C, APT, AS, and WPT, with 78.28, 79.93, 76.75, and 83.74%, respectively. When compared to the control MCC, APT, AS and WPT have excellent CI of 83.68, 82.50, and 81.53%, respectively, using the peak area or convulsion method.
- The size of the crystallite is also an essential component that is related to the CI. Also, reveals the average crystallite size in crystallographic planes of extracted MCC of SBWs. The CI and crystallite size of the autoclave pre-treated samples performed better than C-MCC.

- In DSC, the highest thermal resistance was found in MCC samples of AS, AP and WPT with 263, 259 and 255 respectively as compared commercial MCC. The result showed satisfactory thermal stability as well as the purity of extracted MCC samples as compared to the control (C) MCC sample. Also the source of the raw material, hydrothermal pre-treatments and alkali-acid hydrolysis affect the thermal stability of the extracted MCC samples from shallot bio waste.
- Thus, in overall structural, crystallization and thermal characterization, AS, AP and APT showed excellent values as compared to commercial MCC. Thus present study showed that higher crystalline cellulose can be extracted from the shallot bio-waste while comparing with other source of agro-industrial bio-waste.
- Low fat stirred yogurt was prepared from skim milk with the addition of SBW's MCC (0.25 to 1.0%) at two different temperatures, 37˚C (T_1) & 45°C (T_2). The addition of MCC at given concentrations had no marked effect on the value trends of pH, TA and total solids contents as compared to SMC (Skim milk control) and C-MCC (Commercial MCC). The addition of SBW's MCC had not significantly affected the whole fermentation process.
    - Rheological parameters like flow index(n) showed improvements as compared to Commercial MCC samples and blank samples.
    - The flow index was increased in the T_2 treated sample with an increasing concentration of MCC. The highest flow index was observed in PT_4 and S_4 (1%) with 5.947 and 4.866 respectively at T_2 than C-MCC and SMC.
    - Textural parameters are vital sensory attributes for the acceptance of consumers. The highest firmness was observed in the PT4 samples with 29.54g at T_2 respectively with insignificant difference ($p<0.05$) between control samples. In the case of consistency, the samples treated at T_2 showed increased values mainly in P_4,

WMC and P_3 with values of 684.51, 675.4 and 576.7g.s respectively with no significant difference from other samples.

- In each interval of time, serum separation was higher in SMC with 68% at T_1. While at T_2, the same phenomenon was observed with 66% at 15min. The least serum separation of 40, 42.7% was observed after 15 min at T_2 by addition of MCC to PT_4 and P_4 as compared to SMC. A higher rate of syneresis was found in SMC at 68 % (T_2). While MCC added samples, PT_4 and P_4 were more stable than other concentrations.
- Overall acceptability depends upon the flavor, taste and texture and colour is the vital trait that stimuli customer's acceptability. Plain Yogurt is predominantly sour and bland taste due to the lactic acid formed by the LAB fermentation. For the enhanced acceptance of the product, fruit based puree or crush are added to yoghurt to improve flavor and colour attributes of yogurt samples.
- The overall acceptability of fruit crush concentration was 10% (JBY5) with highest score values of 7.77 points. After the incorporation of the fruit crush along with optimized SBW's MCC (1%) in low fat stirred plain yogurt was further stored in refrigerated condition for 28days. The quality evaluation was done at the intervals of 0, 7, 14 and 28 days. The addition of the fruit crush after the fermentation decreased the pH and significantly decreased in all yogurt samples during the end of the cold storage period. Also vice versa for the TA (Titratable acidity). TA and pH was showed sudden peak at 14^{th} day of storage period in PFY and SFY with significant stability shown as compared to SMF and PTFY.
- The TSS, moisture, protein and fat showed slightly reduced during storage, while SBW's MCC in each sample remain constant in concentration as compared to SMC. The colour parameters of MCC's incorporated stirred yogurt showed a slight

decrease as compared to SMF. PTFY showed better lightness as compared to PFY and SFY. In conclusion, as the storage period increases, a significant decrease in moisture content as compared to SMF was observed, no significant difference was found in protein and ash content in yogurt samples. Also, no significant degradation was observed in MCC concentration.

- During the storage period, the syneresis rate was significantly decreased up to the 7^{th} day as compared to SMF. Also, the syneresis rate was significantly increased after the 14^{th} day. But as compared to control and SMF, the syneresis rate was lowered in PFY and SFY. The probiotic survival of Lactobacillus was observed on the 14^{th} day of the product storage period. SMF showed the highest probiotic growth along with SFY and PTFY of 8.73, 7.6 and 7.46 CFU/ml. It also showed comparable findings regarding the influence of fibre addition on probiotic bacteria survival in probiotic yoghurt supplemented with other sources of fibre extracted from agro-industrial waste source.
- Total bacterial load was found to be within the limited level (10CFU/g) under the FSSAI regulation. Also, SBWs MCC incorporated samples showed significant decrease ($p \leq 0.05$) in bacterial count especially PFY and PTFY as compared to control sample.
- It was confirmed that, the presence of mold and yeast in all the samples were maintained as safe level in all the treated samples stored at 4°C for 28 days of storage period. Also, proved that, due to the presence of SBW MCCs, growth of yeast and mold was delayed during the storage period which approves the addition of SBW MCC have significant impact on yeast growth especially in SFY and PFY.
- According to FSSAI, the completed products ought to have coliform less than 10cfu/ml (BSTI) and for international standard, zero coliforms in all food products.

During the storage period, no coliforms were found in the prepared samples including control sample.

- Thus, PFY, SFY and SFY showed significant reduction of syneresis by 30.67, 34 and 38.33% respectively observed on 28th day of storage.
- In SBWs MCC added samples, whey release was significantly controlled by absorption of excess of serum by the MCC samples and made more rigid bonding between casein and water molecule. Thus, PFY, SFY and SFY showed significant reduction of syneresis by 30.67, 34 and 38.33% respectively observed on 28th day of storage as compared to control samples.
- PTFY and SFY (1% MCC) samples showed excellent flow behaviour as compared to PFY due to the MCC concentration, stable cellulosic structure and chain length as well the increased zeta potential efficacy as compared to control samples. Thus, SBW MCCs can be utilized to performed as a better food additive which can improve significantly the viscosity of the yogurt samples.
- Overall textural parameters, PFY and PTFY plays a critical role to improve the textural parameters such as firmness, consistency, cohesiveness and intrinsic viscosity as good stabilizing agent to improve the storage stability of low fat fruit flavoured stirred yogurt (LFFSY). Thus, SBW MCCs samples can be promoted as stabilizer and fat replacing agent and functional ingredient in the field of dairy processing.
- The scores were composed of quality attributes of different yogurt formulations. Panelists evaluated the acceptability of developed stirred yogurts samples with sensory scores of 8.1and 7.55 points which is indicating that they liked JSY and JPT samples. The results showed better AI in JSY and JPY of 85.75 and 84.91% respectively.

- Overall, the extraction of MCC by hydrothermal pre-treatments in which low-concentrated bleaching and alkali-acid hydrolysis were beneficial for the transformation of cellulose to MCC. As a result, shallot bio wastes are potentially low-cost sources of cellulose and MCC that are also environmentally benign. Thus, SBW MCCs samples demonstrated superior food additives as stabilizers, fat-replacing agents, and beneficial components in the field of dairy processing.

Future works

The findings of the present research give an insight towards future works and can be suggested on the following possible research areas.

- Novel technologies can be utilized for improvement in the extraction efficiency
- The crystallographic phases and structural modification and polymerization of extracted SBWs MCC to be evaluated
- Effect of SBW MCC samples can be combined with other hydrocolloids to increase functionality and nutraceutical applications
- Applications in formulation of SBWs MCC based hydrogel and aerogel as stabilizer and pickering emulsion agent.
- Studies on in-vivo effects of SBWs MCC on the human body

REFERENCE

Adel, A. M., Abd El-Wahab, Z. H., Ibrahim, A. A., & Al-Shemy, M. T. (2011). Characterization of microcrystalline cellulose prepared from lignocellulosic materials. Part II: Physicochemical properties. *Carbohydrate Polymers*, *83*(2), 676–687. https://doi.org/10.1016/j.carbpol.2010.08.039

Agarwal, U. P., Ralph, S. A., Reiner, R. S., & Baez, C. (2018). New cellulose crystallinity estimation method that di ff erentiates between organized and crystalline phases. *Carbohydrate Polymers*, *190*(March), 262–270. https://doi.org/10.1016/j.carbpol.2018.03.003

Ahmad, A., Anjum, F. M., Zahoor, T., Nawaz, H., & Din, A. (2009). Physicochemical and functional properties of barley b-glucan as affected by different extraction procedures. *International Journal of Food Science and Technology*, *44*(1), 181–187. https://doi.org/10.1111/j.1365-2621.2008.01721.x

Ahmed-Haras, M. R., Kao, N., Ward, L., & Islam, M. S. (2020). Insights into the production and physicochemical properties of oxycellulose microcrystalline with coexisting crystalline forms. *International Journal of Biological Macromolecules*, *146*, 150–161. https://doi.org/10.1016/j.ijbiomac.2019.12.083

Ahsan, H. M., Zhang, X., Li, Y., Li, B., & Liu, S. (2019). Surface modification of microcrystalline cellulose: Physicochemical characterization and applications in the Stabilization of Pickering emulsions. *International Journal of Biological Macromolecules*, *132*, 1176–1184. https://doi.org/10.1016/J.IJBIOMAC.2019.04.051

Ahvenainen, P., Kontro, I., & Svedström, K. (2016). Comparison of sample crystallinity determination methods by X-ray diffraction for challenging cellulose I materials. *Cellulose*, *23*(2), 1073–1086. https://doi.org/10.1007/s10570-016-0881-6

Akatan, K. (2022). Highly-efficient Isolation of Microcrystalline Cellulose and

Nanocellulose From Sun Flower Seeds Waste via Environmentally Benign “ soft ” Method. *Cellulose 2022*, 1–16. https://doi.org/10.1007/S10570-022-04527-4

Alam, A., Al Arif Jahan, A., Bari, M. S., Khandokar, L., Mahmud, M. H., Junaid, M., Chowdhury, M. S., Khan, M. F., Seidel, V., & Haque, M. A. (2022). Allium vegetables: Traditional uses, phytoconstituents, and beneficial effects in inflammation and cancer. *Critical Reviews in Food Science and Nutrition*, 1–35. https://doi.org/10.1080/10408398.2022.2036094

Alba, K., MacNaughtan, W., Laws, A. P., Foster, T. J., Campbell, G. M., & Kontogiorgos, V. (2018). Fractionation and characterisation of dietary fibre from blackcurrant pomace. *Food Hydrocolloids*, *81*, 398–408. https://doi.org/10.1016/j.foodhyd.2018.03.023

Alotabi, M. D., Alshammari, B. A., Saba, N., Alothman, O. Y., Kian, L. K., Khan, A., & Jawaid, M. (2020). Microcrystalline Cellulose from Fruit Bunch Stalk of Date Palm: Isolation and Characterization. *Journal of Polymers and the Environment*, *28*(6), 1766–1775. https://doi.org/10.1007/s10924-020-01725-8

Altemimi, A. B. (2018). Extraction and optimization of potato starch and its application as a stabilizer in yogurt manufacturing. *Foods*, *7*(2). https://doi.org/10.3390/foods7020014

Amaro, C., Alves, L. P., Malheiro, T., Rafaela, A., Barros, A., & Lima, G. De. (2019). an Alternative Source of Cellulose for Production of Pharmaceutical Dosage Forms and Biomaterials : Extraction and Characterization. *Polymers*, 1–13.

Amin, M. C. I. M., Abadi, A. G., & Katas, H. (2014). Purification, characterization and comparative studies of spray-dried bacterial cellulose microparticles. *Carbohydrate Polymers*, *99*, 180–189. https://doi.org/10.1016/j.carbpol.2013.08.041

AOAC. (1995). Official Method of Analysis of AOAC Intl. 16. In *Maryland: Association*

of Official Analytical Chemists (21st ed.). AOAC international.

APEDA. (n.d.). *ONION*. Retrieved April 30, 2022, from http://apeda.in/agriexchange/prodgallery/PrdProfile_moa.aspx?hscode=07031010

Askari-Khorasgani, O., & Pessarakli, M. (2020). Evaluation of cultivation methods and sustainable agricultural practices for improving shallot bulb production – a review. *Journal of Plant Nutrition*, *43*(1), 148–163. https://doi.org/10.1080/01904167.2019.1659329

Azubuike, C. P., Odulaja, J. O., & Okhamafe, A. O. (2012). Physicotechnical, spectroscopic and thermogravimetric properties of powdered cellulose and microcrystalline cellulose derived from groundnut shells. *Journal of Excipients and Food Chemicals*, *3*(3), 106–115.

Azubuike, C. P., & Okhamafe, A. O. (2012). Physicochemical, spectroscopic and thermal properties of microcrystalline cellulose derived from corn cobs.pdf. *Journal of Recycling of Organic Waste in Agriculture*, *1*(9), 1–7.

Bai, C., Huang, X., Xie, F., & Xiong, X. (2018). Microcrystalline Cellulose Surface-Modified with Acrylamide for Reinforcement of Hydrogels. *ACS Sustainable Chemistry and Engineering*, *6*(9), 12320–12327. https://doi.org/10.1021/ACSSUSCHEMENG.8B02781/ASSET/IMAGES/MEDIUM/SC-2018-02781Q_0009

Bandi, R., Gangapuram, B. R., Dadigala, R., Eslavath, R., Singh, S. S., & Guttena, V. (2016). Facile and green synthesis of fluorescent carbon dots from onion waste and their potential applications as sensor and multicolour imaging agents. *RSC Advances*, *6*(34), 28633–28639. https://doi.org/10.1039/C6RA01669C

Baruah, J., Deka, R. C., & Kalita, E. (2020). Greener production of microcrystalline cellulose (MCC) from Saccharum spontaneum (Kans grass): Statistical optimization.

International Journal of Biological Macromolecules, *154*, 672–682. https://doi.org/10.1016/j.ijbiomac.2020.03.158

Bede, D., & Zaixiang, L. (2020). Dietary Polysaccharides from Allium Species: A Critical Review in Dietary Polysaccharides from Allium Species: Extraction, Characterization, Bioactivity, And Potential Utilization. *Acta Scientific Agriculture*, *4*(2), 01–15. https://doi.org/10.31080/asag.2020.04.0780

Bedrníček, J., Laknerová, I., Linhartová, Z., Kadlec, J., Samková, E., Bárta, J., Bártová, V., Mráz, J., Pešek, M., Winterová, R., Vrchotová, N., Tříska, J., & Smetana, P. (2019). Onion waste as a rich source of antioxidants for meat products. *Czech Journal of Food Sciences*, *37*(4), 268–275. https://doi.org/10.17221/68/2018-CJFS

BeMiller, J. N. (2019). Cellulose and Cellulose-Based Hydrocolloids. In *Carbohydrate Chemistry for Food Scientists* (pp. 223–240). https://doi.org/10.1016/b978-0-12-812069-9.00008-x

Benitez, V., Mollá, E., Martín-Cabrejas, A., López-Andréu, F. J., Downes, K., Terry, L. A., & Esteban, R. M. (2011). Study of Bioactive Compound Content in Different Onion Sections. *Plant Food for Human Nitrition*, *66*(1), 48–57.

Benítez, V., Mollá, E., Martín-Cabrejas, M. A., Aguilera, Y., López-Andréu, F. J., Cools, K., Terry, L. A., & Esteban, R. M. (2011). Characterization of Industrial Onion Wastes (A*llium cepa L.*): Dietary Fibre and Bioactive Compounds. *Plant Foods for Human Nutrition*, *66*(1), 48–57. https://doi.org/10.1007/s11130-011-0212-x

Beroual, M., Boumaza, L., Mehelli, O., Trache, D., Tarchoun, A. F., & Khimeche, K. (2021). Physicochemical Properties and Thermal Stability of Microcrystalline Cellulose Isolated from Esparto Grass Using Different Delignification Approaches. *Journal of Polymers and the Environment*, *29*(1), 130–142. https://doi.org/10.1007/s10924-020-01858-w

Beroual, M., Trache, D., Mehelli, O., Boumaza, L., Tarchoun, A. F., Derradji, M., & Khimeche, K. (2021). Effect of the Delignification Process on the Physicochemical Properties and Thermal Stability of Microcrystalline Cellulose Extracted from Date Palm Fronds. *Waste and Biomass Valorization*, *12*(5), 2779–2793. https://doi.org/10.1007/s12649-020-01198-9

Bhandari, K., Adaval, A., Bhattacharyya, A. R., & Maulik, S. R. (2020). Synthesis of Microcrystalline Cellulose from Carpenter Waste and Its Characterizations. *Journal of Natural Fibers*, 1–15. https://doi.org/10.1080/15440478.2020.1788688

Bhandari, K., Roy Maulik, S., & Bhattacharyya, A. R. (2020a). Synthesis and Characterization of Microcrystalline Cellulose from Rice Husk. *Journal of The Institution of Engineers (India): Series E*, *101*(2), 99–108. https://doi.org/10.1007/s40034-020-00160-7

Bhat, S. V., Deva, A. M., & Amin, T. (2018). Physicochemical and textural properties of yogurt fortified with psyllium (Plantago ovate) husk. *Journal of Food Processing and Preservation*, *42*(2), 1–9. https://doi.org/10.1111/jfpp.13425

Bhosale, Y. K., Perumal, T., Varghese, S. M., Vincent, H., & Ramachandran, S. V. (2022a). Utilization of shallot bio-waste (Allium cepa L. var. aggregatum) fractions for the production of functional cookies. *International Journal of Food Engineering*, *18*(1), 27–39. https://doi.org/10.1515/ijfe-2021-0169

Bhosale, Y. K., Varghese, S. M., Thivya, P., Hema, V., & Sinija, V. R. (2020). Studies on assessment of safety and nutritional quality of shallot waste fractions. *Journal of Food Processing and Preservation*, e15147. https://doi.org/10.1111/jfpp.15147

Bicu, I., & Mustata, F. (2011). Cellulose extraction from orange peel using sulfite digestion reagents. *Bioresource Technology*, *102*(21), 10013–10019. https://doi.org/10.1016/j.biortech.2011.08.041

Bicu, I., & Mustata, F. (2013). Optimization of isolation of cellulose from orange peel using sodium hydroxide and chelating agents. *Carbohydrate Polymers*, *98*(1), 341–348. https://doi.org/10.1016/j.carbpol.2013.06.009

Buchilina, A., & Aryana, K. (2021). Physicochemical and microbiological characteristics of camel milk yogurt as influenced by monk fruit sweetener. *Journal of Dairy Science*, *104*(2), 1484–1493. https://doi.org/10.3168/jds.2020-18842

Buffiere, J., Balogh-Michels, Z., Borrega, M., Geiger, T., Zimmermann, T., & Sixta, H. (2017). The chemical-free production of nanocelluloses from microcrystalline cellulose and their use as Pickering emulsion stabilizer. *Carbohydrate Polymers*, *178*, 48–56. https://doi.org/10.1016/J.CARBPOL.2017.09.028

Çalışkan Koç, G., & Özçıra, N. (2019). Chemical composition, functional, powder, and sensory properties of tarhana enriched with wheat germ. *Journal of Food Science and Technology*, *56*(12), 5204–5213. https://doi.org/10.1007/s13197-019-03989-y

Chadorshabi, S., Hallaj-Nezhadi, S., & Ghasempour, Z. (2022). Red onion skin active ingredients, extraction and biological properties for functional food applications. *Food Chemistry*, *386*, 132737. https://doi.org/10.1016/J.FOODCHEM.2022.132737

Chai, S. Y., Abbasiliasi, S., Lee, C. K., Ibrahim, T. A. T., Kadkhodaei, S., Mohamed, M. S., Hashim, R., & Tan, J. S. (2018). Extraction of fresh banana waste juice as non-cellulosic and non-food renewable feedstock for direct lipase production. *Renewable Energy*, *126*, 431–436. https://doi.org/10.1016/j.renene.2018.03.050

Chang, C. C., & Li, R. (2019). Agricultural waste. *Water Environment Research*, *91*(10), 1150–1167. https://doi.org/10.1002/wer.1211

Charoenchai L, C Luprasong, D. M. (2018). Characterization of some organosulfur compound in shallot bulb. *Thai Journal of Pharmaceutical Sciences (TJPS)*, *42*(supplement), 93–97.

http://www.tjps.pharm.chula.ac.th/proceedings/backend/proceeding_file/24_PN_FP_Sukanya Settharaksa (102-106).pdf

Chen, B., Cai, Y., Liu, T., Huang, L., Zhao, X., Zhao, M., Deng, X., & Zhao, Q. (2020). Formation and performance of high acyl gellan hydrogel affected by the addition of physical-chemical treated insoluble soybean fiber. *Food Hydrocolloids*, *101*, 105526. https://doi.org/10.1016/j.foodhyd.2019.105526

Chen, H. (2014). Biotechnology of lignocellulose: Theory and practice. In *Biotechnology of Lignocellulose: Theory and Practice*. https://doi.org/10.1007/978-94-007-6898-7

Chen, L., Ding, X., He, Z., Huang, Z., Kunnath, K. T., Zheng, K., & Davé, R. N. (2018). Surface engineered excipients: I. improved functional properties of fine grade microcrystalline cellulose. *International Journal of Pharmaceutics*, *536*(1), 127–137. https://doi.org/10.1016/j.ijpharm.2017.11.060

Cheng, L., Zhang, X., Hong, Y., Li, Z., Li, C., & Gu, Z. (2017). Characterisation of physicochemical and functional properties of soluble dietary fibre from potato pulp obtained by enzyme-assisted extraction. *International Journal of Biological Macromolecules*, *101*, 1004–1011. https://doi.org/10.1016/j.ijbiomac.2017.03.156

Choi, I. S., Cho, E. J., Moon, J. H., & Bae, H. J. (2015a). Onion skin waste as a valorization resource for the by-products quercetin and biosugar. *Food Chemistry*, *188*, 537–542. https://doi.org/10.1016/j.foodchem.2015.05.028

Collazo-Bigliardi, S., Ortega-Toro, R., & Chiralt Boix, A. (2018). Isolation and characterisation of microcrystalline cellulose and cellulose nanocrystals from coffee husk and comparative study with rice husk. *Carbohydrate Polymers*, *191*, 205–215. https://doi.org/10.1016/j.carbpol.2018.03.022

Costa, M. F., Pimentel, T. C., Guimaraes, J. T., Balthazar, C. F., Rocha, R. S., Cavalcanti, R. N., Esmerino, E. A., Freitas, M. Q., Raices, R. S. L., Silva, M. C., & Cruz, A. G.

