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Photocatalytic dye degradation activities of green synthesis of cuprous oxide nanoparticles from Sargassum wightii extract

P. Ramesh *, A. Rajendran

Research Department of Physics, Nehru Memorial College (Autonomous), Affiliated to Bharathidasan University, Puthanampatti, Tiruchirappalli, Tamil Nadu 621007, India

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ABSTRACT

In the present study, we report on the green synthesis of cuprous oxide (Cu_2O) nanoparticles using *Sargassum wightii* by single-step straightforward co-precipitation method. The preparation of Cu_2O nanoparticles is a fully green and environmentally beneficial process. The samples exhibit a homogeneous distribution and a uniform spherical shape and the size ranged between 20 and 30 nm, according to studies using the Field emission scanning electron microscope (FESEM). The existence of elements, Cu (42.09%) and O (57.91%), was confirmed by EDAX analysis. The X-ray Powder Diffraction (XRD) pattern supports the polydispersity and crystalline structure of the material. The presence of metal oxide functional groups was proven using Fourier-transform infrared spectroscopy(FTIR). The optical band gap of the Cu_2O is estimated to be about 3.11 eV based on the optical transmission at 383 nm using frequently used ultraviolet spectra. The photocatalytic degradation ability of the as-prepared sample was investigated against the methylene Blue (MB), methylene Orange (MO), and Rhodamine B (RB) dye under UV, and sunlight irradiation. According to the photocatalytic degradation analysis, the green synthesized Cu_2O nanoparticles show high photocatalytic degradation of methylene Orange 92% after 180 min under Sunlight irradiation. Cuprous oxide nanoparticles are an efficient activity that is advantageous to water purification and environmentally favourable applications.

1. Introduction

Nanotechnology is claimed to be an active place for researchers in the subject of contemporary material science, and it has been described as a modern scientific phenomenon. The top-down and bottom-up methods of nanoparticle synthesis are used in this study. These two techniques are entirely based on three distinct synthesis methods: chemical, biological, and physical [1]. Beyond the synthesis of metallic nanoparticles in general, and copper nanoparticles in particular, the basic idea is to provide three main components: a precursor to providing copper ions, a reducing agent to provide electrons required to reduce the copper ions into copper atoms, and a surfactant to control the size of the copper nanoparticles under the control of the third component, the surfactant, at the right pH and temperature [2].

Transitional metal oxides, such as Cu_2O, TiO_2, Fe_3O_4, ZnO, and NiO NPs, have shown promise as advanced nanomaterials in the fields of energy, biomedicine, and the environment. These NPs' excellent adsorption capacity considerably improves their performance and applications. Cuprous oxide nanoparticles (Cu_2ONPs) have been identified as having potential uses in gas sensors, waste treatment, catalysis, batteries, food preservation, high-temperature superconductors, solar energy conversion, photovoltaic devices, dye removal, field emission emitters, and agriculture [3]. On the one hand, even though noble metals like silver (Ag) and gold (Au) have been the subject of substantial investigation, their studies are still constrained by their expensive costs. However, copper (Cu) is an essential trace metal for people, plants, and animals [4].

In comparison to other metal particles (including gold, silver, iron, palladium, and zinc) copper nanoparticles are the most commonly utilised materials in the world due to their uses in electrical, optical, catalytic, antifungal, antibacterial, and biomedical fields. Due to their high surface-to-volume ratio, copper nanoparticles are highly reactive and may quickly interact with other particles [5]. Due to their affordability and availability compared to gold and silver metals, as well as their potential for use in a variety of industries, copper oxide nanoparticles are likewise becoming more and more popular [6].

Biosynthesis is being used extensively in the production of metal oxide nanoparticles. It removes the organic solvent from synthetic

* Corresponding author.
E-mail address: p.ramesh704@gmail.com (P. Ramesh).

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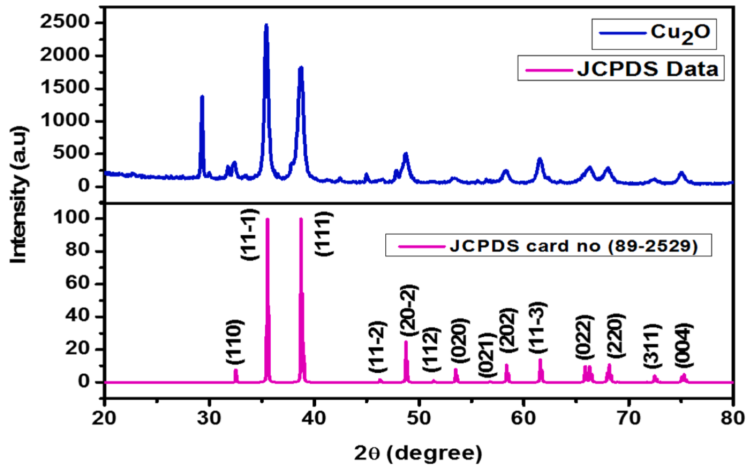

Fig. 1. Shows powder XRD of Cu_2O.

processes and eliminates harmful compounds created by chemical reactions. According to current research, plant-derived metal nanoparticles are safe, dependable, and environmentally friendly in physical and chemical systems. A Plant-mediated nanoparticle synthesis is a low-cost approach that will soon be a viable option for large-scale manufacturing [7]. As can be shown, there is a workable alternative known as "green" synthesis that enables reducing environmental impacts and improving yields when metal particles are impregnated on a nanometric scale. The "green" production of metal nanoparticles utilising a variety of natural reducing agents is the subject of several investigations [8].