(2019). Impact of prebiotics on the rheological characteristics and volatile compounds of Greek yogurt. *Lwt*, *105*(November 2018), 371–376. https://doi.org/10.1016/j.lwt.2019.02.007

Current Status | Ministry of New and Renewable Energy, Government of India. (n.d.). Retrieved February 23, 2022, from https://www.mnre.gov.in/bio-energy/current-status#energy_o

Dahlawi, S. M., Nazir, W., Iqbal, R., Asghar, W., & Khalid, N. (2020). Formulation and characterization of oil-in-water nanoemulsions stabilized by crude saponins isolated from onion skin waste. *RSC Advances*, *10*(65), 39700–39707. https://doi.org/10.1039/d0ra07756a

Dai, H., Chen, Y., Ma, L., Zhang, Y., & Cui, B. (2021). Direct regeneration of hydrogels based on lemon peel and its isolated microcrystalline cellulose: Characterization and application for methylene blue adsorption. *International Journal of Biological Macromolecules*, *191*, 129–138. https://doi.org/10.1016/J.IJBIOMAC.2021.09.063

Dai, H., Li, X., Du, J., Ma, L., Yu, Y., Zhou, H., Guo, T., & Zhang, Y. (2020). Effect of interaction between sorbitol and gelatin on gelatin properties and its mechanism under different citric acid concentrations. *Food Hydrocolloids*, *101*, 105557. https://doi.org/10.1016/j.foodhyd.2019.105557

Das, H., & Singh, S. K. (2004). Useful Byproducts from Cellulosic Wastes of Agriculture and Food Industry - A Critical Appraisal. *Critical Reviews in Food Science and Nutrition*, *44*(2), 77–89. https://doi.org/10.1080/10408690490424630

Das, K., Ray, D., Bandyopadhyay, N. R., Ghosh, T., Mohanty, A. K., & Misra, M. (2009). A study of the mechanical, thermal and morphological properties of microcrystalline cellulose particles prepared from cotton slivers using different acid concentrations. *Cellulose*, *16*(5), 783–793. https://doi.org/10.1007/s10570-009-9280-6

Dhingra, D., Michael, M., Rajput, H., & Patil, R. T. (2012). Dietary fibre in foods: A review. In *Journal of Food Science and Technology* (Vol. 49, Issue 3, pp. 255–266). https://doi.org/10.1007/s13197-011-0365-5

Diarsa, M., & Gupte, A. (2021). Preparation, characterization and its potential applications in Isoniazid drug delivery of porous microcrystalline cellulose from banana pseudostem fibers. *3 Biotech*, *11*(7). https://doi.org/10.1007/S13205-021-02838-0

Ding, Q., Li, Z., Wu, W., Su, Y., Sun, N., Luo, L., Ma, H., & He, R. (2020). Physicochemical and functional properties of dietary fiber from Nannochloropsis oceanica: A comparison of alkaline and ultrasonic-assisted alkaline extractions. *LWT*, *133*(February), 110080. https://doi.org/10.1016/j.lwt.2020.110080

Dong, W., Wang, D., Hu, R., Long, Y., & Lv, L. (2020). Chemical composition, structural and functional properties of soluble dietary fiber obtained from coffee peel using different extraction methods. *Food Research International*, *136*, 109497. https://doi.org/10.1016/j.foodres.2020.109497

Drake, M. A., Gerard, P. D., & Chen, X. Q. (2001). Effects Of Sweetener, Sweetener Concentration, And Fruit Flavor On Sensory Properties Of Soy Fortified Yogurt. *Journal of Sensory Studies*, *16*(4), 393–405. https://doi.org/10.1111/j.1745-459X.2001.tb00309.x

Dyk, J. S. Van, Gama, R., Morrison, D., Swart, S., & Pletschke, B. I. (2013). Food processing waste : Problems , current management and prospects for utilisation of the lignocellulose component through enzyme synergistic degradation. *Renewable and Sustainable Energy Reviews*, *26*, 521–531. https://doi.org/10.1016/j.rser.2013.06.016

El-Sakhawy, M., & Hassan, M. L. (2007). Physical and mechanical properties of microcrystalline cellulose prepared from agricultural residues. *Carbohydrate Polymers*, *67*(1), 1–10. https://doi.org/10.1016/j.carbpol.2006.04.009

El-sayed, S., Mahmoud, K. H., Fatah, A. A., & Hassen, A. (2011). DSC , TGA and dielectric properties of carboxymethyl cellulose / polyvinyl alcohol blends. *Physica B: Physics of Condensed Matter*, *406*(21), 4068–4076. https://doi.org/10.1016/j.physb.2011.07.050

Espírito-Santo, A. P., Lagazzo, A., Sousa, A. L. O. P., Perego, P., Converti, A., & Oliveira, M. N. (2013). Rheology, spontaneous whey separation, microstructure and sensorial characteristics of probiotic yoghurts enriched with passion fruit fiber. *Food Research International*, *50*(1), 224–231. https://doi.org/10.1016/j.foodres.2012.09.012

Fang, D., Wang, Q., Chen, C., Li, Z., Li, S., Chen, W., & Zheng, Y. (2020). Structural characteristics, physicochemical properties and prebiotic potential of modified dietary fibre from the basal part of bamboo shoot. *International Journal of Food Science and Technology*, 0–1. https://doi.org/10.1111/ijfs.14709

FAO. (2018). Food Loss Analysis: Causes and Solutions; FAO: Rome, Italy. *Food Loss Analysis: Causes and Solutions - Case Studies in the Small-Scale Agriculture and Fisheris Subsector*, *May*, 1–72. http://www.fao.org/3/a-az568e.pdf

Fattorusso, E., Iorizzi, M., Lanzotti, V., & Taglialatela-Scafati, O. (2002). Chemical composition of shallot (*Allium ascalonicum Hort.*). *Journal of Agricultural and Food Chemistry*, *50*(20), 5686–5690. https://doi.org/10.1021/JF020396T

Fawcett, T. G., Crowder, C. E., Kabekkodu, S. N., Needham, F., Kaduk, J. A., Blanton, T. N., Petkov, V., Bucher, E., & Shpanchenko, R. (2013). Reference materials for the study of polymorphism and crystallinity in cellulosics. *Powder Diffraction*, *28*(1), 18–31. https://doi.org/10.1017/S0885715612000930

Fazilah, N. F., Ariff, A. B., Khayat, M. E., Rios-Solis, L., & Halim, M. (2018). Influence of probiotics, prebiotics, synbiotics and bioactive phytochemicals on the formulation of functional yogurt. *Journal of Functional Foods*, *48*(July), 387–399.

https://doi.org/10.1016/j.jff.2018.07.039

Feng, Y. H., Cheng, T. Y., Yang, W. G., Ma, P. T., He, H. Z., Yin, X. C., & Yu, X. X. (2018). Characteristics and environmentally friendly extraction of cellulose nanofibrils from sugarcane bagasse. *Industrial Crops and Products*, *111*(October 2017), 285–291. https://doi.org/10.1016/j.indcrop.2017.10.041

Fitriana, N., & Susandarini, R. (2019). Short Communication: Morphology and taxonomic relationships of shallot (*Allium cepa L.* group aggregatum) cultivars from Indonesia. *Biodiversitas Journal of Biological Diversity*, *20*(10), 2809–2814. https://doi.org/10.13057/biodiv/d201005

Food and Agriculture Organization of the United Nations (FAO). (2018). *FAOSTAT_data_7-23-2020 (1)*.

Gad, A. S., Kholif, A. M., & Sayed, A. F. (2010). Evaluation of the nutritional value of functional yogurt resulting from combination of date palm syrup and skim milk. *American Journal of Food Technology*, *5*(4), 250–259. https://doi.org/10.3923/ajft.2010.250.259

Galanakis, C. M. (2015). Food Waste Recovery: Processing Technologies and Industrial Techniques. In *Food Waste Recovery: Processing Technologies and Industrial Techniques*. https://doi.org/10.1016/C2013-0-16046-1

Galiwango, E., Abdel Rahman, N. S., Al-Marzouqi, A. H., Abu-Omar, M. M., & Khaleel, A. A. (2019). Isolation and characterization of cellulose and α-cellulose from date palm biomass waste. *Heliyon*, *5*(12), e02937. https://doi.org/10.1016/j.heliyon.2019.e02937

Gao, Y., Zhou, Y., Zhang, Q., Zhang, K., Peng, P., Chen, L., & Xiao, B. (2017). Hydrothermal extraction, structural characterization, and inhibition HeLa cells proliferation of functional polysaccharides from Chinese tea Zhongcha 108. *Journal of Functional Foods*, *39*, 1–8. https://doi.org/10.1016/j.jff.2017.09.057

Ghaemi, F., Abdullah, L. C., & Ariffin, H. (2019). Lignocellulose Structure and the Effect on Nanocellulose Production. In *Lignocellulose for Future Bioeconomy*. Elsevier. https://doi.org/10.1016/B978-0-12-816354-2.00002-5

Gibis, M., Schuh, V., & Weiss, J. (2015). Food Hydrocolloids Effects of carboxymethyl cellulose (CMC) and microcrystalline cellulose (MCC) as fat replacers on the microstructure and sensory characteristics of fried beef patties. *Food Hydrocolloids*, *45*, 236–246. https://doi.org/10.1016/j.foodhyd.2014.11.021

Gilbert, A., Rioux, L. E., St-Gelais, D., & Turgeon, S. L. (2020). Characterization of syneresis phenomena in stirred acid milk gel using low frequency nuclear magnetic resonance on hydrogen and image analyses. *Food Hydrocolloids*, *106*(April), 105907. https://doi.org/10.1016/j.foodhyd.2020.105907

Gontard, N., Sonesson, U., Birkved, M., Majone, M., Bolzonella, D., Celli, A., Angellier-Coussy, H., Jang, G.-W., Verniquet, A., Broeze, J., Schaer, B., Batista, A. P., & Sebok, A. (2018a). A research challenge vision regarding management of agricultural waste in a circular bio-based economy. *Critical Reviews in Environmental Science and Technology*, *48*(6), 614–654. https://doi.org/10.1080/10643389.2018.1471957

Gontard, N., Sonesson, U., Birkved, M., Majone, M., Bolzonella, D., Celli, A., Angellier-Coussy, H., Jang, G. W., Verniquet, A., Broeze, J., Schaer, B., Batista, A. P., & Sebok, A. (2018b). A research challenge vision regarding management of agricultural waste in a circular bio-based economy. *Critical Reviews in Environmental Science and Technology*, *48*(6), 614–654. https://doi.org/10.1080/10643389.2018.1471957

Gonzalez, N. J., Adhikari, K., & Sancho-Madriz, M. F. (2011). Sensory characteristics of peach-flavored yogurt drinks containing prebiotics and synbiotics. *LWT - Food Science and Technology*, *44*(1), 158–163. https://doi.org/10.1016/j.lwt.2010.06.008

HadiNezhad, M., Duc, C., Han, N. F., & Hosseinian, F. (2013). Flaxseed Soluble Dietary

Fibre Enhances Lactic Acid Bacterial Survival and Growth in Kefir and Possesses High Antioxidant Capacity. *Journal of Food Research*, *2*(5), 152. https://doi.org/10.5539/jfr.v2n5p152

Hakeem, K. R., Jawaid, M., & Alothman, O. Y. (2015). Agricultural biomass based potential materials. In *Agricultural Biomass Based Potential Materials*. https://doi.org/10.1007/978-3-319-13847-3

Hamza, H. J. (2014). In vitro antimicrobial activity of garlic, onion, garlic-onion combination (aquatic and oil) extract on some microbial pathogens in Babylon Province, Iraq. *World Journal of Pharmacy and Pharmaceutical Sciences*, *3*(8), 65–78. http://www.wjpps.com/wjpps_controller/abstract_id/1731%5Cnhttp://ovidsp.ovid.com/ovidweb.cgi?T=JS&CSC=Y&NEWS=N&PAGE=fulltext&D=cagh&AN=20143284121%5Cnhttp://oxfordsfx.hosted.exlibrisgroup.com/oxford?sid=OVID:caghdb&id=pmid:&id=doi:&issn=2278-4357&isbn=&vo

Harini, K., Ramya, K., & Sukumar, M. (2018). Extraction of nano cellulose fibers from the banana peel and bract for production of acetyl and lauroyl cellulose. *Carbohydrate Polymers*, *201*, 329–339. https://doi.org/10.1016/j.carbpol.2018.08.081

Hasani, S., Sari, A. A., Heshmati, A., & Karami, M. (2017). Physicochemical and sensory attributes assessment of functional low-fat yogurt produced by incorporation of barley bran and Lactobacillus acidophilus. *Food Science and Nutrition*, *5*(4), 875–880. https://doi.org/10.1002/fsn3.470

Hashemi, K., & Hosseini, E. (2021). The stabilizing and prebiotic potential of water-soluble phase of bitter almond gum exudate in probiotic yogurt drink. *Carbohydrate Polymers*, *255*(September), 117395. https://doi.org/10.1016/j.carbpol.2020.117395

Hassan, S. S., Williams, G. A., & Jaiswal, A. K. (2018). Emerging Technologies for the Pretreatment of Lignocellulosic Biomass School of Food Science and Environmental

Health , College of Sciences and Health , Dublin School of Biological Sciences , College of Sciences and Health , Dublin Institute of. *Bioresource Technology*, *April*. https://doi.org/10.1016/j.biortech.2018.04.099

He, S., Tang, M., Sun, H., Ye, Y., Cao, X., & Wang, J. (2019). Potential of water dropwort (Oenanthe javanica DC.) powder as an ingredient in beverage: Functional, thermal, dissolution and dispersion properties after superfine grinding. *Powder Technology*, *353*, 516–525. https://doi.org/10.1016/j.powtec.2019.05.048

He, X., Lu, W., Sun, C., Khalesi, H., Mata, A., Andaleeb, R., & Fang, Y. (2021). Cellulose and cellulose derivatives: Different colloidal states and food-related applications. *Carbohydrate Polymers*, *255*, 117334. https://doi.org/10.1016/j.carbpol.2020.117334

Heng, P. W. S., & Koo, O. M. Y. (2001). A study of the effects of the physical characteristics of microcrystalline cellulose on performance in extrusion spheronization. *Pharmaceutical Research*, *18*(4), 480–487. https://doi.org/10.1023/a:1011054310895

Hindi, S., & Z Hindi, S. S. (2017). Microcrystalline cellulose: The inexhaustible treasure for pharmaceutical industry. *Nanoscience and Nanotechnology Research*, *4*(1), 17–24. https://doi.org/10.12691/nnr-4-1-3

Horticulture :: Vegetables::Small onion. (2015). *Horticulture :: Vegetables::Small onion*. https://agritech.tnau.ac.in/horticulture/horti_vegetables_small onion.html

Huang, X., Wang, L., Zhang, J., Du, X., Wu, S., Wang, H., & Wei, X. (2020). A novel ε-polylysine-modified microcrystalline cellulose based antibacterial hydrogel for removal of heavy metal. *International Journal of Biological Macromolecules*, *163*, 1915–1925. https://doi.org/10.1016/J.IJBIOMAC.2020.09.047

Ibrahim, M. M., El-Zawawy, W. K., Jüttke, Y., Koschella, A., & Heinze, T. (2013). Cellulose and microcrystalline cellulose from rice straw and banana plant waste: Preparation and characterization. *Cellulose*, *20*(5), 2403–2416.

https://doi.org/10.1007/s10570-013-9992-5

Ilyas, R. A., Sapuan, S. M., & Ishak, M. R. (2018). Isolation and characterization of nanocrystalline cellulose from sugar palm fibres (*Arenga Pinnata)*. *Carbohydrate Polymers*, *181*, 1038–1051. https://doi.org/10.1016/j.carbpol.2017.11.045

Jafari, S. M., Vakili, S., & Dehnad, D. (2019). Production of a Functional Yogurt Powder Fortified with Nanoliposomal Vitamin D Through Spray Drying. *Food and Bioprocess Technology*, *12*(7), 1220–1231. https://doi.org/10.1007/s11947-019-02289-9

Jahan, M. S., Saeed, A., He, Z., & Ni, Y. (2011). Jute as raw material for the preparation of microcrystalline cellulose. *Cellulose*, *18*(2), 451–459. https://doi.org/10.1007/s10570-010-9481-z

Jaya, E. R., Yohanes Parlindungan Situmeang, & Anak Agung Sagung Putri Risa Andriani. (2021). Effect of Biochar from Urban Waste and Eco-enzymes on Growth and Yield of Shallots (*Allium ascalonicum, L*). *SEAS (Sustainable Environment Agricultural Science)*, *5*(2), 105–113. https://doi.org/10.22225/seas.5.2.3871.105-113

Jia, X., Chen, Y., Shi, C., Ye, Y., Abid, M., Jabbar, S., Wang, P., Zeng, X., & Wu, T. (2014). Rheological properties of an amorphous cellulose suspension. *Food Hydrocolloids*, *39*, 27–33. https://doi.org/10.1016/j.foodhyd.2013.12.026

Jiang, G., Ramachandraiah, K., Wu, Z., Li, S., & Eun, J. B. (2020). Impact of ball-milling time on the physical properties, bioactive compounds, and structural characteristics of onion peel powder. *Food Bioscience*, *36*(July 2019), 100630. https://doi.org/10.1016/j.fbio.2020.100630

Jo, S., Park, S., Oh, Y., Hong, J., Kim, H. J., Kim, K. J., Oh, K. K., & Lee, S. H. (2019). Development of Cellulose Hydrogel Microspheres for Lipase Immobilization. *Biotechnology and Bioprocess Engineering 2019 24:1*, *24*(1), 145–154. https://doi.org/10.1007/S12257-018-0335-0

Ju, X., Bowden, M., Brown, E. E., & Zhang, X. (2015). An improved X-ray diffraction method for cellulose crystallinity measurement. *Carbohydrate Polymers*, *123*, 476–481. https://doi.org/10.1016/j.carbpol.2014.12.071

Jung, S., Myung, Y., Das, G. S., Bhatnagar, A., Park, J. W., Tripathi, K. M., & Kim, T. (2020). Carbon nano-onions from waste oil for application in energy storage devices. *New Journal of Chemistry*, *44*(18), 7369–7375. https://doi.org/10.1039/D0NJ00699H

Kale, R. D., Gorade, V. G., Madye, N., Chaudhary, B., Bangde, P. S., & Dandekar, P. P. (2018). Preparation and characterization of biocomposite packaging film from poly(lactic acid) and acylated microcrystalline cellulose using rice bran oil. *International Journal of Biological Macromolecules*, *118*, 1090–1102. https://doi.org/10.1016/J.IJBIOMAC.2018.06.076

Kambli, N. D., Mageshwaran, V., Patil, P. G., Saxena, S., & Deshmukh, R. R. (2017). Synthesis and characterization of microcrystalline cellulose powder from corn husk fibres using bio-chemical route. *Cellulose*, *24*(12), 5355–5369. https://doi.org/10.1007/s10570-017-1522-4

Kamenetsky, R., & Rabinowitch, H. D. (2017). Physiology of Domesticated Alliums: Onions, Garlic, Leek, and Minor Crops. In *Encyclopedia of Applied Plant Sciences* (Second Edi, Vol. 3, pp. 255–261). Elsevier. https://doi.org/10.1016/B978-0-12-394807-6.00064-2

Karaman, E., Yılmaz, E., & Tuncel, N. B. (2017). Physicochemical, microstructural and functional characterization of dietary fibers extracted from lemon, orange and grapefruit seeds press meals. *Bioactive Carbohydrates and Dietary Fibre*, *11*, 9–17. https://doi.org/10.1016/j.bcdf.2017.06.001

Karimi, R., Azizi, M. H., Xu, Q., Sahari, M. A., & Hamidi, Z. (2018). Enzymatic removal of starch and protein during the extraction of dietary fiber from barley bran. *Journal of*

Cereal Science, *83*, 259–265. https://doi.org/10.1016/j.jcs.2018.07.012

Kavaya, R. I., Omwamba, M. N., Chikamai, B. N., & Mahungu, S. M. (2019). Sensory Evaluation of Syneresis Reduced Jam and Marmalade Containing Gum Arabic from *Acacia senegal var. kerensis*; *Food and Nutrition Sciences*, *10*(11), 1334–1343. https://doi.org/10.4236/fns.2019.1011096

Khiari, Z., Makris, D. P., & Kefalas, P. (2009). An investigation on the recovery of antioxidant phenolics from onion solid wastes employing water/ethanol-based solvent systems. *Food and Bioprocess Technology*, *2*(4), 337–343. https://doi.org/10.1007/s11947-007-0044-8

Kia, L., Jawaid, M., Ariffin, H., & Alothman, O. Y. (2017). International Journal of Biological Macromolecules Isolation and characterization of microcrystalline cellulose from roselle fibers. *International Journal of Biological Macromolecules*, *103*, 931–940. https://doi.org/10.1016/j.ijbiomac.2017.05.135

Kieserling, K., Vu, T. M., Drusch, S., & Schalow, S. (2019). Impact of pectin-rich orange fibre on gel characteristics and sensory properties in lactic acid fermented yoghurt. *Food Hydrocolloids*, *94*(November 2018), 152–163. https://doi.org/10.1016/j.foodhyd.2019.02.051

Kim, S. Y., Hyeonbin, O., Lee, P., & Kim, Y. S. (2020). The quality characteristics, antioxidant activity, and sensory evaluation of reduced-fat yogurt and nonfat yogurt supplemented with basil seed gum as a fat substitute. *Journal of Dairy Science*, *103*(2), 1324–1336. https://doi.org/10.3168/jds.2019-17117

Kiros, E., Seifu, E., Bultosa, G., & Solomon, W. K. (2016). Effect of carrot juice and stabilizer on the physicochemical and microbiological properties of yoghurt. *LWT - Food Science and Technology*, *69*, 191–196. https://doi.org/10.1016/j.lwt.2016.01.026

Kiziltas, A., Gardner, D. J., Han, Y., & Yang, H. S. (2014). Mechanical Properties of

Microcrystalline Cellulose (MCC) Filled Engineering Thermoplastic Composites. *Journal of Polymers and the Environment 2014 22:3*, *22*(3), 365–372. https://doi.org/10.1007/S10924-014-0676-5

Koç, B., Sakin-YIlmazer, M., Kaymak-Ertekin, F., & BalkIr, P. (2014). Physical properties of yoghurt powder produced by spray drying. *Journal of Food Science and Technology*, *51*(7), 1377–1383. https://doi.org/10.1007/s13197-012-0653-8

Kolakovic, R., Peltonen, L., Laaksonen, T., Putkisto, K., Laukkanen, A., & Hirvonen, J. (2011). Spray-dried cellulose nanofibers as novel tablet excipient. *AAPS PharmSciTech*, *12*(4), 1366–1373. https://doi.org/10.1208/s12249-011-9705-z

Kothari, D., Lee, W.-D., & Kim, S.-K. (2020). Allium Flavonols: Health Benefits, Molecular Targets, and Bioavailability. *Antioxidants*, *9*(9), 888. https://doi.org/10.3390/antiox9090888

Krawczyk, G. R., Venables, A., & Tuason, D. (2009). Microcrystalline cellulose. In *Handbook of Hydrocolloids: Second Edition* (Issue Mcc). Woodhead Publishing Limited. https://doi.org/10.1533/9781845695873.740

Krontal, Y., Kamenetsky, R., & Rabinowitch, H. D. (2000). Flowering physiology and some vegetative traits of short-day shallot: A comparison with bulb onion. *The Journal of Horticultural Science and Biotechnology*, *75*(1), 35–41. https://doi.org/10.1080/14620316.2000.11511197

Kumar, M., Barbhai, M. D., Hasan, M., Punia, S., Dhumal, S., Radha, Rais, N., Chandran, D., Pandiselvam, R., Kothakota, A., Tomar, M., Satankar, V., Senapathy, M., Anitha, T., Dey, A., Sayed, A. A. S., Gadallah, F. M., Amarowicz, R., & Mekhemar, M. (2022). Onion (*Allium cepa L.*) peels: A review on bioactive compounds and biomedical activities. *Biomedicine and Pharmacotherapy*, *146*. https://doi.org/10.1016/J.BIOPHA.2021.112498

Kunusa, W. R., Isa, I., Laliyo, L. A. R., & Iyabu, H. (2018). FTIR, XRD and SEM Analysis of Microcrystalline Cellulose (MCC) Fibers from Corncorbs in Alkaline Treatment. *Journal of Physics: Conference Series*, *1028*(1). https://doi.org/10.1088/1742-6596/1028/1/012199

Kurek, M. A., Karp, S., Wyrwisz, J., & Niu, Y. (2018). Physicochemical properties of dietary fibers extracted from gluten-free sources: quinoa (*Chenopodium quinoa*), amaranth (*Amaranthus caudatus*) and millet (*Panicum miliaceum*). *Food Hydrocolloids*, *85*, 321–330. https://doi.org/10.1016/j.foodhyd.2018.07.021

Kusumattaqiin, F., & Chonkaew, W. (2015). Preparation and characterization of microcrystalline cellulose (MCC) by acid hydrolysis using microwave assisted method from cotton wool. *Macromolecular Symposia*, *354*(1), 35–41. https://doi.org/10.1002/masy.201400110

Kycia, K., Chlebowska-Śmigiel, A., Gniewosz, M., & Sokół, E. (2018). Effect of pullulan on the physicochemical properties of yoghurt. *International Journal of Dairy Technology*, *71*(1), 64–70. https://doi.org/10.1111/1471-0307.12401

Lafarga, T., Acién-Fernández, F. G., Castellari, M., Villaró, S., Bobo, G., & Aguiló-Aguayo, I. (2019). Effect of microalgae incorporation on the physicochemical, nutritional, and sensorial properties of an innovative broccoli soup. *LWT*, *111*, 167–174. https://doi.org/10.1016/j.lwt.2019.05.037

Law, M. F. L., & Deasy, P. B. (1998). Use of hydrophilic polymers with microcrystalline cellulose to improve extrusion–spheronization. *European Journal of Pharmaceutics and Biopharmaceutics*, *45*(1), 57–65. https://doi.org/10.1016/S0939-6411(97)00123-9

Lee, J. H., Park, H. Y., Won, J. I., Park, H. I., Choi, I. D., Lee, S. K., Park, J. Y., Joe, D. H., Jeon, Y. H., Oh, S. K., Han, S. I., & Choi, H. S. (2017). Quality characteristics of commercial liquid type yogurt in Korea. *Korean Journal of Food Preservation*, *24*(6),

865–870. https://doi.org/10.11002/kjfp.2017.24.6.865

Leelarungrayub, N., Rattanapanone, V., Chanarat, N., & Gebicki, J. M. (2006). Quantitative evaluation of the antioxidant properties of garlic and shallot preparations. *Nutrition*, *22*(3), 266–274. https://doi.org/10.1016/J.NUT.2005.05.010

Li, J. B., Dong, H. L., Xiu, H. J., Zhang, M. Y., Reddy, K. S., Zhang, X. F., & Ji, Y. (2016). Extraction, separation and refining of microcrystalline cellulose from wheat straw using various pretreatments. *International Journal of Agricultural and Biological Engineering*, *9*(2), 137–145. https://doi.org/10.3965/j.ijabe.20160902.1386

Lim, T. K. (2015). *Edible Medicinal and Non Medicinal Plants* (Vol. 9). https://doi.org/10.1007/978-94-017-9511-1

Limwong, V., Sutanthavidul, N., & Kulvanich, P. (2004). Spherical composite particles of rice starch and microcrystalline cellulose: A new coprocessed excipient for direct compression. *AAPS PharmSciTech 2004 5:2*, *5*(2), 40–49. https://doi.org/10.1208/PT050230

Ling, Z., Tang, W., Su, Y., Shao, L., Wang, P., Ren, Y., Huang, C., Lai, C., & Yong, Q. (2021). Promoting enzymatic hydrolysis of aggregated bamboo crystalline cellulose by fast microwave-assisted dicarboxylic acid deep eutectic solvents pretreatments. *Bioresource Technology*, *333*(April), 125122. https://doi.org/10.1016/j.biortech.2021.125122

Liu, K., Du, H., Zheng, T., Liu, H., Zhang, M., Zhang, R., Li, H., Xie, H., Zhang, X., Ma, M., & Si, C. (2021). Recent advances in cellulose and its derivatives for oilfield applications. *Carbohydrate Polymers*, *259*(January), 117740. https://doi.org/10.1016/j.carbpol.2021.117740

Liu, Y., Liu, A., Ibrahim, S. A., Yang, H., & Huang, W. (2018). Isolation and characterization of microcrystalline cellulose from pomelo peel. *International Journal*

of Biological Macromolecules, *111*, 717–721. https://doi.org/10.1016/j.ijbiomac.2018.01.098

Lu, H., Gui, Y., Zheng, L., & Liu, X. (2013). Morphological, crystalline, thermal and physicochemical properties of cellulose nanocrystals obtained from sweet potato residue. *Food Research International*, *50*(1), 121–128. https://doi.org/10.1016/j.foodres.2012.10.013

Lubbers, S., Decourcelle, N., Vallet, N., & Guichard, E. (2004). Flavor Release and Rheology Behavior of Strawberry Fat free Stirred Yogurt during Storage. *Journal of Agricultural and Food Chemistry*, *52*(10), 3077–3082. https://doi.org/10.1021/jf0352374

Lucas, B. F., Morais, M. G. de, Santos, T. D., & Costa, J. A. V. (2018a). Spirulina for snack enrichment: Nutritional, physical and sensory evaluations. *LWT - Food Science and Technology*, *90*, 270–276. https://doi.org/10.1016/j.lwt.2017.12.032

Lucas, B. F., Morais, M. G. de, Santos, T. D., & Costa, J. A. V. (2018b). Spirulina for snack enrichment: Nutritional, physical and sensory evaluations. *LWT - Food Science and Technology*, *90*, 270–276. https://doi.org/10.1016/J.LWT.2017.12.032

Major, N., Goreta Ban, S., Urlić, B., Ban, D., Dumičić, G., & Perković, J. (2018). Morphological and Biochemical Diversity of Shallot Landraces Preserved Along the Croatian Coast. *Frontiers in Plant Science*, *9*. https://doi.org/10.3389/fpls.2018.01749

Mandal, A., & Chakrabarty, D. (2011). Isolation of nanocellulose from waste sugarcane bagasse (SCB) and its characterization. *Carbohydrate Polymers*, *86*(3), 1291–1299. https://doi.org/10.1016/j.carbpol.2011.06.030

Mansour, M. P., Cornell, H. J., Holt, L. A., & Rivett, D. E. (1989). The effect of solvent extraction on the hydrothermal and alkaline stability of wool. *Journal of the Textile Institute*, *80*(2), 261–267. https://doi.org/10.1080/00405008908659205

Marlin, M., Hartal, H., Romeida, A., Herawati, R., & Simarmata, M. (2021). Morphological and Flowering Characteristics of Shallot (Allium cepa Var. Aggregatum) in Response to Gibberellic Acid and Vernalization. *Emirates Journal of Food and Agriculture*, *33*(5), 388. https://doi.org/10.9755/ejfa.2021.v33.i5.2697

Mehare, M. D., Deshmukh, A. D., & Dhoble, S. J. (2019). Preparation of porous agro-waste-derived carbon from onion peel for supercapacitor application. *Journal of Materials Science 2019 55:10*, *55*(10), 4213–4224. https://doi.org/10.1007/S10853-019-04236-7

Merci, A., Urbano, A., Victória, M., Grossmann, E., & Tischer, C. A. (2015). Properties of microcrystalline cellulose extracted from soybean hulls by reactive extrusion. *FRIN*, *73*, 38–43. https://doi.org/10.1016/j.foodres.2015.03.020

Michalak-Majewska, M., Teterycz, D., Muszyński, S., Radzki, W., & Sykut-Domańska, E. (2020). Influence of onion skin powder on nutritional and quality attributes of wheat pasta. *PLoS ONE*, *15*(1), e0227942. https://doi.org/10.1371/journal.pone.0227942

Microcrystalline Cellulose Market | 2022 - 27 | Industry Share, Size, Report. (n.d.). Retrieved April 18, 2022, from https://www.mordorintelligence.com/industry-reports/microcrystalline-cellulose-market

Milea, Ștefania A., Aprodu, I., Mihalcea, L., Enachi, E., Bolea, C. A., Râpeanu, G., Bahrim, G. E., & Stănciuc, N. (2020). Bovine β-lactoglobulin peptides as novel carriers for flavonoids extracted with supercritical fluids from yellow onion skins. *Journal of Food Science*, *85*(12), 4290–4299. https://doi.org/10.1111/1750-3841.15513

Mokhtar, S. M., Swailam, H. M., & Embaby, H. E. S. (2018). Physicochemical properties, nutritional value and techno-functional properties of goldenberry (Physalis peruviana) waste powder concise title: Composition of goldenberry juice waste. *Food Chemistry*, *248*, 1–7. https://doi.org/10.1016/j.foodchem.2017.11.117

Mourtzinos, I., Prodromidis, P., Grigorakis, S., Makris, D. P., Biliaderis, C. G., &

Moschakis, T. (2018a). Natural food colorants derived from onion wastes: Application in a yoghurt product. *Electrophoresis*, *39*(15), 1975–1983. https://doi.org/10.1002/elps.201800073

Mousavi, M., Heshmati, A., Daraei Garmakhany, A., Vahidinia, A., & Taheri, M. (2019). Texture and sensory characterization of functional yogurt supplemented with flaxseed during cold storage. *Food Science and Nutrition*, *7*(3), 907–917. https://doi.org/10.1002/fsn3.805

Mousavi, M., Heshmati, A., Garmakhany, A. D., Vahidinia, A., & Taheri, M. (2019). Optimization of the viability of Lactobacillus acidophilus and physico-chemical, textural and sensorial characteristics of flaxseed-enriched stirred probiotic yogurt by using response surface methodology. *LWT*, *102*(December 2018), 80–88. https://doi.org/10.1016/j.lwt.2018.12.023

Naduparambath, S., & Purushothaman, E. (2016). Sago seed shell: determination of the composition and isolation of microcrystalline cellulose (MCC). *Cellulose*, *23*(3), 1803–1812. https://doi.org/10.1007/s10570-016-0904-3

Nair, A., Varghese, S. M., Nair K, A. U., & R, S. V. (2021). *Effect of incorporation of microcrystalline cellulose extracted from shallot bio-waste into low-fat stirred yogurt and its characterization*. *6*(5), 577–585. www.botanyjournals.com

Nakamori, T., Miyagishim, A., Nozawa, Y., Sadzuka, Y., & Sonobe, T. (2008). Influence of load on particle size distribution of lactose-crystalline cellulose mixed powder. *International Journal of Pharmaceutics*, *354*(1–2), 255–259. https://doi.org/10.1016/j.ijpharm.2007.11.043

Nihayah, B., Maftukhah, R., Nugroho, B. D. A., & Prasetyatama, Y. D. (2020). The response of growth rate in different husk biochar application of *Allium cepa L* in sandy soil. *IOP Conference Series: Earth and Environmental Science*, *542*(1), 012052.

https://doi.org/10.1088/1755-1315/542/1/012052

Nile, A., Gansukh, E., Park, G. S., Kim, D. H., & Hariram Nile, S. (2021). Novel insights on the multi-functional properties of flavonol glucosides from red onion (*Allium cepa L*) solid waste – In vitro and in silico approach. *Food Chemistry*, *335*(July 2020), 127650. https://doi.org/10.1016/j.foodchem.2020.127650

Nile, A., Nile, S. H., Kim, D. H., Keum, Y. S., Seok, P. G., & Sharma, K. (2018). Valorization of onion solid waste and their flavonols for assessment of cytotoxicity, enzyme inhibitory and antioxidant activities. *Food and Chemical Toxicology*, *119*(January), 281–289. https://doi.org/10.1016/j.fct.2018.02.056

Nile, S. H., Nile, A. S., Keum, Y. S., & Sharma, K. (2017). Utilization of quercetin and quercetin glycosides from onion (*Allium cepa L.*) solid waste as an antioxidant, urease and xanthine oxidase inhibitors. *Food Chemistry*, *235*, 119–126. https://doi.org/10.1016/j.foodchem.2017.05.043

Noishiki, Y., Nishiyama, Y., Wada, M., Kuga, S., & Magoshi, J. (2002). Mechanical properties of silk fibroin–microcrystalline cellulose composite films. *Journal of Applied Polymer Science*, *86*(13), 3425–3429. https://doi.org/10.1002/APP.11370

Nsor-Atindana, J., Chen, M., Goff, H. D., Zhong, F., Sharif, H. R., & Li, Y. (2017). Functionality and nutritional aspects of microcrystalline cellulose in food. In *Carbohydrate Polymers* (Vol. 172, pp. 159–174). Elsevier Ltd. https://doi.org/10.1016/j.carbpol.2017.04.021

Nsor-Atindana, J., Zhou, Y. X., Saqib, M. N., Chen, M., Douglas Goff, H., Ma, J., & Zhong, F. (2020). Enhancing the prebiotic effect of cellulose biopolymer in the gut by physical structuring via particle size manipulation. *Food Research International*, *131*(February 2019), 108935. https://doi.org/10.1016/j.foodres.2019.108935

Ohwoavworhua, F., Adelakun, T., & Okhamafe, A. (2009). Processing pharmaceutical

grade microcrystalline cellulose from groundnut husk: Extraction methods and characterization. *International Journal of Green Pharmacy*, *3*(2), 97. https://doi.org/10.4103/0973-8258.54895

Ohwoavworhua, F. O., & Adelakun, T. A. (2010). Non-wood fibre production of microcrystalline cellulose from Sorghum caudatum: Characterisation and tableting properties. *Indian Journal of Pharmaceutical Sciences*, *72*(3), 295–301. https://doi.org/10.4103/0250-474X.70473

Oliveira, A. A. A., Andrade, A. C., Bastos, S. C., Condino, J. P. F., Curzi Júnior, A., & Pinheiro, A. C. M. (2021). Use of strawberry and vanilla natural flavors for sugar reduction: A dynamic sensory study with yogurt. *Food Research International*, *139*, 109972. https://doi.org/10.1016/j.foodres.2020.109972

Oluseun Adejumo, I., & Adebukola Adebiyi, O. (2021). Agricultural Solid Wastes: Causes, Effects, and Effective Management. In *Strategies of Sustainable Solid Waste Management*. IntechOpen. https://doi.org/10.5772/intechopen.93601

Orasugh, J. T., Saha, N. R., Sarkar, G., Rana, D., Mondal, D., Ghosh, S. K., & Chattopadhyay, D. (2018). A facile comparative approach towards utilization of waste cotton lint for the synthesis of nano-crystalline cellulose crystals along with acid recovery. *International Journal of Biological Macromolecules*, *109*, 1246–1252. https://doi.org/10.1016/j.ijbiomac.2017.11.123

Osorio-Arias, J., Pérez-Martínez, A., Vega-Castro, O., & Martínez-Monteagudo, S. I. (2020). Rheological, texture, structural, and functional properties of Greek-style yogurt fortified with cheese whey-spent coffee ground powder. *Lwt*, *129*(May), 109523. https://doi.org/10.1016/j.lwt.2020.109523

Otles, S., Despoudi, S., Bucatariu, C., & Kartal, C. (2015). Food waste management, valorization, and sustainability in the food industry. In *Food Waste Recovery:*

Processing Technologies and Industrial Techniques (pp. 3–23). Elsevier. https://doi.org/10.1016/B978-0-12-800351-0.00001-8

Pan, L., Li, P., & Tao, Y. (2020). Preparation and Properties of Microcrystalline Cellulose/Fish Gelatin Composite Film. *Materials 2020, Vol. 13, Page 4370*, *13*(19), 4370. https://doi.org/10.3390/MA13194370

Panesar, P. S., & Bali, V. (2015). Prebiotics. *Encyclopedia of Food and Health*, 464–471. https://doi.org/10.1016/B978-0-12-384947-2.00560-2

Pareek, S., Sagar, N. A., Sharma, S., & Kumar, V. (2017). Onion (Allium cepa L.). In *Fruit and Vegetable Phytochemicals* (Vol. 2, Issue October, pp. 1145–1162). John Wiley & Sons, Ltd. https://doi.org/10.1002/9781119158042.ch58

Pastell, H., Putkonen, T., & Rita, H. (2019). Dietary fibre in legumes, seeds, vegetables, fruits and mushrooms: Comparing traditional and semi-automated filtration techniques. *Journal of Food Composition and Analysis*, *75*(September 2018), 1–7. https://doi.org/10.1016/j.jfca.2018.09.011

Pérez, J., Arteaga, M., Andrade, R., Durango, A., & Salcedo, J. (2021). Effect of yam (Dioscorea spp.) starch on the physicochemical, rheological, and sensory properties of yogurt. *Heliyon*, *7*(1), e05987. https://doi.org/10.1016/j.heliyon.2021.e05987

Peterson, J. (2000). The Allium Species (Onions, Garlic, Leeks, Chives, and Shallots). In *The Cambridge World History of Food* (pp. 249–271). Cambridge University Press. https://doi.org/10.1017/CHOL9780521402149.028

Pezeshk, S., Rezaei, M., & Hosseini, H. (2011). Effects of Turmeric, Shallot Extracts, and Their Combination on Quality Characteristics of Vacuum-Packaged Rainbow Trout Stored at 4 ± 1 °C. *Journal of Food Science*, *76*(6), M387–M391. https://doi.org/10.1111/J.1750-3841.2011.02242.X

Phaiphan, A., Panichakool, P., Jinawan, S., & Penjumras, P. (2019). Effects of heat and

shallot (Allium ascalonicum L.) supplementation on nutritional quality and enzymatic browning of apple juice. *Journal of Food Science and Technology 2019 56:9*, *56*(9), 4121–4128. https://doi.org/10.1007/S13197-019-03882-8

Piechowiak, T., & Balawejder, M. (2019). Onion skin extract as a protective agent against oxidative stress in Saccharomyces cerevisiae induced by cadmium. *Journal of Food Biochemistry*, *43*(7), 1–9. https://doi.org/10.1111/jfbc.12872

Piechowiak, T., Grzelak-Błaszczyk, K., Bonikowski, R., & Balawejder, M. (2020). Optimization of extraction process of antioxidant compounds from yellow onion skin and their use in functional bread production. *Lwt*, *117*(September 2019). https://doi.org/10.1016/j.lwt.2019.108614

Pontonio, E., Raho, S., Dingeo, C., Centrone, D., Carofiglio, V. E., & Rizzello, C. G. (2020). Nutritional, Functional, and Technological Characterization of a Novel Gluten- and Lactose-Free Yogurt-Style Snack Produced With Selected Lactic Acid Bacteria and Leguminosae Flours. *Frontiers in Microbiology*, *11*(July), 1–12. https://doi.org/10.3389/fmicb.2020.01664

Puri, S., Sharma, S., Kumari, A., Sharma, M., Sharma, U., & Kumar, S. (2020). Extraction of lignocellulosic constituents from cow dung: preparation and characterisation of nanocellulose. *Biomass Conversion and Biorefinery*. https://doi.org/10.1007/s13399-020-01119-9

Quilliam, R. S., Nuku-Adeku, C., Maquart, P., Little, D., Newton, R., & Murray, F. (2020). Integrating insect frass biofertilisers into sustainable peri-urban agro-food systems. *Https://Doi.Org/10.3920/JIFF2019.0049*, *6*(3), 315–322. https://doi.org/10.3920/JIFF2019.0049

Rasheed, M., Jawaid, M., Karim, Z., & Abdullah, L. C. (2020). Morphological, Physiochemical and Thermal Properties of Microcrystalline Cellulose (MCC)

Extracted from Bamboo Fiber. *Molecules*, *25*(12). https://doi.org/10.3390/molecules25122824

Reddy, J. P., & Rhim, J. W. (2018). Extraction and Characterization of Cellulose Microfibers from Agricultural Wastes of Onion and Garlic. *Journal of Natural Fibers*, *15*(4), 465–473. https://doi.org/10.1080/15440478.2014.945227

Ren, H., Shen, J., Pei, J., Wang, Z., Peng, Z., Fu, S., & Zheng, Y. (2019). Characteristic microcrystalline cellulose extracted by combined acid and enzyme hydrolysis of sweet sorghum. *Cellulose*, *26*(15), 8367–8381. https://doi.org/10.1007/s10570-019-02712-6

Rodríguez, R., Jiménez, A., Fernández-Bolaños, J., Guillén, R., & Heredia, A. (2006). Dietary fibre from vegetable products as source of functional ingredients. *Trends in Food Science and Technology*, *17*(1), 3–15. https://doi.org/10.1016/j.tifs.2005.10.002

Sadh, P. K., Duhan, S., & Duhan, J. S. (2018). Agro-industrial wastes and their utilization using solid state fermentation: a review. *Bioresources and Bioprocessing*, *5*(1), 1–15. https://doi.org/10.1186/s40643-017-0187-z

Sah, B. N. P., Vasiljevic, T., McKechnie, S., & Donkor, O. N. (2016). Physicochemical, textural and rheological properties of probiotic yogurt fortified with fibre-rich pineapple peel powder during refrigerated storage. *LWT - Food Science and Technology*, *65*, 978–986. https://doi.org/10.1016/j.lwt.2015.09.027

Sainorudin, M. H., Mohammad, M., Huda, N., Kadir, A., Abdullah, N. A., & Yaakob, Z. (2018). *Characterization of Several Microcrystalline Cellulose (MCC) -Based Agricultural Wastes via X-Ray Diffraction Method*. *280*, 340–345. https://doi.org/10.4028/www.scientific.net/SSP.280.340

Saleh, A., Mohamed, A. A., Alamri, M. S., Hussain, S., Qasem, A. A., & Ibraheem, M. A. (2020). Effect of Different Starches on the Rheological, Sensory and Storage Attributes of Non-fat Set Yogurt. *Foods*, *9*(1), 61. https://doi.org/10.3390/foods9010061

Santiago, B., Arias Calvo, A., Gullón, B., Feijoo, G., Moreira, M. T., & González-García, S. (2020). Production of flavonol quercetin and fructooligosaccharides from onion (*Allium cepa L.*) waste: An environmental life cycle approach. *Chemical Engineering Journal*, *392*, 123772. https://doi.org/10.1016/j.cej.2019.123772

Saraswathi, T., Sapthiyamurthy, V. A., Tamilselvi, N. A., & Harsh, S. (2017). Review on aggregatum Onion (*Allium cepa L. var. aggregatum* Don.). *International Journal of Current Microbiology and Applied Sciences*, *6*(4), 1649–1667.