Rapid worldwide development, the depletion of fossil fuels, unregulated urbanisation, and rising pollution levels have made the need for effective, affordable, sustainable, and environmentally responsible energy sources critical. As the textile and printing industries have grown, environmental contamination from organic pollutants and their effluents has become a major concern for water bodies, rendering them unsafe for drinking. Moreover, industrial waste from the processing of coal, industrial waste from the refining of petroleum, pesticides, and pharmaceutical manufacturing harm the environment by releasing harmful and hazardous phenolic chemicals [9–11]. MO is an azo dye that is used in the textile, food stuffs paper, and leather industries. The release of MO and its products into the environment, on the other hand, causes major pollution issues. Several countries are currently devoting significant resources to the development of water purification technology. Many dye removal processes have been developed by researchers all around the world, including photodegradation, adsorption, reverse osmosis, nanofiltration membrane, and oxidation [12].

The underutilised plant *Sargassum wightii* was used to prepare the Cu_2O nanoparticles in an environmentally friendly manner. The study of photocatalytic degradation showed high degradation efficiency against the MO dye under Sunlight irradiation. Our investigation indicates that the studies have used *Sargassum wightii* extract to create cuprous oxide nanoparticles and development of water purification technology.

2. Experimental procedure

2.1. Sample plant collection of Sargassum wightii

Sargassum wightii is a marine algal that was found on Rameswaram's east coast in Tamil Nadu, India's Ramanathapuram district. The sample area's latitude and longitude are 9.2843 °N and 79.3157 °E. Known norms to identify the culture as *Sargassum wightii* after sorting and classifying the collected algal samples according to species. The utilisation of antiquated guidelines allowed the culture to be identified as *Sargassum wightii* [13,14].

2.2. Preparation of Sargassum wightii extract

Initially, the freshly collected *Sargassum wightii* algae were sterile with running water and distilled water to get rid of any dust particles. The washed *Sargassum wightii* were dried at room temperature until the *Sargassum wightii* were completely dry and then completely crushed into a powdered form. Mix the *Sargassum wightii* powder with 100 ml of distilled water and then incubate the mixture at 40 °C for 10 min. Once the extract has cooled, filter it through Whatman No. 1 paper and store it at 4 °C for future research [15].

2.3. Preparation of cuprous oxide nanoparticles

The copper nitrate hexahydrate (0.5 M) powder was completely mixed with 100 ml of double-distilled water at room temperature for 30 min while being constantly stirred. The copper nitrate hexahydrate solution was then gradually given 2 g of sodium hydroxide. Then, 5 ml of *Sargassum wightii* extract was added to the above mixture and continually agitated for 2 h After that, the solution was centrifuged at 6000 RPM 3 times. The obtained product was dried for 120 min. The final product was crushed into a fine powder and then annealed at 400 °C in a furnace. Before the characterization, the obtained substance is mashed in a mortar and pestle [9–11]. The green synthesized Cu_2O nanoparticle was named cuprous oxide NPs.

2.4. Characterisation of cuprous oxide nanoparticles

XRD (model: X'PERT PRO PANalytical), FTIR (Perkin-Elmer Spectrometer), UV (Model: lambda 35, Make: Perkin Elmer), and FE-SEM (FE-SEM, JEOLJSM 6390) were used to characterize the green synthesised Cu_2O nanoparticles. The phase purity and crystallite size of the as-prepared sample was studied using X-ray diffraction (XRD). Additionally, the functional group of the green synthesized sample has been investigated using Fourier transform infrared spectroscopy (FT-IR). The optical behaviour of Cu_2O nanoparticles was investigated by UV–Visible absorption spectra in the wavelength range of 200–900 nm. The morphology, topography, and size of the nanoparticles were studied using field emission scanning electron microscopes (FE-SEM).