Sarwono, Santosa, S., & Prameswari, N. S. (2019). Short Communication: The color fastness of cotton cloth dyed with dye extracted from skin of shallot (*Allium ascalonicum*). *Biodiversitas Journal of Biological Diversity*, *20*(9), 2475–2479. https://doi.org/10.13057/BIODIV/D200907

Satyamurthy, P., Jain, P., Balasubramanya, R. H., & Vigneshwaran, N. (2011). Preparation and characterization of cellulose nanowhiskers from cotton fibres by controlled microbial hydrolysis. *Carbohydrate Polymers*, *83*(1), 122–129. https://doi.org/10.1016/j.carbpol.2010.07.029

Schuh, V., Allard, K., Herrmann, K., Gibis, M., Kohlus, R., & Weiss, J. (2013). Impact of carboxymethyl cellulose (CMC) and microcrystalline cellulose (MCC) on functional characteristics of emulsified sausages. *MESC*, *93*(2), 240–247. https://doi.org/10.1016/j.meatsci.2012.08.025

Ścibisz, I., Ziarno, M., & Mitek, M. (2019). Color stability of fruit yogurt during storage. *Journal of Food Science and Technology*, *56*(4), 1997–2009. https://doi.org/10.1007/s13197-019-03668-y

Seddiqi, H., Oliaei, E., Honarkar, H., Jin, J., Geonzon, L. C., Bacabac, R. G., & Klein-Nulend, J. (2021). Cellulose and its derivatives: towards biomedical applications. In *Cellulose* (Vol. 28, Issue 4). Springer Netherlands. https://doi.org/10.1007/s10570-

020-03674-w

Seehra, M. S., Popp, B. V., Goulay, F., Pyapalli, S. K., Gullion, T., & Poston, J. (2014). Hydrothermal treatment of microcrystalline cellulose under mild conditions: characterization of solid and liquid-phase products. *Cellulose*, *21*(6), 4483–4495. https://doi.org/10.1007/s10570-014-0424-y

Seera, S. D. K., Kundu, D., & Banerjee, T. (2020). Physical and chemical crosslinked microcrystalline cellulose-polyvinyl alcohol hydrogel: freeze–thaw mediated synthesis, characterization and in vitro delivery of 5-fluorouracil. *Cellulose 2020 27:11*, *27*(11), 6521–6535. https://doi.org/10.1007/S10570-020-03249-9

Senaka Ranadheera, C., Evans, C. A., Adams, M. C., & Baines, S. K. (2012). Probiotic viability and physico-chemical and sensory properties of plain and stirred fruit yogurts made from goat's milk. *Food Chemistry*, *135*(3), 1411–1418. https://doi.org/10.1016/j.foodchem.2012.06.025

Setyadjit, & Sukasih, E. (2015). Effect of Addition of Filler on the Production of Shallot (*Allium Cepa Var. Ascalonicum L.*) Powder with Drum Dryer. *Procedia Food Science*, *3*, 396–408. https://doi.org/10.1016/J.PROFOO.2015.01.044

Seyfi, P., Mostafaie, A., Mansouri, K., Arshadi, D., Mohammadi-Motlagh, H. R., & Kiani, A. (2010). In vitro and in vivo anti-angiogenesis effect of shallot (*Allium ascalonicum*): A heat-stable and flavonoid-rich fraction of shallot extract potently inhibits angiogenesis. *Toxicology in Vitro*, *24*(6), 1655–1661. https://doi.org/10.1016/J.TIV.2010.05.022

Shahbazi, M., Jäger, H., & Ettelaie, R. (2022). A Promising Therapeutic Soy-Based Pickering Emulsion Gel Stabilized by a Multifunctional Microcrystalline Cellulose: Application in 3D Food Printing. *Journal of Agricultural and Food Chemistry*, *70*(7), 2374–2388. https://doi.org/10.1021/ACS.JAFC.1C05644

Shao, X., Wang, J., Liu, Z., Hu, N., Liu, M., & Xu, Y. (2020). Preparation and Characterization of Porous Microcrystalline Cellulose from Corncob. *Industrial Crops and Products*, *151*(April), 1–6. https://doi.org/10.1016/j.indcrop.2020.112457

Sharma, K., Mahato, N., Nile, S. H., Lee, E. T., & Lee, Y. R. (2016a). Economical and environmentally-friendly approaches for usage of onion (*Allium cepa L.*) waste. *Food and Function*, *7*(8), 3354–3369. https://doi.org/10.1039/c6fo00251j

Shea, N. O., Arendt, E. K., & Gallagher, E. (2012). Dietary fi bre and phytochemical characteristics of fruit and vegetable by-products and their recent applications as novel ingredients in food products. *Innovative Food Science and Emerging Technologies*, *16*, 1–10. https://doi.org/10.1016/j.ifset.2012.06.002

Sheng, S., Meiling, Z., Chen, L., Wensheng, H., & Zhifeng, Y. (2018). Extraction and characterization of microcrystalline cellulose from waste cotton fabrics via hydrothermal method. *Waste Management*, *82*, 139–146. https://doi.org/10.1016/j.wasman.2018.10.023

Shery M Varghese, Aruna Nair U K, S. V. R. (2021). Effect of incorporation of microcrystalline cellulose extracted from shallot bio-waste into low-fat stirred yogurt and its characterization. *International Journal of Botany Studies*, *6*(5), 577–584.

Sholichah, E., Nugroho, P., & Purwono, B. (2018). Preparation and characterization of active film made from arrowroot starch/PVA film and isolated quercetin from shallot (Allium cepa L. var, aggregatum). *AIP Conference Proceedings*, *2024*(1), 020013. https://doi.org/10.1063/1.5064299

Silva, A. N. da, Silva, R. de C. dos S. N. da, Ferreira, M. A. M., Minim, V. P. R., Costa, T. de M. T. da, & Perez, R. (2013). Performance of hedonic scales in sensory acceptability of strawberry yogurt. *Food Quality and Preference*, *30*(1), 9–21. https://doi.org/10.1016/j.foodqual.2013.04.001

Simin, N., Orcic, D., Cetojevic-Simin, D., Mimica-Dukic, N., Anackov, G., Beara, I., Mitic-Culafic, D., & Bozin, B. (2013). Phenolic profile, antioxidant, anti-inflammatory and cytotoxic activities of small yellow onion (*Allium flavum L. subsp. flavum*, Alliaceae). *LWT - Food Science and Technology*, *54*(1), 139–146. https://doi.org/10.1016/j.lwt.2013.05.023

Slavin, J. (2013). *Fiber and Prebiotics: Mechanisms and Health Benefits*. 1417–1435. https://doi.org/10.3390/nu5041417

Small onion cultivation will be profitable, TNAU _ Business Standard News. (n.d.).

Sobhay, A., A., A., Hassan, Z., & El-Batawy, O. (2019). Properties of Drinking Yoghurt Using Different Types of Stabilizers. *Arab Universities Journal of Agricultural Sciences*, *27*(1), 431–440. https://doi.org/10.21608/ajs.2019.43582

Srimali, S. V. H., Weerasingha, V. V. ., & Nayananjalie, W. A. . (2019). Development of drinking yogurt using citrus fiber as a stabilizer. *Annals. Food Science and Technology*, *20*(3), 420–426. www.afst.valahia.ro

Strydom, S. J., Otto, D. P., Liebenberg, W., Lvov, Y. M., & De Villiers, M. M. (2011). Preparation and characterization of directly compactible layer-by-layer nanocoated cellulose. *International Journal of Pharmaceutics*, *404*(1–2), 57–65. https://doi.org/10.1016/j.ijpharm.2010.10.056

Sukaimi, J., Wan Abdullah, W. N. H., Supian, M. A. F., Jamari, S. S., Mohd Azman, N. A., Mohamad, S., & Ali, M. F. (2019). Properties of microcrystalline cellulose prepared from oil palm empty fruit bunch at different sulfuric acid concentration and hydrolysis temperature. *IOP Conference Series: Materials Science and Engineering*, *702*(1). https://doi.org/10.1088/1757-899X/702/1/012035

Sukasih, E., Setyadjit, & Musadad, D. (2018). Physico-chemical characteristics of shallot New-Superior Varieties (NSV) from Indonesia. *IOP Conference Series: Earth and*

Environmental Science, *102*(1), 012037. https://doi.org/10.1088/1755-1315/102/1/012037

Sumonsiri, N., Kundacha, N., & Pom-Iam, N. (2018). Effect of microcrystalline cellulose on physical characteristics and sensory acceptance of chocolate flavored milk. *Current Research in Nutrition and Food Science*, *6*(3), 781–788. https://doi.org/10.12944/CRNFSJ.6.3.20

Sun-Waterhouse, D., Smith, B. G., O'Connor, C. J., & Melton, L. D. (2008). Effect of raw and cooked onion dietary fibre on the antioxidant activity of ascorbic acid and quercetin. *Food Chemistry*, *111*(3), 580–585. https://doi.org/10.1016/j.foodchem.2008.04.023

Sundarraj, A. A., & Ranganathan, T. V. (2018). Extraction and Functional Properties of Cellulose From Jackfruit (*Artocarpus Integer)* Waste. *International Journal of Pharmaceutical Sciences and Research 4309 IJPSR*, *9*(10), 4309–4317. https://doi.org/10.13040/IJPSR.0975-8232.9(10).4309-17

Swamy, K. R. M., & Veere Gowda, R. (2006). Leek and shallot. In *Handbook of Herbs and Spices* (Vol. 3, pp. 365–389). Elsevier. https://doi.org/10.1533/9781845691717.3.365

Tabor, G. (2018). Development of seed propagated shallot (*Allium cepa L var. aggregatum*) varieties in Ethiopia. *Scientia Horticulturae*, *240*, 89–93. https://doi.org/10.1016/j.scienta.2018.05.046

Tan Sian Hui Abdullah, H. S., Aqlili Riana Mohd Asseri, S. N., Khursyiah Wan Mohamad, W. N., Kan, S. Y., Azmi, A. A., Yong Julius, F. S., & Chia, P. W. (2021). Green synthesis, characterization and applications of silver nanoparticle mediated by the aqueous extract of red onion peel. *Environmental Pollution*, *271*, 116295. https://doi.org/10.1016/J.ENVPOL.2020.116295

Tarchoun, A. F., Trache, D., Klapötke, T. M., Derradji, M., & Bessa, W. (2019). Ecofriendly

isolation and characterization of microcrystalline cellulose from giant reed using various acidic media. *Cellulose 2019 26:13*, *26*(13), 7635–7651. https://doi.org/10.1007/S10570-019-02672-X

Teles, C. D., & Flôres, S. H. (2007). The influence of additives on the rheological and sensory properties of nonfat yogurt. *International Journal of Dairy Technology*, *60*(4), 270–276. https://doi.org/10.1111/j.1471-0307.2007.00354.x

Thivya, P., Bhosale, Y. K., Anandakumar, S., Hema, V., & Sinija, V. R. (2021a). Exploring the Effective Utilization of Shallot Stalk Waste and Tamarind Seed for Packaging Film Preparation. *Waste and Biomass Valorization 2021 12:10*, *12*(10), 5779–5794. https://doi.org/10.1007/S12649-021-01402-4

Thivya, P., Bhosale, Y. K., Anandakumar, S., Hema, V., & Sinija, V. R. (2021b). Development of active packaging film from sodium alginate/carboxymethyl cellulose containing shallot waste extracts for anti-browning of fresh-cut produce. *International Journal of Biological Macromolecules*, *188*, 790–799. https://doi.org/10.1016/J.IJBIOMAC.2021.08.039

Thomsen, M. H., Thygesen, A., & Thomsen, A. B. (2008). Hydrothermal treatment of wheat straw at pilot plant scale using a three-step reactor system aiming at high hemicellulose recovery, high cellulose digestibility and low lignin hydrolysis. *Bioresource Technology*, *99*(10), 4221–4228. https://doi.org/10.1016/j.biortech.2007.08.054

Tian, S., Zhao, R., & Chen, Z. (2018). Review of the pretreatment and bioconversion of lignocellulosic biomass from wheat straw materials. *Renewable and Sustainable Energy Reviews*, *91*(March), 483–489. https://doi.org/10.1016/j.rser.2018.03.113

Torrico, D. D., Tam, J., Fuentes, S., Gonzalez Viejo, C., & Dunshea, F. R. (2020). Consumer rejection threshold, acceptability rates, physicochemical properties, and shelf-life of strawberry-flavored yogurts with reductions of sugar. *Journal of the Science of Food*

and Agriculture, *100*(7), 3024–3035. https://doi.org/10.1002/jsfa.10333

Trache, D., Khimeche, K., Donnot, A., & Benelmir, R. (2013). FTIR spectroscopy and X-ray powder diffraction characterization of microcrystalline cellulose obtained from alfa fibers. *MATEC Web of Conferences*, *3*, 01023. https://doi.org/10.1051/matecconf/20130301023

Tran, B. G., & Nguyen, N. H. (2020). Chemical composition and antioxidant, anti-inflammatory, and anticancer effects of ethanol extract of black shallot (*Allium ascalonicum*). *Pharmacophore*, *11*(3), 30–37.

Tril, U., Fernández-López, J., Álvarez, J. Á. P., & Viuda-Martos, M. (2014). Chemical, physicochemical, technological, antibacterial and antioxidant properties of rich-fibre powder extract obtained from tamarind (*Tamarindus indica L.*). *Industrial Crops and Products*, *55*, 155–162. https://doi.org/10.1016/j.indcrop.2014.01.047

Trilokesh, C., & Uppuluri, K. B. (2019). Isolation and characterization of cellulose nanocrystals from jackfruit peel. *Scientific Reports*, *9*(1), 1–8. https://doi.org/10.1038/s41598-019-53412-x

Tripathi, N., Hills, C. D., Singh, R. S., & Atkinson, C. J. (2019). Biomass waste utilisation in low-carbon products: harnessing a major potential resource. *Npj Climate and Atmospheric Science*, *2*(1). https://doi.org/10.1038/s41612-019-0093-5

Turgut, T., & Cakmakci, S. (2018). Probiotic Strawberry Yogurts: Microbiological, Chemical and Sensory Properties. *Probiotics and Antimicrobial Proteins*, *10*(1), 64–70. https://doi.org/10.1007/s12602-017-9278-6

Twarogowska, A., Van Poucke, C., & Van Droogenbroeck, B. (2020). Upcycling of Belgian endive (*Cichorium intybus var. foliosum*) by-products. Chemical composition and functional properties of dietary fibre root powders. *Food Chemistry*, *332*, 127444. https://doi.org/10.1016/j.foodchem.2020.127444

Uesu, N. Y., Go, E. A., Adelina, A., & Hechenleitner, W. (2000). *Microcrystalline cellulose from soybean husk : effects of solvent treatments on its properties as acetylsalicylic acid carrier*. *206*, 85–96.

Vanegas-Azuero, A. M., & Gutiérrez, L. F. (2018). Physicochemical and sensory properties of yogurts containing sacha inchi (*Plukenetia volubilis L.*) seeds and β-glucans from Ganoderma lucidum. *Journal of Dairy Science*, *101*(2), 1020–1033. https://doi.org/10.3168/jds.2017-13235

Varghese, S. M., Bhosale, Y. K., Aruna Nair, U. K., Hema, V., & Sinija, V. R. (2022). Valorisation and Characterization of *Allium cepa var. aggregatum* Biowastes for the Production of Microcrystalline Cellulose. *Waste and Biomass Valorization*, *13*(4), 1931–1944. https://doi.org/10.1007/s12649-021-01625-5

Vénica, C. I., Spotti, M. J., Pavón, Y. L., Molli, J. S., & Perotti, M. C. (2020). Influence of carrot fibre powder addition on rheological, microstructure and sensory characteristics of stirred-type yogurt. *International Journal of Food Science and Technology*, *55*(5), 1916–1923. https://doi.org/10.1111/ijfs.14415

Ventura-Cruz, S., & Tecante, A. (2021). Nanocellulose and microcrystalline cellulose from agricultural waste: Review on isolation and application as reinforcement in polymeric matrices. *Food Hydrocolloids*, *118*(March), 106771. https://doi.org/10.1016/j.foodhyd.2021.106771

Wang, K., Li, M., Wang, Y., Liu, Z., & Ni, Y. (2021). Effects of extraction methods on the structural characteristics and functional properties of dietary fiber extracted from kiwifruit (*Actinidia deliciosa*). *Food Hydrocolloids*, *110*(17). https://doi.org/10.1016/j.foodhyd.2020.106162

Wang, M.-R., Hamborg, Z., Slimestad, R., Elameen, A., Blystad, D.-R., Haugslien, S., Skjeseth, G., & Wang, Q.-C. (2021). Assessments of rooting, vegetative growth, bulb

production, genetic integrity and biochemical compounds in cryopreserved plants of shallot. *Plant Cell, Tissue and Organ Culture (PCTOC)*, *144*(1), 123–131. https://doi.org/10.1007/s11240-020-01820-7

Wang, X., Kristo, E., & LaPointe, G. (2019). The effect of apple pomace on the texture, rheology and microstructure of set type yogurt. *Food Hydrocolloids*, *91*(January), 83–91. https://doi.org/10.1016/j.foodhyd.2019.01.004

Wang, X., Kristo, E., & LaPointe, G. (2020). Adding apple pomace as a functional ingredient in stirred-type yogurt and yogurt drinks. *Food Hydrocolloids*, *100*, 105453. https://doi.org/10.1016/j.foodhyd.2019.105453

Wang, Y. X., Li, L. Y., Zhang, T., Wang, J. Q., Huang, X. J., Hu, J. L., Yin, J. Y., & Nie, S. P. (2021). Fractionation, physicochemical and structural characterization of polysaccharides from barley water-soluble fiber. *Food Hydrocolloids*, *113*(December 2020), 106539. https://doi.org/10.1016/j.foodhyd.2020.106539

Wei, X., Huang, T., Nie, J., Yang, J. hui, Qi, X. dong, Zhou, Z. wan, & Wang, Y. (2018). Bio-inspired functionalization of microcrystalline cellulose aerogel with high adsorption performance toward dyes. *Carbohydrate Polymers*, *198*, 546–555. https://doi.org/10.1016/J.CARBPOL.2018.06.112

Wenli, S., Mohamad, H. S., & Qi, C. (2019). The insight and survey on medicinal properties and nutritive components of Shallot. *Journal of Medicinal Plants Research*, *13*(18), 452–457. https://doi.org/10.5897/jmpr2019.6836

Wijayanti, I., Benjakul, S., & Sookchoo, P. (2021). Preheat-Treatment and Bleaching Agents Affect Characteristics of Bio-calcium from Asian Sea Bass (*Lates calcarifer*) Backbone. *Waste and Biomass Valorization*, *12*(6), 3371–3382. https://doi.org/10.1007/s12649-020-01224-w

Xiang, L. Y., Mohammed, M. A. P., & Baharuddin, A. S. (2016). Characterisation of

microcrystalline cellulose from oil palm fibres for food applications. *Carbohydrate Polymers*, *148*, 11–20. https://doi.org/10.1016/j.carbpol.2016.04.055

Xu, K., Guo, M., Du, J., & Zhang, Z. (2019). Okra polysaccharide: Effect on the texture and microstructure of set yoghurt as a new natural stabilizer. *International Journal of Biological Macromolecules*, *133*, 117–126. https://doi.org/10.1016/j.ijbiomac.2019.04.035

Yang, H., Yan, R., Chen, H., Lee, D. H., & Zheng, C. (2007). Characteristics of hemicellulose, cellulose and lignin pyrolysis. *Fuel*, *86*(12–13), 1781–1788. https://doi.org/10.1016/j.fuel.2006.12.013

Yekta, M., & Ansari, S. (2019). Jujube mucilage as a potential stabilizer in stirred yogurt: Improvements in the physiochemical, rheological, and sensorial properties. *Food Science and Nutrition*, *7*(11), 3709–3721. https://doi.org/10.1002/fsn3.1230

Yohana Chaerunisaa, A., Sriwidodo, S., & Abdassah, M. (2020). Microcrystalline Cellulose as Pharmaceutical Excipient. *Pharmaceutical Formulation Design - Recent Practices*. https://doi.org/10.5772/intechopen.88092

Yousefi, M., & Jafari, S. M. (2019). Recent advances in application of different hydrocolloids in dairy products to improve their techno-functional properties. *Trends in Food Science and Technology*, *88*(October 2018), 468–483. https://doi.org/10.1016/j.tifs.2019.04.015

Yu, D., Kwon, G., An, J., Lim, Y.-S., Jhoo, J.-W., & Chung, D. (2021). Influence of prebiotic biopolymers on physicochemical and sensory characteristics of yoghurt. *International Dairy Journal*, *115*, 104915. https://doi.org/10.1016/j.idairyj.2020.104915

Yuvraj Khasherao, B. (2021). *The Pharma Innovation Journal 2021; 10(4): 92-97 Effect of shallot waste (Allium cepa L. var. aggregatum) and their extracts on quality of*

developed pasta. http://www.thepharmajournal.com

Zanhi, N. K., & Jideani, I. A. (2012). Physico-chemical and sensory qualities of soy and milk solids fortified low fat yoghurt. *African Journal of Agricultural Research*, *7*(38), 5336–5343. https://doi.org/10.5897/AJAR11.449

Zbikowska, A., Kowalska, M., & Pieniowska, J. (2018). Assessment of shortcrust biscuits with reduced fat content of microcrystalline cellulose and psyllium as fat replacements. *Journal of Food Processing and Preservation*, *42*(8), 1–10. https://doi.org/10.1111/jfpp.13675

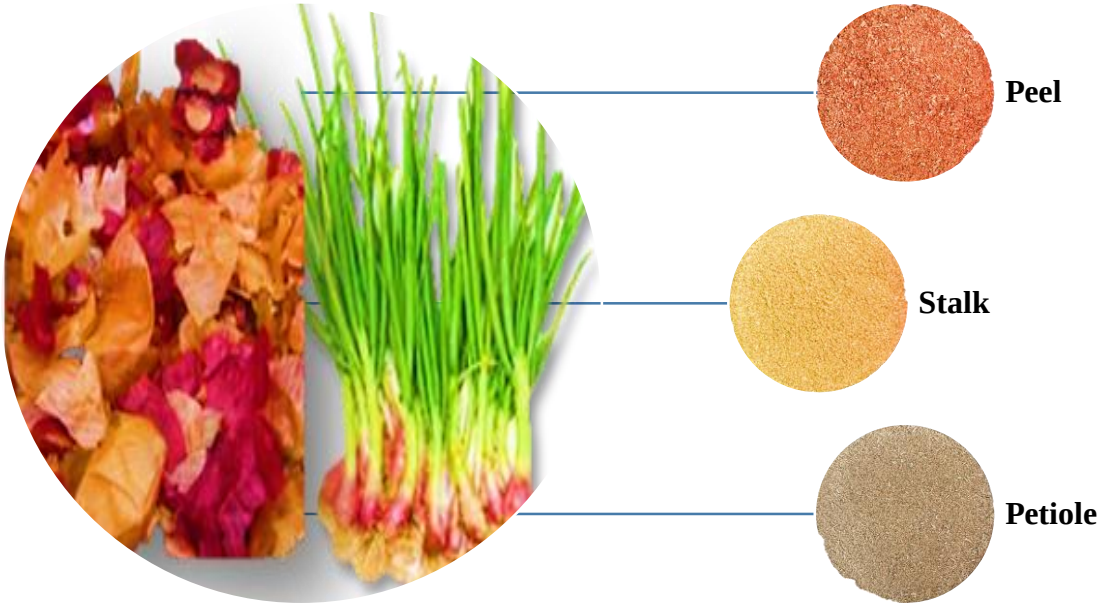
Zhang, T., Mccarthy, J., Wang, G., Liu, Y., & Guo, M. (2015). Physiochemical Properties, Microstructure, and Probiotic Survivability of Nonfat Goats' Milk Yogurt Using Heat-Treated Whey Protein Concentrate as Fat Replacer. *Journal of Food Science*, *80*(4), M788–M794. https://doi.org/10.1111/1750-3841.12834

Zhao, L. L., Wang, X. L., Tian, Q., & Mao, X. Y. (2016). Effect of casein to whey protein ratios on the protein interactions and coagulation properties of low-fat yogurt. *Journal of Dairy Science*, *99*(10), 7768–7775. https://doi.org/10.3168/jds.2015-10794

Zhao, T., Chen, Z., Lin, X., Ren, Z., Li, B., & Zhang, Y. (2018). Preparation and characterization of microcrystalline cellulose (MCC) from tea waste. *Carbohydrate Polymers*, *184*(September 2017), 164–170. https://doi.org/10.1016/j.carbpol.2017.12.024

ANNEXURES

Annexure 1. Pictorial representation of Shallot waste streams (SBWs) into fine powder samples



Shallot waste streams

Annexure 2(a): Effect of time, temperature and concentration factors convoluted in extraction of cellulose and MCC from shallot wastes pre-treated with hot water (W) assistance.

Sample	Conc. Of NaOH (N)	Cellulose (%)	Total Ash (%)	MCC (%)	Total Ash (%)	Colour		
						L*	a*	b*
WP1	1	46.55±3.05a	18.9±0.12a	35.51±0.51a	12.55±0.17a	31.72±0.67h	3.21±2.33d	10.44±0.13h
WP2	1.5	46.22±0.97a	18.0±0.41a	30.46±5.06ab	4.18±0.13bc	33.19±0.01h	2.603±2.41e	11.42±0.10g
WP3	2	38.48±2.79b	13.3±0.23c	19.75±0.69ab	2.82±0.14cd	47.66±0.42g	7.003±2.42a	21.65±0.07b
WP4	1	38.87±0.96b	14.4±0.42b	27.53±5.06b	6.50±0.29b	53.25±0.01f	5.02±0.01b	23.25±0.01a
WP5	1.5	35.89±0.29bc	10.6±0.34e	28.17±2.78ab	7.25±0.23b	65.71±0.36b	2.34±0.08f	11.90±0.17ef
WP6	2	31.58±0.92c	3.11±0.36g	15.11±0.48d	0.941±0.05d	76.73±0.01b	1.84±0.01g	11.54±0.01fg
WP7	1	34.72±1.16bc	11.9±0.10d	19.97±1.02cd	2.991±0.01cd	57.36±1.04e	4.03±0.06c	15.52±0.05d
WP8	1.5	26.94±0.40d	5.35±0.34f	24.55±0.63bc	3.221±0.52cd	69.04±0.20d	2.73±0.06e	16.03±0.33c
WP9	2	26.85±0.40d	4.92±0.09f	19.09±0.46cd	1.416±0.31d	71.2±0.87c	2.23±0.01f	12.12±0.02e
WS1	1	46.31±0.46a	15.8±0.47a	33.04±1.53a	4.27±0.05a	47.79±0.01h	6.99±0.03a	21.69±0.01c
WS2	1.5	45.11±0.52ab	14.4±0.17b	32.99±2.22a	3.82±0.11ab	53.41±0.01g	5.32±0.04b	25.12±0.01a
WS3	2	41.89±0.91bc	11.42±0.12c	20.68±0.55d	1.76±0.18ef	65.88±0.04c	3.71±0.01d	16.08±0.06e
WS4	1	42.91±2.19ab	8.38±0.21d	29.14±1.00b	3.08±0.05bc	48.21±0.03h	6.98±0.03a	21.70±0.02c
WS5	1.5	38.64±0.18cd	5.79±0.3e	24.23±1.73cd	1.36±0.27fg	62.17±0.55e	4.01±0.08c	23.72±0.19b
WS6	2	34.48±0.20e	3.64±0.37f	13.41±1.28e	0.95±0.05g	74.58±0.46a	2.07±0.04f	15.78±0.07f
WS7	1	41.18±3.03bc	10.87±0.11c	28.11±0.47bc	3.28±0.43bc	57.23±0.06f	4.09±0.01c	15.49±0.03g

WS8	1.5	36.42±0.24de	3.56±0.19f	20.98±0.83d	2.19±0.14e	64.62±0.08d	3.70±0.05d	16.38±0.10d
WS9	2	36.22±1.28de	2.77±0.28f	10.45±0.21e	1.34±0.26fg	67.53±0.35b	2.76±0.031e	15.96±0.13ef
WPT1	1	42.83±10.24a	13.8±0.54a	34.42±2.88a	9.226±0.2a	59.88±0.02d	6.75±0.03a	26.43±1.07ab
WPT2	1.5	43.90±3.30a	11.8±0.11b	29.03±4.83ab	7.452±0.304b	64.12±2.45c	3.20±0.43e	12.78±1.40e
WPT3	2	39.09±0.50a	6.78±0.56d	21.32±0.40bc	4.56±0.26c	62.45±0.05cd	4.20±0.02cd	10.50±0.06f
WPT4	1	40.61±0.63a	8.33±0.64c	28.90±5.22ab	6.287±0.311b	60.72±0.01d	4.62±0.02bc	25.70±0.06b
WPT5	1.5	34.89±1.23ab	4.82±0.62e	19.78±2.34c	3.067±0.661de	60.72±0.01d	4.64±0.01bc	25.79±0.01b
WPT6	2	25.697±0.441b	2.74±0.10f	16.92±3.14cd	1.191±0.485f	81.73±0a	0.71±0.01f	11.54±0.01ef
WPT7	1	39.15±2.78a	7.08±0.52cd	29.14±0.57ab	3.474±0.315cd	53.24±0.01e	5.02±0.01b	23.23±0.02c
WPT8	1.5	26.69±4.30b	1.13±0.07f	19.88±1.15c	1.972±0.06ef	73.26±1.02b	3.98±0.17d	21.29±0.32d
WPT9	2	22.81±4.55b	1.59±0.28f	10.74±0.53d	1.28±0.4255f	71.22±1.07b	3.19±0.23e	27.56±0.38a

*Results are expressed as mean ± SD (n=3). Different Superscripts within the same table mean statistical difference ($p < 0.05$) with confidence level 95% interval. Samples were termed as WP1(1N NaOH, 30min, 60°C), WP2(1.5N NaOH, 30min, 60°C), WP3(2.0N NaOH, 30min, 60°C), WP4(1N NaOH, 60min, 60°C), WP5((1.5N NaOH, 60min, 60°C), WP6(2N NaOH, 1h, 60°C), WP7(1N NaOH, 2h, 60°C), WP8(1.5N NaOH, 2h, 60°C), WP9(1.5N NaOH, 2h, 60°C)

Annexure 2(b): Effect of time, temperature and concentration factors convoluted in extraction of cellulose and MCC from shallot wastes pre-treated with pressurised steam (A) assistance

Sample	Conc. of NaOH	Cellulose (%)	Total Ash (%)	MCC (%)	Total Ash (%)	Colour Index		
						L*	a*	b*
AP	1N	46.67±1.15a	12.14±0.31a	31.41±1.71a	6.01±5.94a	32.75±0.31c	2.42±0.14b	10.91±0.41c
	1.5N	35.55±1.33bc	8.03±0.13b	23.39±2.20b	5.2±4.99ab	56.51±0.69b	4.03±0.05a	15.51±2.40b
	2N	29.31±1.94cde	1.55±0.20c	17.36±1.64e	0.049±0.06c	84.73±0.01a	3.91±0.18a	27.65±0.06a
AS	1N	37.51±1.13ab	14.1±0.24a	27.72±0.77ab	7.2±8.71a	44.80±0.66c	6.96±0.004a	23.08±0.22a
	1.5N	31.99±0.24bcd	7.29±0.33b	25.39±0.26abc	3.813±4.04b	47.97±0.19a	6.83±0.13a	26.65±0.32a
	2N	27.94±1.56de	3.32±0.20c	21.98±0.84cde	0.071±0.34c	79.54±0.87a	2.58±0.08b	13.71±0.005cd
APT	1N	38.41±1.34ab	5.66±0.73a	29.08±2.76a	3.237±3.64a	48.64±0.41c	6.80±0.01a	24.28±2.40a
	1.5N	32.91±0.91bcde	3.90±0.74a	24.45±1.73a	2.29±1.9ab	55.66±3.16b	4.73±0.09b	9.913±0.05b
	2N	25.70±0.34e	1.20±0.62b	14.0±0.84b	0.039±0.078b	82.41±0.01a	0.26±0.01c	9.416±0.005b

*****Results were expressed as mean ± SD (n=3). Different Superscripts within the table mean statistical difference ($p < 0.05$) with confidence level 95% interval

Annexure 3: Pictorial representation of extracted SBWs MCC samples (Peel(P), Stalk(S) and Petiole (S)).

PMCC

SMCC

PTMCC

Annexure 4(a): Qualitative analysis of extracted SBW's MCC samples

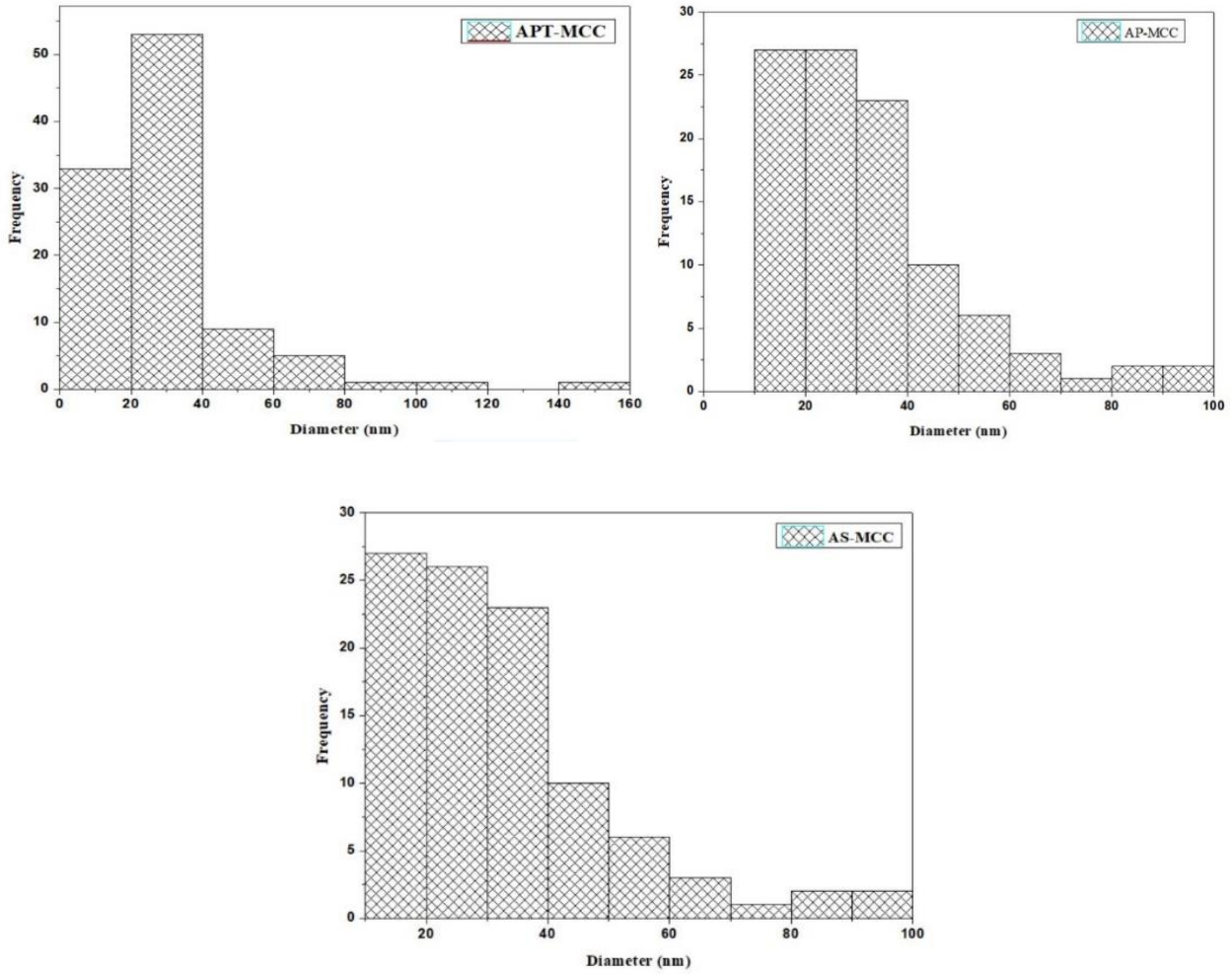
Organoleptic parameters	**PMCC**	**SMCC**	**PTMCC**
Colour	White	White	Off-white
Forms	Powder	Powder	Powder
Odour	-	-	-
Starch test	Negative	Negative	Negative
Sugar test	Negative	Negative	Negative
Crystalline test	Positive (red ppt)	Positive (red ppt)	Positive (red ppt)

Annexure 4(b): Comparative evaluation of selected samples of MCC through hot water and steam assisted pre-treatments extracted from shallot bio-waste.

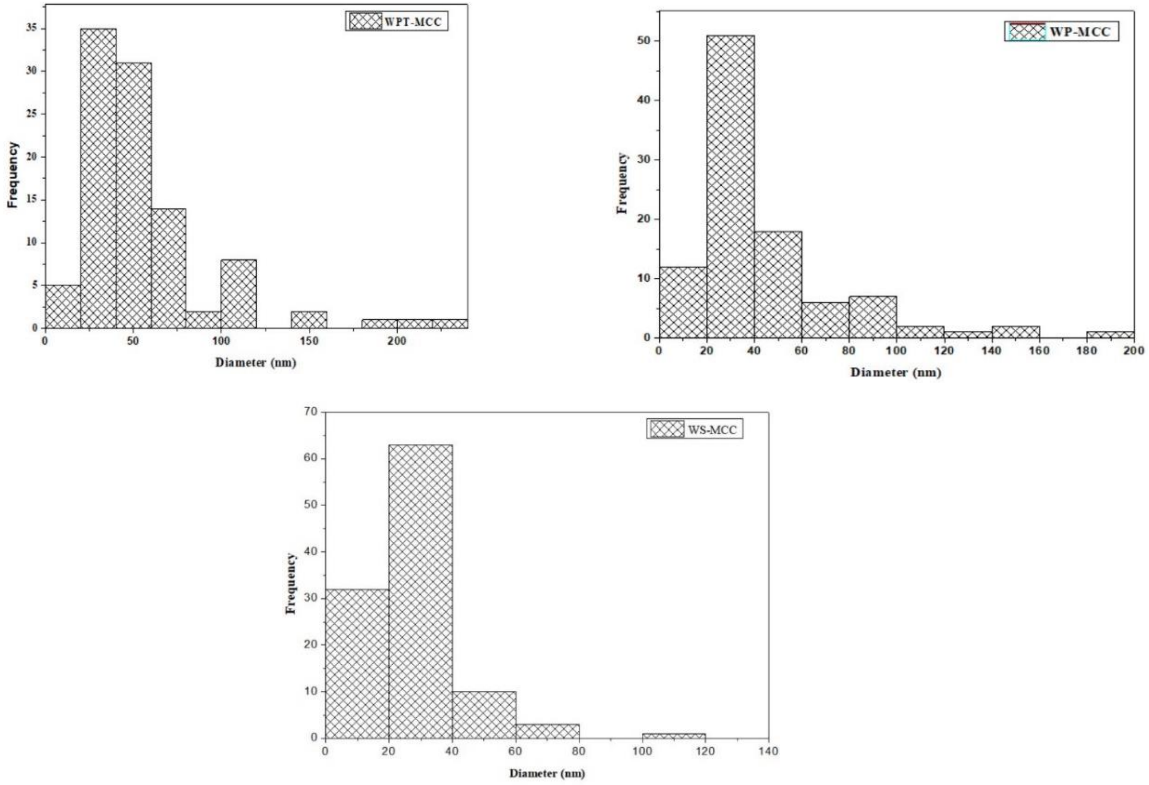
Samples	**Moisture (%)**	**Protein (%)**	**Fat (%)**	**Ash (%)**	**pH**	**ΔE**	**°H**
WP-MCC	3.67±0.5	0.154±0.08	0.397±0.001	0.941±0.05	6.25	10.19	1.55
WS-MCC	3.69±0.50	-	0.197±0.15	0.95±0.05	7.1	16.31	1.37
WPT-MCC	1.66±0.5	0.149±0.075	-	1.191±0.485	6.16		1.37
AP-MCC	1.42±0.25	-	-	0.185±0.113	6.5	19.09	1.54
AS-MCC	1.004±0.19	-	-	0.065±0.018	7	22.13	1.45
APT-MCC	1.53±0.35	-	-	0.065±0.037	6.8	91.97	1.42

*Pre-treatments denoted as W- hot water assisted and A-steam assisted. Values were analysed as % of wet basis and represented as mean ± SD (n=3)

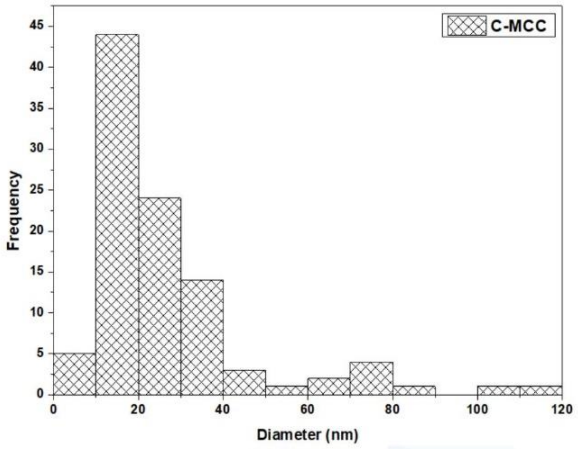
Annexure 5(a): Histogram of the Particle size distributions of MCC using imageJ software from SEM Images of hot water assisted samples at 500nm



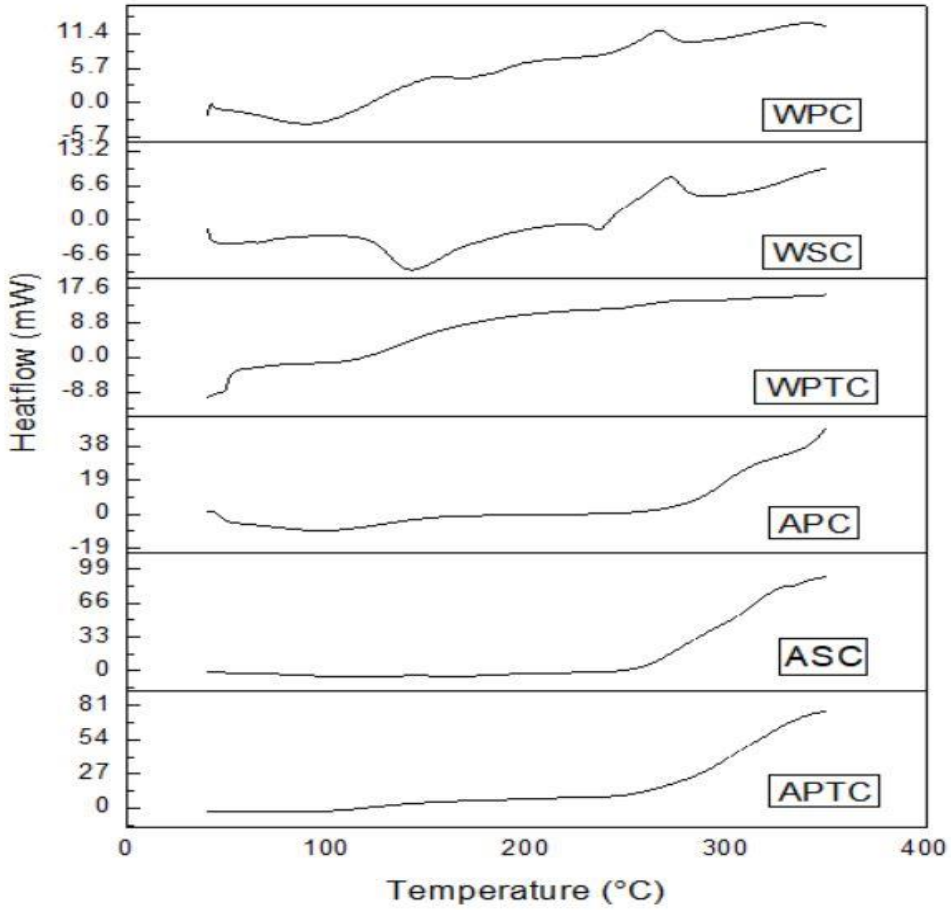
Annexure 5(b): Histogram of the Particle size distributions of MCC using imageJ software from SEM Images of steam assisted pre-treated samples at 500nm



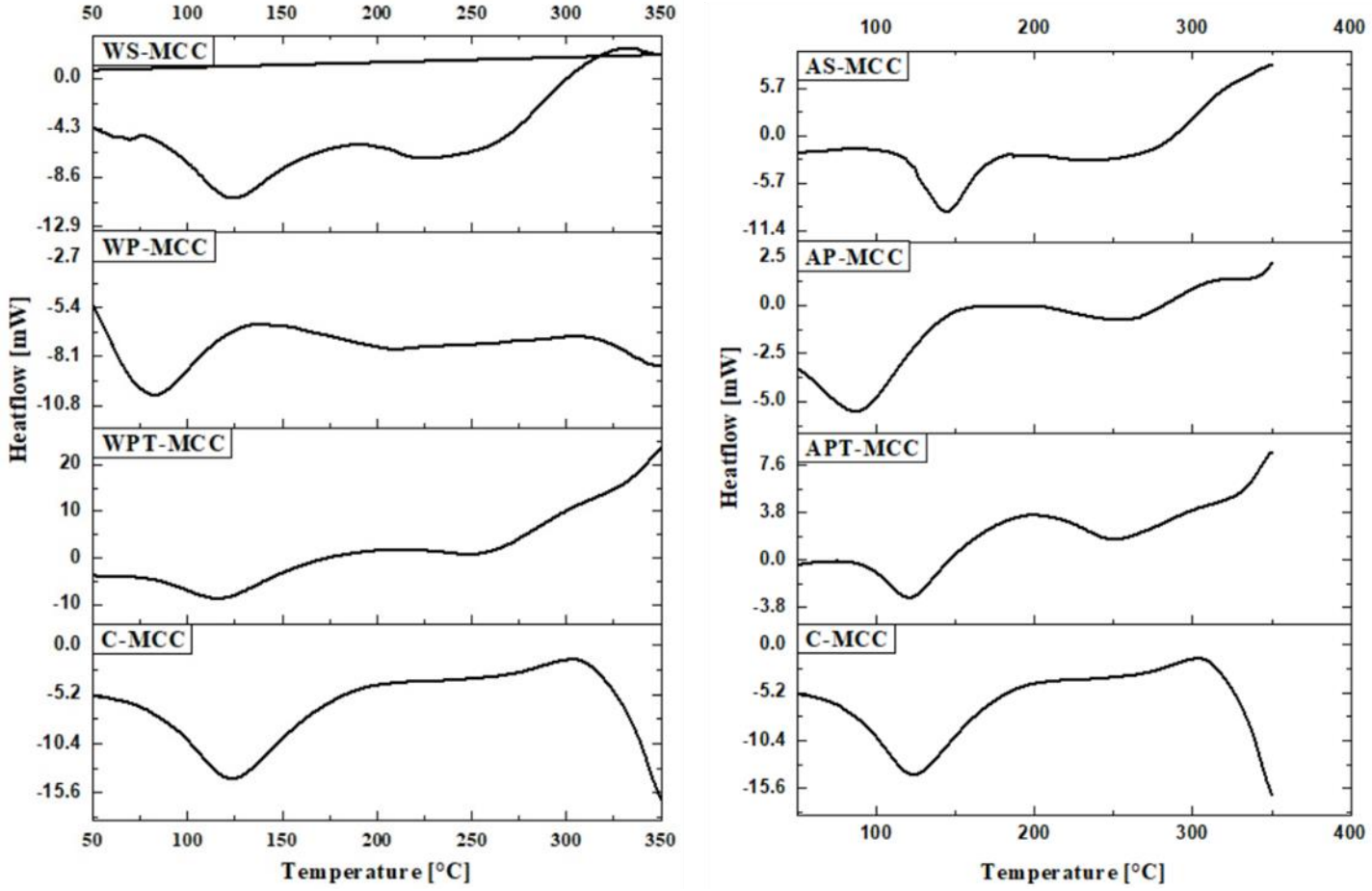
Annexure 5(c): Histogram of the Particle size distributions of MCC using imageJ software from SEM Images of control sample at 500nm



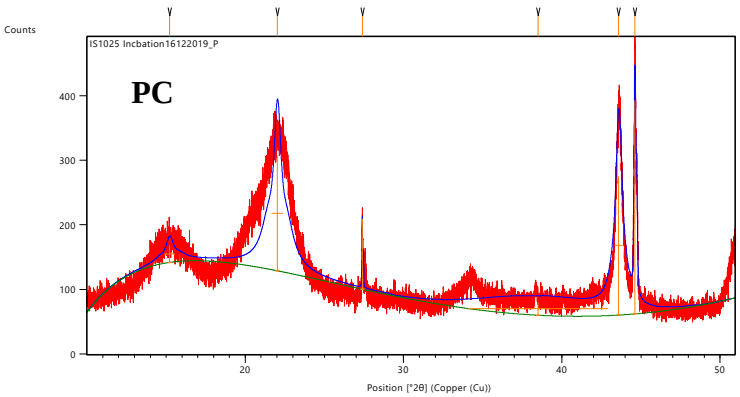
Annexure 6(a): DSC thermographs of hot water assisted samples (WPC, WSC &WPTC) and steam assisted extracted cellulose (APC, ASC & APTC) samples.

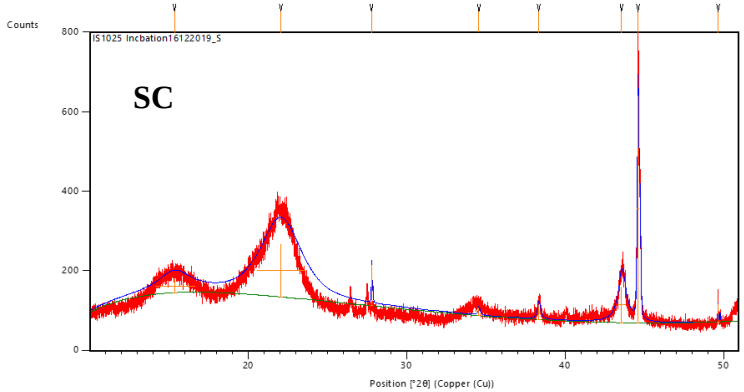


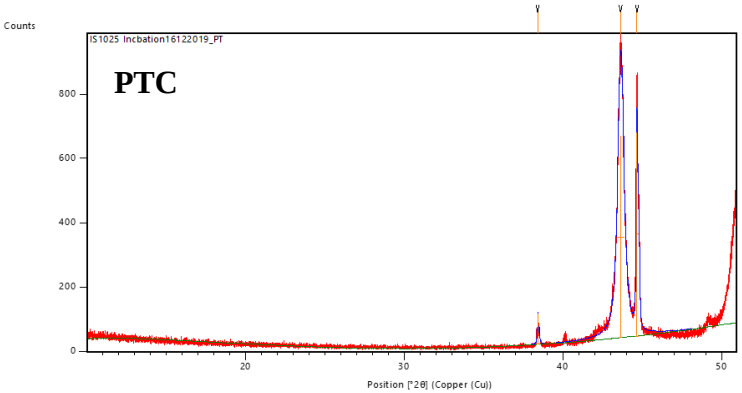
Annexure 6(b): DSC thermographs of hot water assisted samples and steam assisted extracted MCC samples.



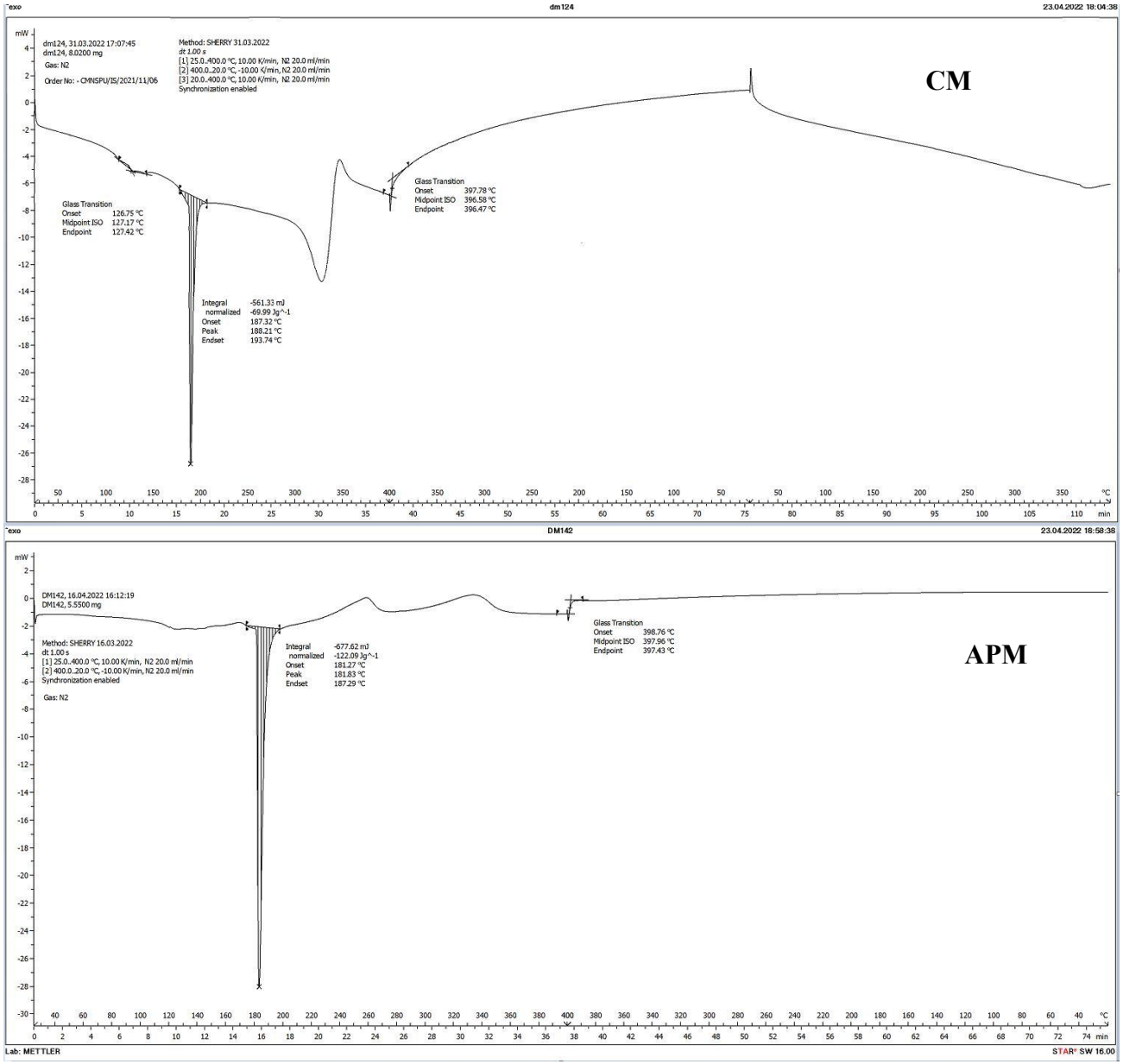
Annexure 7: XRD graphs of extracted SBWs cellulose samples (peel, stalk and petiole)

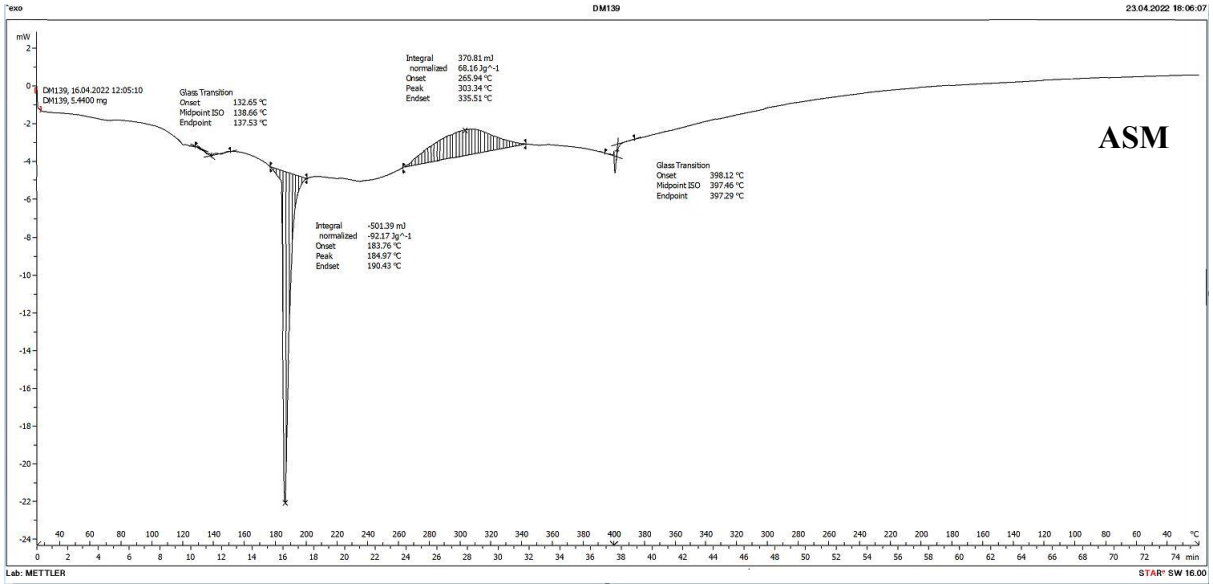


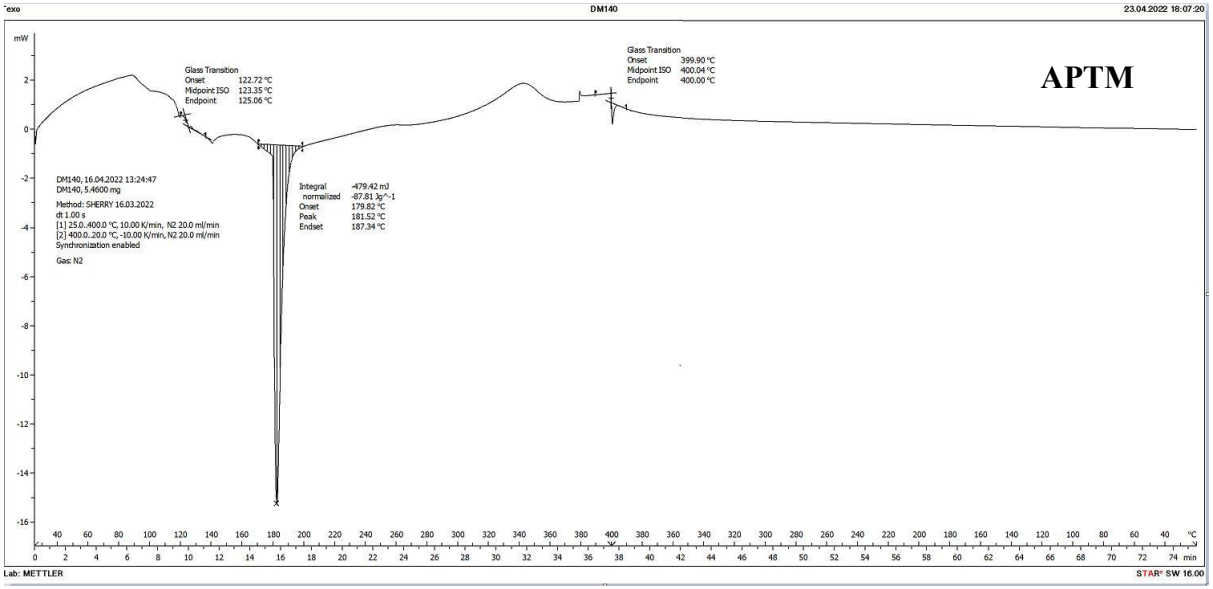


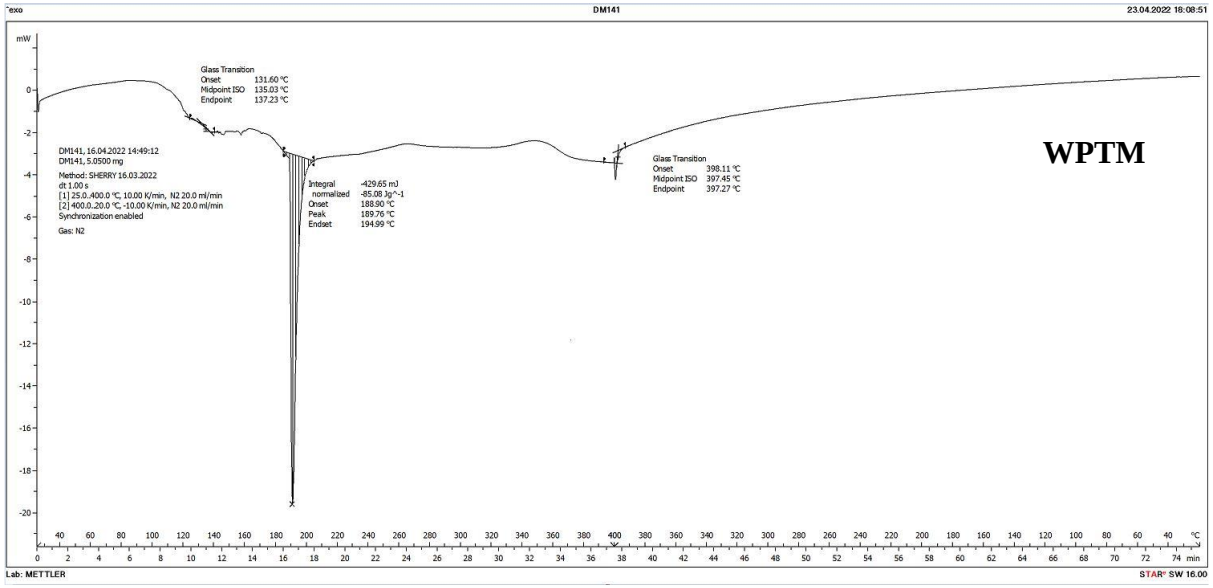


Annexure 8: DSC graphs of SBWs MCC samples ((CM (Commercial MCC), APM (Peel MCC), ASM (Stalk MCC) and APTM (Petiole MCC))

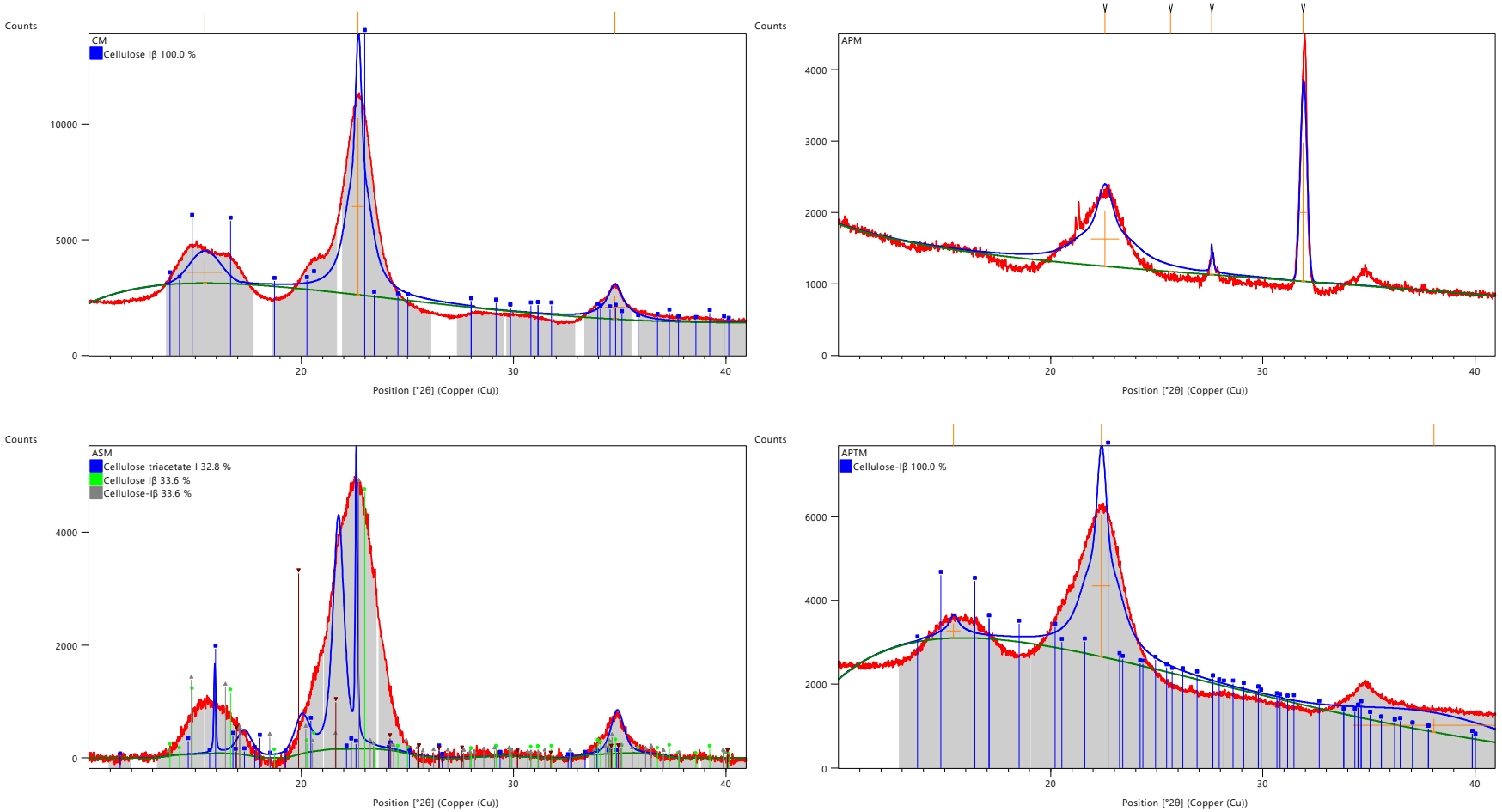


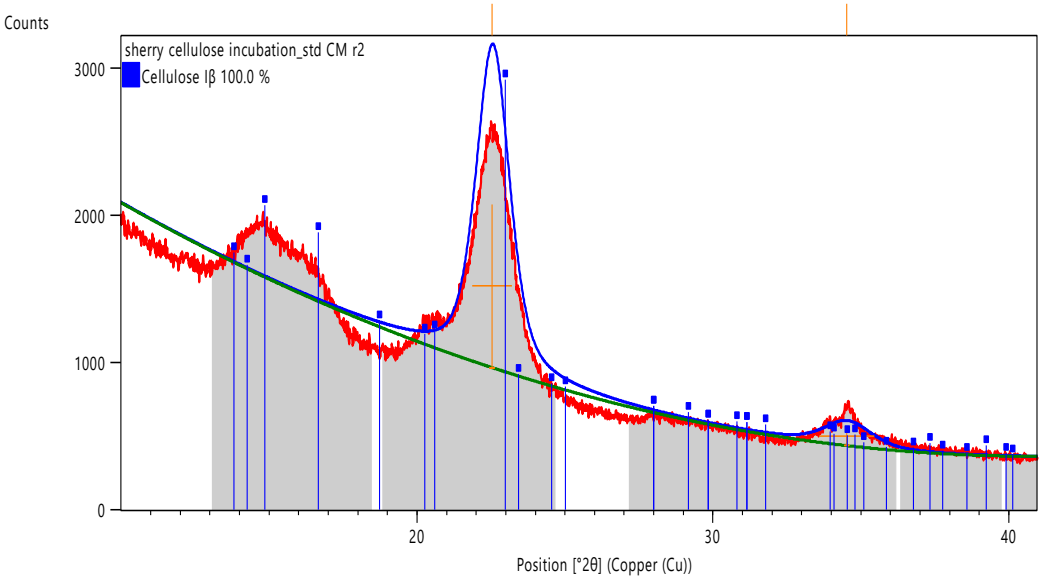

exo
DM139
23.04.2022 18:06:07
mW
DM139, 16.04.2022 12:05:10
DM139, 5.4400 mg
Glass Transition
Onset 132.65 °C
Midpoint ISO 138.66 °C
Endpoint 137.53 °C
Integral 370.81 mJ
normalized 68.16 Jg^-1
Onset 265.94 °C
Peak 303.34 °C
Endset 335.51 °C
Integral -501.39 mJ
normalized -92.17 Jg^-1
Onset 183.76 °C
Peak 184.97 °C
Endset 190.43 °C
Glass Transition
Onset 398.12 °C
Midpoint ISO 397.46 °C
Endpoint 397.29 °C
ASM
°C
min
Lab: METTLER
STAR° SW 16.00

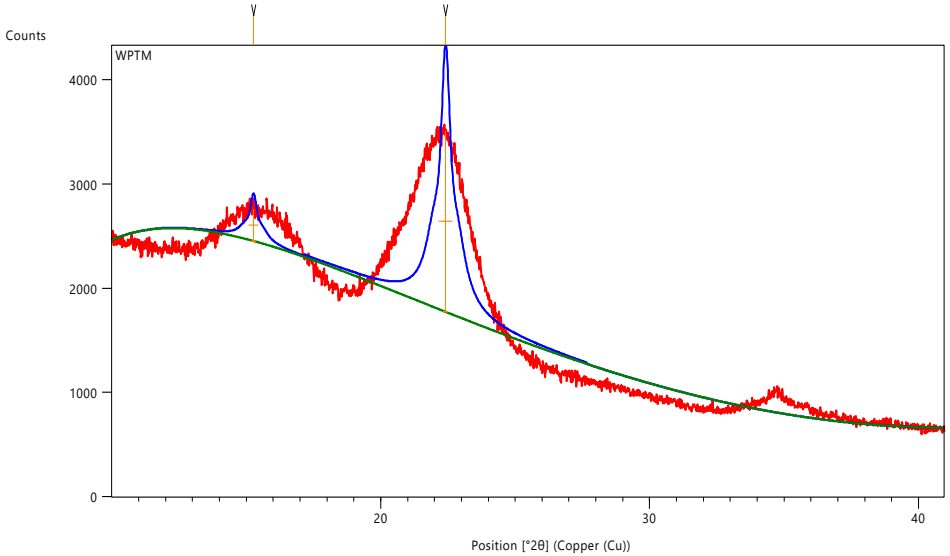

exo
DM140
23.04.2022 18:07:20
mW
Glass Transition
Onset 122.72 °C
Midpoint ISO 123.35 °C
Endpoint 125.06 °C
Glass Transition
Onset 399.90 °C
Midpoint ISO 400.04 °C
Endpoint 400.00 °C
APTM
DM140, 16.04.2022 13:24:47
DM140, 5.4600 mg
Method: SHERRY 16.03.2022
dt 1.00 s
[1] 25.0..400.0 °C, 10.00 K/min, N2 20.0 ml/min
[2] 400.0..20.0 °C, -10.00 K/min, N2 20.0 ml/min
Synchronization enabled
Gas: N2
Integral -479.42 mJ
normalized -87.81 Jg^-1
Onset 179.82 °C
Peak 181.52 °C
Endset 187.34 °C
°C
min
Lab: METTLER
STAR° SW 16.00


exo
DM141
23.04.2022 18:08:51
mW
Glass Transition
Onset 131.60 °C
Midpoint ISO 135.03 °C
Endpoint 137.23 °C
DM141, 16.04.2022 14:49:12
DM141, 5.0500 mg
Method: SHERRY 16.03.2022
dt 1.00 s
[1] 25.0..400.0 °C, 10.00 K/min, N2 20.0 ml/min
[2] 400.0..20.0 °C, -10.00 K/min, N2 20.0 ml/min
Synchronization enabled
Gas: N2
Integral -429.65 mJ
normalized -85.08 Jg^-1
Onset 188.90 °C
Peak 189.76 °C
Endset 194.99 °C
Glass Transition
Onset 398.11 °C
Midpoint ISO 397.45 °C
Endpoint 397.27 °C
WPTM
°C
min
Lab: METTLER
STARe SW 16.00

Annexure 9(a): XRD crystal phase changes in SBWs MCC (APM, ASM, APTM, WPTM) samples as compared to CC and CM.




Counts
sherry cellulose incubation_std CM r2
Cellulose Iβ 100.0 %
3000
2000
1000
0
20
30
40
Position [°2θ] (Copper (Cu))

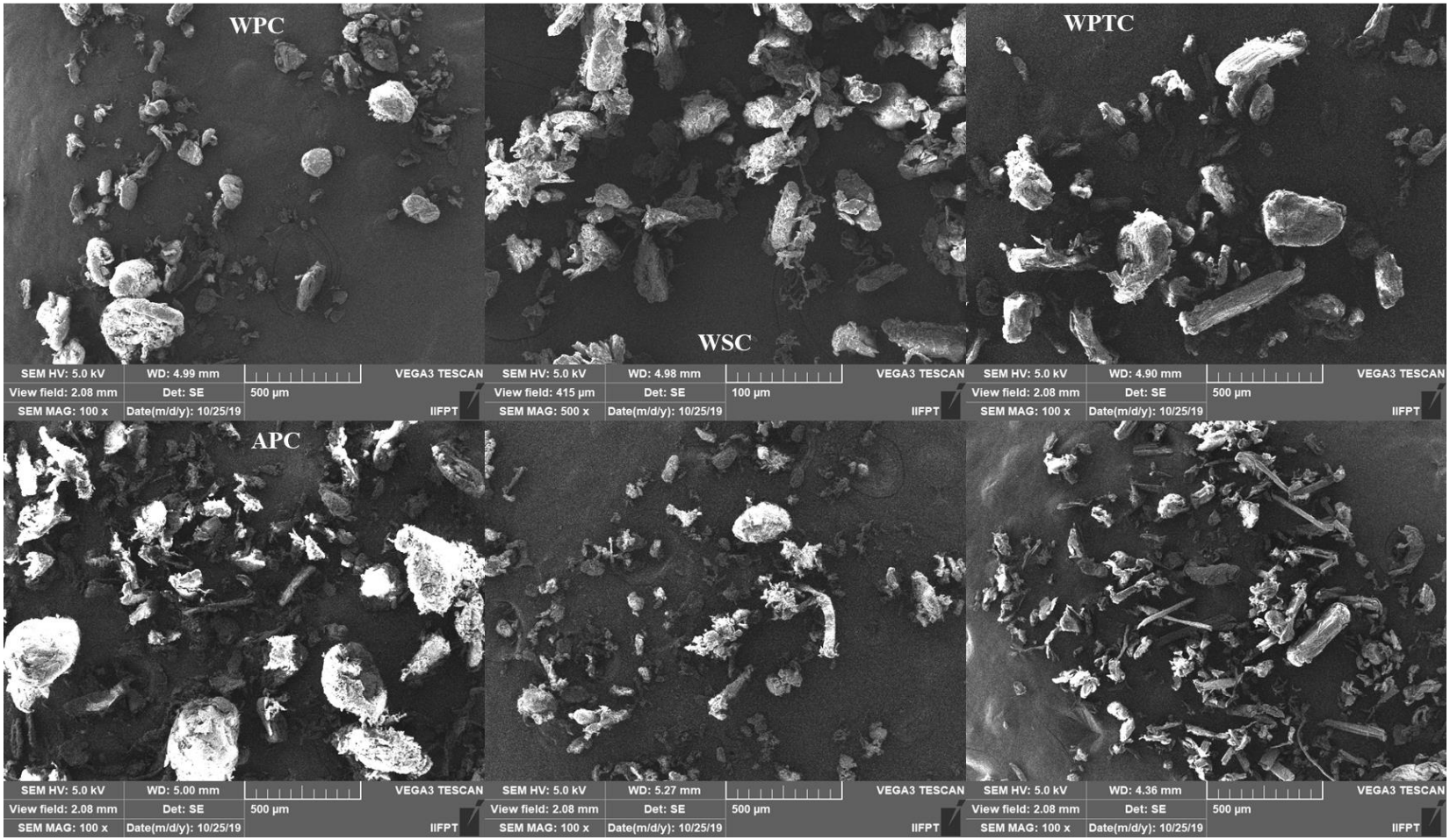

Counts
WPTM
4000
3000
2000
1000
0
20
30
40
Position [°2θ] (Copper (Cu))

Annexure 9(b): Crystallographic evidence of SBWs MCC samples (CM (Commercial MCC, APM (peel MCC), ASM (stalk MCC) and APTM (Petiole MCC).

Position angle [°2θ]	d-spacing [Å]	Height [cts]	FWHM Left [°2θ]	Shape Left	Integral Breadth [°2θ]	Area [°2θ]	Chemical Formula	Crystal System	Compound Name	Crystallite Size [Å]	Micro Strain only [%]	Crystallite Size [Å]	Micro Strain [%]
CC													
15.46	5.728	931.06	1.6986	-0.1	1.7516	1630.88	(C6 H10 O5)n	Monoclinic	Cellulose Iβ	51	5.629	-1245	5.62837
22.67	3.91851	7646.54	0.5478	1.5	1.1290	8633.49				86	2.28282	86	-0.13159
34.75	2.57918	1013.12	0.9312	1.136	1.5641	1584.64				62	2.06746	62	-0.07924
CM													
22.55	3.94046	1132.47	1.3432	1.212	2.346366	2657.18	(C6 H10 O5)n	Monoclinic	Cellulose Iβ	40	4.94823	40	-0.10837
34.52	2.59641	132.01	1.8793	0.224	2.155957	284.62				44	2.94817	196	2.51078
APM													
22.55	3.93923	768.15	1.3256	1.46	2.665968	2047.87	(C6 H10 O5)n	Monoclinic	Cellulose Iβ	35	5.64272	35	-0.09772
25.65	3.46961	0	0.026	-0.1	1000000	0.01				28625	0.00606	-1215	-0.121
27.59	3.2304	335.37	0.0666	1.5	0.137181	46.01				1458	0.11076	1462	-0.11188
31.90	2.80348	1932.12	0.3575	-0.1	0.368657	712.29				253	0.55475	-1254	0.55468

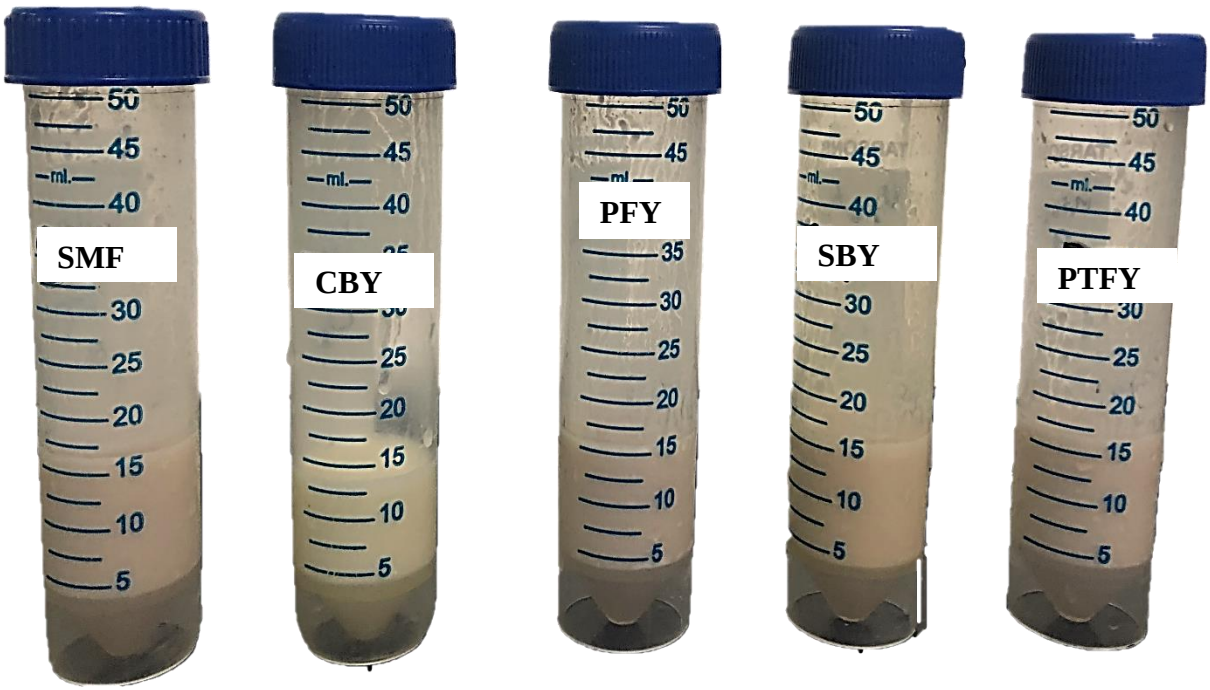
ASM													
15.91	5.56438	1193.14	0.09	0.6	0.12	143.18	(C6 H7 O2)n (C2 H3 O2)3)n	Monoclinic	Cellulose triacetate I	15223	0.01828	24382	-0.04868
17.31	5.12356	382.12	0.8187	0.6	0.807595	308.59	(C6 H10 O5)n	Monoclinic	Cellulose Iβ	111	2.30819	-1215	2.30793
20.01	4.43699	576.63	0.8187	0.6	0.807595	465.68	(C6 H10 O5)n	Monoclinic	Cellulose -Iβ	111	1.99122	-1226	1.99099
21.73	4.0891	3698.83	0.614	0.6	0.605696	2240.37	(C8.45 H14.9 O5)n	Orthorhombic	Methyl cellulose	149	1.36921	-1224	1.36906
22.58	3.93507	4260.35	0.09	0.6	0.12	511.24				13663	0.0144	19783	-0.02648
34.85	2.57411	696.72	0.7164	0.6	0.706645	492.34				132	0.97848	-1282	0.97837
15.42	5.74066	346.08	0.6774	1.316	1.252119	433.33	(C6 H10 O5)n	Monoclinic	Cellulose -Iβ	76	3.77154	76	-0.19544
APTM													
22.3846	3.96852	3385.89	0.8533	1.5	1.758572	5954.33	(C6 H10 O5)n	Monoclinic	Cellulose -Iβ	54	3.6947	54	-0.12359
38.0667	2.36202	310.05	7.6214	-0.1	7.859427	2436.81				12	9.94148	-1707	9.94037
WPTM													
15.2643	5.79991	307.78	0.3841		0.791532	243.62	(C6 H10 O5)n	Monoclinic	Cellulose -Iβ	125	2.31837	125	-0.20464
22.3996	3.9659	1735.48	0.5264		1.084849	1882.73				90	2.21447	90	-0.13382

Annexure 10: SEM micrographs of SBWs Cellulose samples (WPC, WSC, WPTC, APC, ASC, APTC)

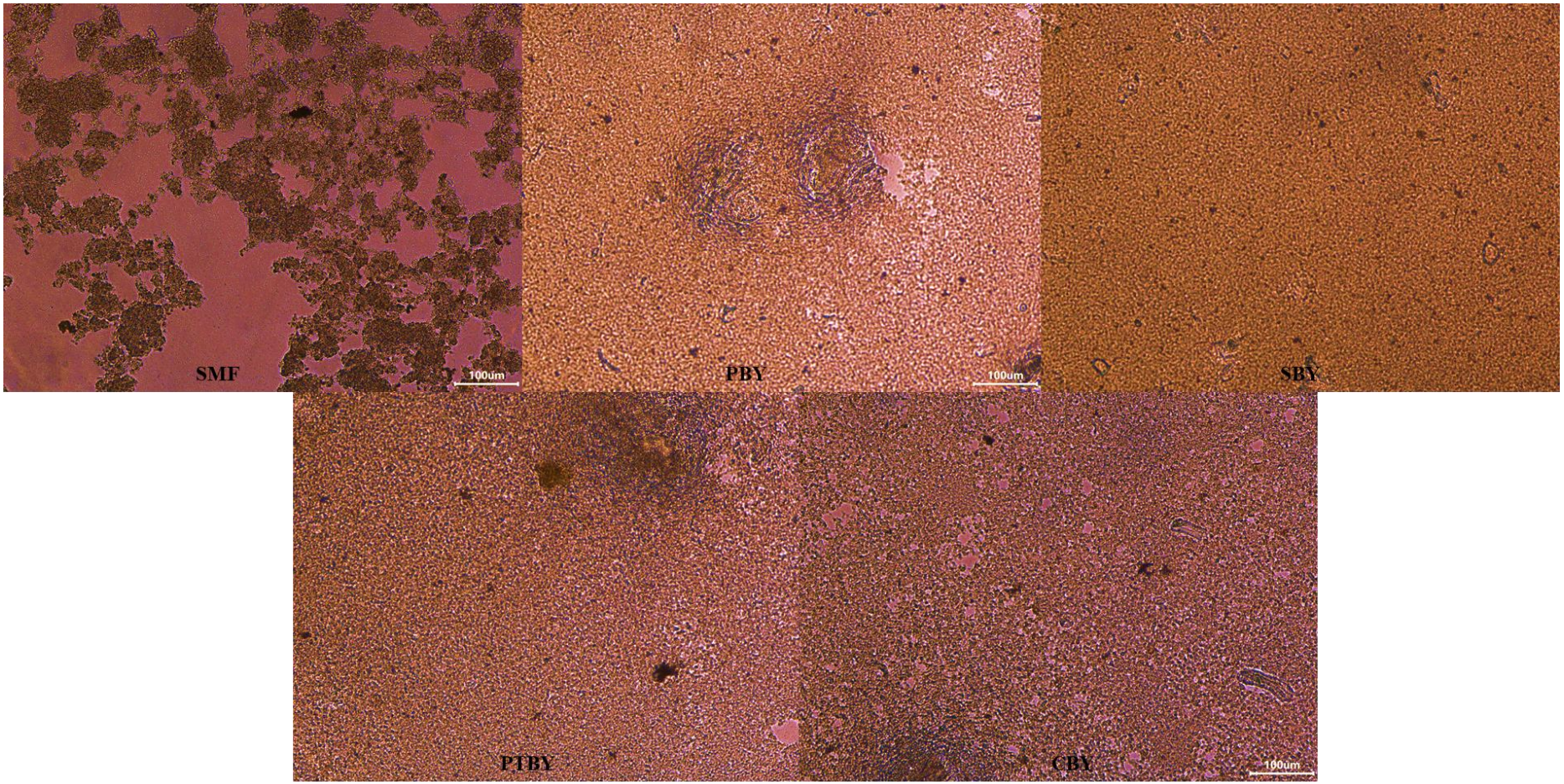


Annexure 11: Pictorial display of the low fat stirred plain yogurt (LFSPY) incorporated with SBWs MCC samples.

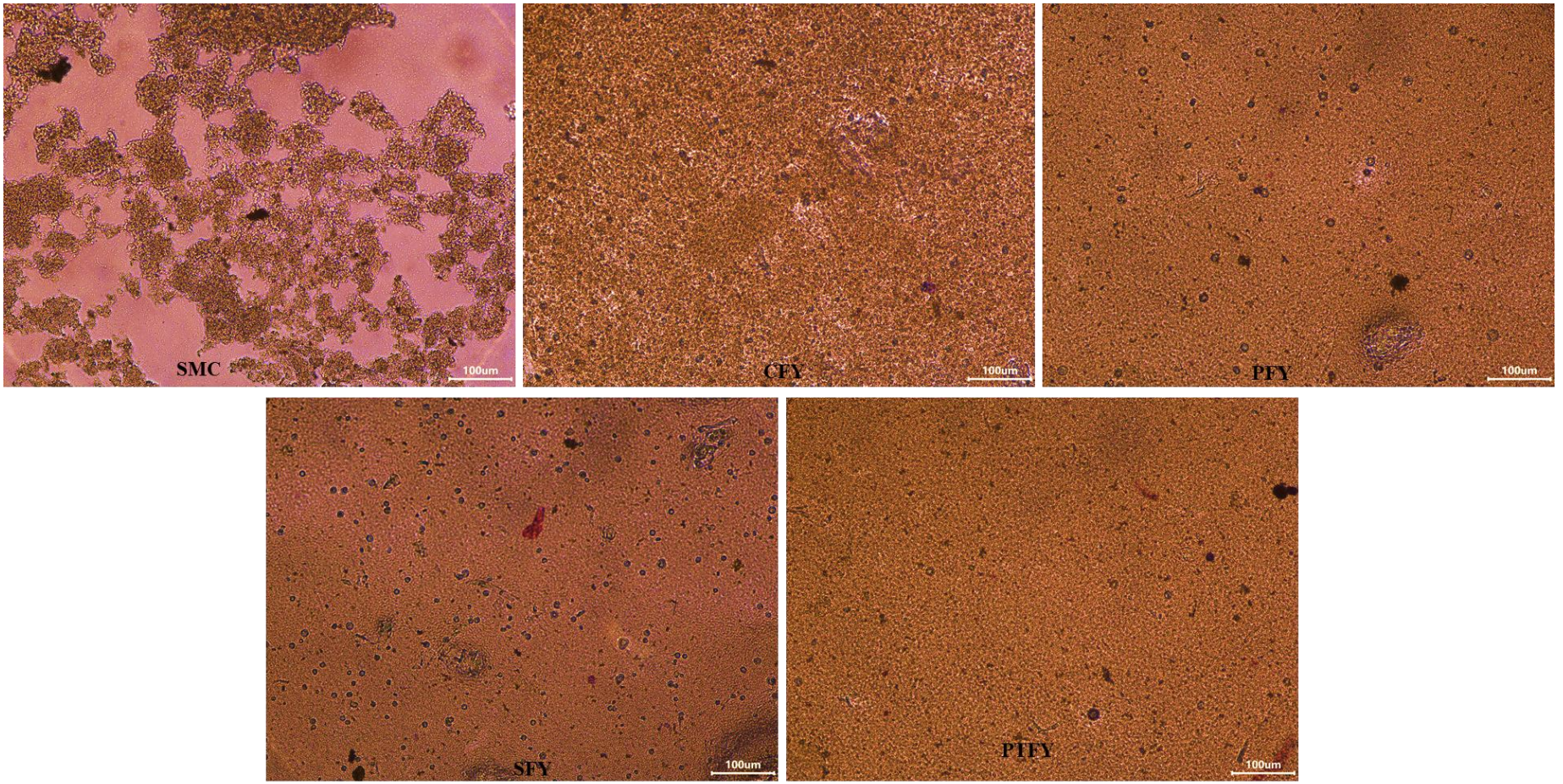
Annexure 12: Display of low fat fruit flavoured stirred plain yogurt (LFSPY) incorporated with SBWs MCC samples.



Annexure 12: microscopic images of the low fat stirred plain yogurt (LFSPY) incorporated with SBWs MCC samples using light microscope. SMF(control), PBY(P-MCC), SBY(SMCC), PTBY(PT-MCC) and CBY(C-MCC)



Annexure 12: microscopic images of the low fat fruit flavoured stirred yogurt (LFFSY) incorporated with SBWs MCC samples using light microscope (SMC-blank without MCC, CFY-LFFSY with commercial MCC, PFY- LFFSY with Peel MCC, SFY- LFFSY with Stalk MCC, PTFY-Petiole MCC)



PUBLICATIONS

List of published articles – Based on thesis

- Varghese, S. M., Bhosale, Y. K., Aruna Nair, U. K., Hema, V., & Sinija, V. R. (2022). Valorisation and Characterization of *Allium cepa var. aggregatum* Biowastes for the Production of Microcrystalline Cellulose. *Waste and Biomass Valorization*, *13*(4), 1931–1944. https://doi.org/10.1007/s12649-021-01625-5
- Shery M Varghese, Aruna Nair U K, S. V. R. (2021). Effect of incorporation of microcrystalline cellulose extracted from shallot bio-waste into low-fat stirred yogurt and its characterization. *International Journal of Botany Studies*, *6*(5), 577–584.
- "Comparative studies on functional and rheological properties of α-cellulose and Microcrystalline cellulose extracted from shallot waste streams with commercial samples" – Submitted to *"Cellulose"* (Springer publications) – Currently under review

Posters presented at International Conferences:

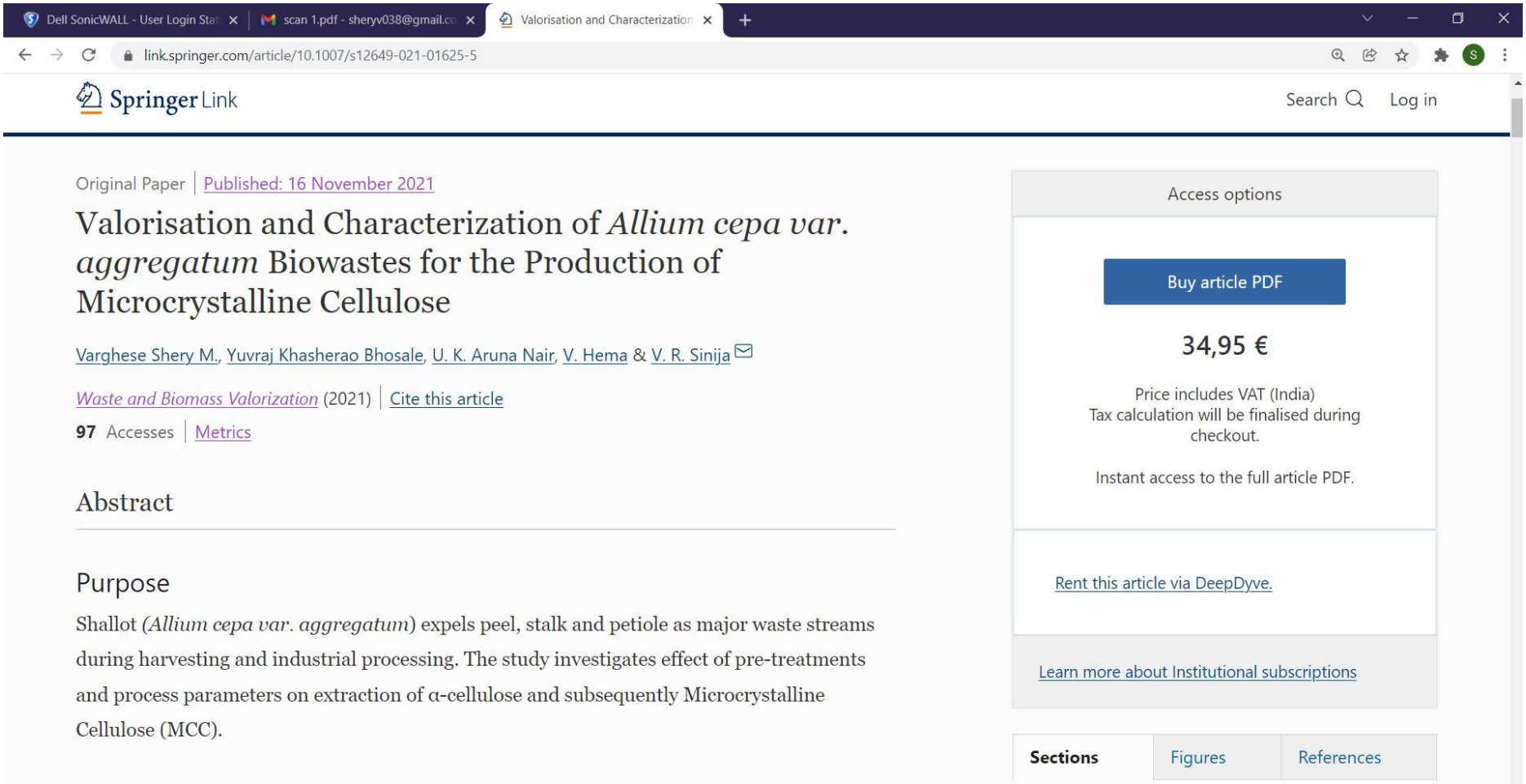
- Shery M Varghese, Bhosale Yuvaraj Khasherao, Aruna Nair U K, V R Sinija* **;** Extraction and characterization of prebiotic components in small onion biowaste; presented at IFCon (8th International Food Convention), December 2018

Other Publications:

- Bhosale, Y. K., Perumal, T., Varghese, S. M., Vincent, H., & Ramachandran, S. V. (2022). Utilization of shallot bio-waste (Allium cepa L. var. aggregatum) fractions for the production of functional cookies. *International Journal of Food Engineering*, *18*(1), 27–39. https://doi.org/10.1515/ijfe-2021-0169
- Bhosale, Y. K., Varghese, S. M., Thivya, P., Hema, V., & Sinija, V. R. (2020). Studies on assessment of safety and nutritional quality of shallot waste fractions. *Journal of Food Processing and Preservation*, e15147. https://doi.org/10.1111/jfpp.15147

Proof for research articles published in journals approved by UGC

Paper 1



Waste and Biomass Valorization
https://doi.org/10.1007/s12649-021-01625-5

ORIGINAL PAPER

Valorisation and Characterization of *Allium cepa var. aggregatum* Biowastes for the Production of Microcrystalline Cellulose

Varghese Shery M.[1,2] · Yuvraj Khasherao Bhosale[1] · U. K. Aruna Nair[1,2] · V. Hema[1] · V. R. Sinija[1]

Received: 26 May 2021 / Accepted: 25 October 2021

Abstract

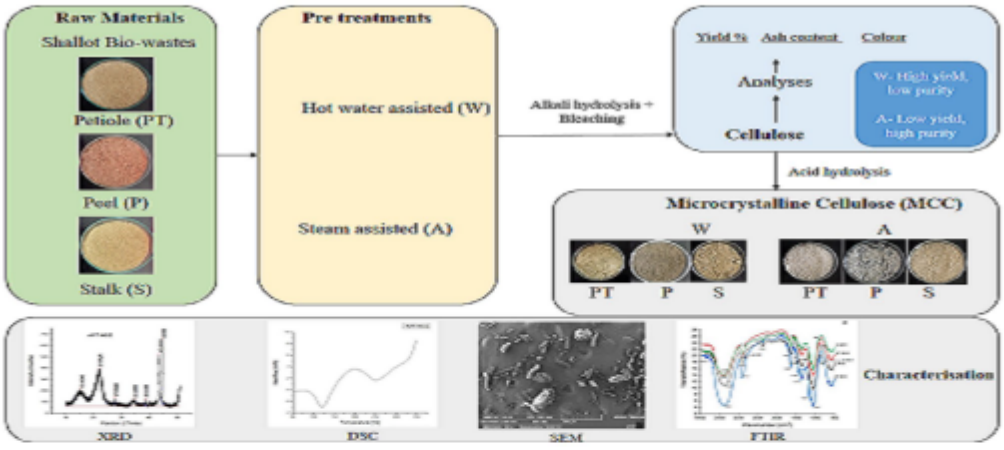
Purpose Shallot (*Allium cepa var. aggregatum*) expels peel, stalk and petiole as major waste streams during harvesting and industrial processing. The study investigates effect of pre-treatments and process parameters on extraction of α-cellulose and subsequently Microcrystalline Cellulose (MCC).

Method The extraction of cellulose and MCC was carried out by two pre-treatments such as hot water (60 °C) and autoclave method (121 °C) along with combined hydrolysis of alkali-bleaching-acid. The efficiency of pre-treatment with time, temperature and concentration of NaOH were evaluated.

Result The recovery yield of MCC from cellulose was high in stalks and peel (autoclave pre-treated) and petiole (hot water pre-treated) with 79.39, 59.23 and 55.63% respectively. The FTIR spectra of samples showed that removal of lignin and hemicellulose was better in autoclave pre-treated samples. Scanning electron micrograph showed the morphological modifications in both pre-treatments. Crystallinity of extracted MCC was analysed with X-ray diffraction method and found high in hot water assisted petiole and autoclave assisted samples of petiole and stalk with 83.74, 79.93 and 76.75% respectively. The thermal resistance of extracted samples was analysed with DSC. The results of various parameters were compared with commercial MCC and showed close proximity.

Conclusion The autoclave assisted pre-treatment was better for the effective transformation of cellulose to MCC with combined chemical hydrolysis which was technically easier, economical with less energy consumption. In a nutshell, selected shallot wastes proved to be source of superior quality microcrystalline cellulose with potential for commercialization.

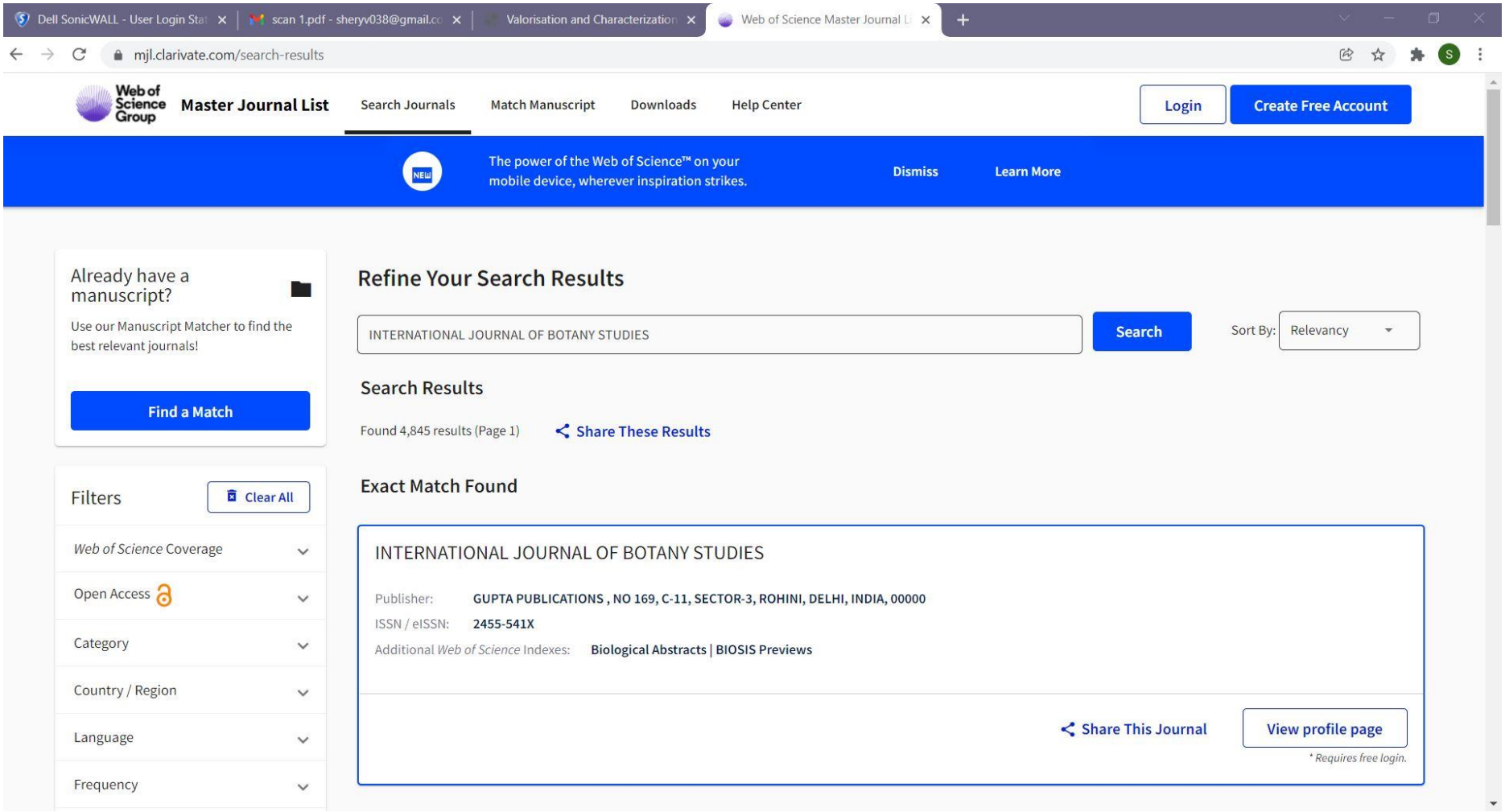
Graphical Abstract



Extended author information available on the last page of the article

Published online: 16 November 2021

Paper 2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Effect of incorporation of microcrystalline cellulose extracted from shallot bio-waste into low-fat stirred yogurt and its characterization

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Abstract

Microcrystalline cellulose (MCC) extracted from shallot peel was incorporated to develop low fat stirred yogurt. MCC was added at various concentrations ranging from 0.25-1% (P_1-P_4) and maintained at incubation temperatures at 37(T_1) and 43°C (T_2). The effect of concentration of MCC and incubation temperatures on the properties of the developed yogurts were studied. The increase in incubation temperature caused an increase in fermentation rate. Hence, pH of yogurt samples was decreased from 6.66 to 4.58 in P_3 and P_4 within 5 hours at T_2 and 10 hours at T_1. The concentration of MCC did not seem to affect the titrable acidity. At T_1, a decrease of TSS in P_4 from 12.7 to 6.3 in the 5th hour of the fermentation was observed. As compared to T_2, T_1 showed uniform increase of LAB count with time. Addition of MCC did not significantly affect the colour parameters, but caused an increase in moisture content upto 92.05±0.074 (P_4). Total ash content increased with increase in MCC at both temperatures, while protein content reduced with increase in MCC. Syneresis was observed to be higher in samples without MCC. With an increase in temperature and concentration of MCC, a significant increase in textural characteristics was observed. The pH and TSS values of yogurt decreased over the storage period with a simultaneous increase in bacterial count, more so at T_1. The sample with concentration of 1 % of MCC treated at 43 °C showed the best results compared to control samples with an acceptable microbial load.

Keywords: microcrystalline cellulose, low-fat, yogurt, shallot, peel, syneresis

Introduction

Now a day, low-fat yogurt is marketed and consumed in the form of plain, set and, stirred types incorporated along with additives and processing methods. Stirred or drinkable yogurts or smoothies are more trending's with increased demand because of their convenience, nutritious and probiotic effect [1]. According to market status, the growth rate of drinkable or stirred yogurt is predicted to reach a CAGR of 18% in 2023 with the takeover of 30% of the Indian yogurt market [2, 3]. However, globally the market sales were increased to 20% with CAGR expecting to be 12.5% in 2022 [4]. The most drawback of milk is the presence of saturated fatty acid which is the leading cause for cardiovascular disorders, obesity and hyper cholesterolemia condition [5]. Thus, consumer's preference for low or non-fat yogurt products has increased the awareness of milk producers to compete with innovative ideas to develop these products [6]. The most challenging part in the development and marketability of low-fat yogurts was product leads to low consumer's preference as compared to whole-fat yogurts [7].

During fermentation of milk, as it reaches the acidification stage, lactic acid bacteria converts lactose to lactic acid, calcium will expel to serum layer and fat molecules interact with casein micelles and aggregate to form three dimensional network with the help of electrostatic and hydrophobic bond formation [8]. The gel strength mainly depends on the amount of fat and protein, calcium, stirring speed and time, temperature and pH. Fat replacers and stabilizers play an important role as hydrocolloids which improve consistency, inhibit the serum separation rate and texture and other sensory qualities. Major fat replacers are sourced from, cellulose, starch and gums from plant or animal resources [9]. This defect can be overcome by addition of good quality hydrocolloids. Commonly utilized for the commercial purpose as fat replacers and extenders are sodium caseinate, whey protein concentrate, carboxy methyl cellulose, pectin, tragacanth, almond gums and locust bean gums [10]. Especially, the modified cellulose becoming more popular in recent years due to its better air entrapment, water holding capacity, creaminess, binding capacity and stabilizer [11]. Currently, microcrystalline cellulose (MCC) is popularly gained as a versatile additive along with combined attributes such as thickener, stabilizer and fat replacer in the dairy industry. MCC is derived from purified and depolymerized cellulose with a crystalline phase [12, 13]. It shows multidimensional functionality with stable viscosity, gelling power, emulsification capacity, textural modifier, fat replacer or mimicker, heat resistance and water binder. Commercially, MCC was produced from cellulose derived from hardwood and softwood with vigorous acid hydrolysis. Recently, it also valorised from agriculture and processed biomass especially sorghum husk, cotton linters, rice husk, palm husk etc., [14] Microcrystalline cellulose has better water holding capacity among modified cellulose to form a gel-like structure that improvises the