2.5. Photocatalytic reaction

The rate of photocatalytic degradation ability of the prepared sample was investigated against methylene Blue (MB), methylene Orange (MO), and Rhodamine B (RB) under UV light, and Sunlight irradiation of Cu_2O NPs. The 0.1 g of dye was dissolved in 100 ml of double-distilled water then 5 mg of Cu_2O NPs catalyst was added to the dye solution. The dye

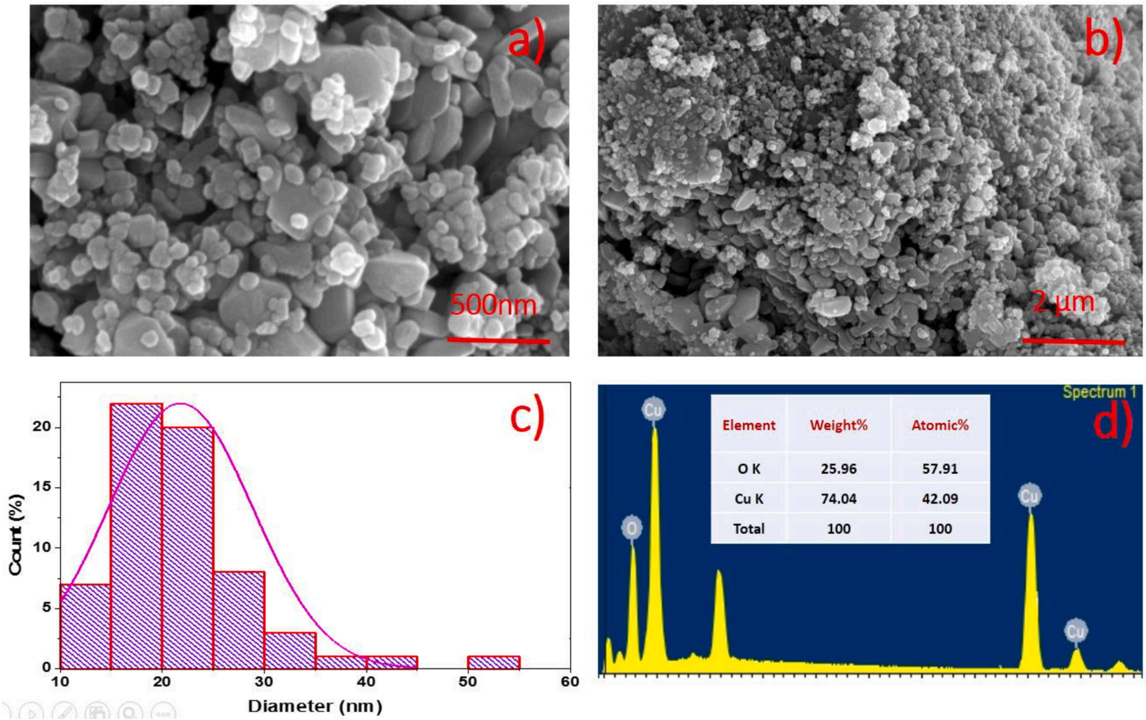

Fig. 2. a, b) FE-SEM image c) Particle Size Distribution Histogram, and d) EDAX Spectrum of Cu_2O NPs.

solution with nanoparticles catalyst was placed in a HEBER multi-lamp photo reactor, and was exposed to direct sunlight to measure the dye solution's capacity to degrade. At intervals of 30 min, the aqueous solution's dye degradation was examined.

3. Results and discussion

3.1. XRD analysis

The prepared Cu_2O NPs were characterized by X-ray diffraction (XRD) analysis and its observed diffraction pattern was shown in Fig. 1. The lattice planes of (002), (110), (111), (202), (020), (113), (310), (311), and (222) can be assigned corresponding to the observed peaks at the 2θ ranges of 28.99°, 32.26°, 35.63°, 38.78°, 48.75°, 53.84°, 58.22°, 61.48°, 73.38°, and 75.13°, respectively. The observed diffraction pattern was by standard JCPDS card No: (89–2529) for Cu_2O [15,16].

Using the Debye-Scherrer equation, the average crystallite size of the produced samples was calculated as follows:

$$\mathbf{D} = \mathbf{K}\lambda/\beta\mathbf{Cos}\theta$$

Where D is the average crystallite size, K is the Scherrer's constant (0.9), β is half of the maximum intensity, λ is the wavelength of X-ray radiation (0.154 nm), and θ is Bragg's angle, respectively. The greenly synthesised Cu_2O NPs had calculated average crystallite sizes of 22.53 nm.

3.2. Field emission scanning electron microscope (FE-SEM) analysis

Fig. 2 depicts the morphology, dimensions, and form of Cu_2O nanoparticles. The microscope pictures show spherical morphology, which was equally dispersed with little accumulation. The grain size of the as-prepared Cu_2O NPs was observed and is shown in Fig. 2a, 2b. The observed result of the particle size distribution histograms ranged from 20 to 30 nm (Fig. 2c) [17–19]. The EDAX spectrum confirms the chemical purity and elemental composition (Cu-42.09%, O-57.91%) of the synthesised Cu_2O NPs, as shown in Fig. 2d) [20].

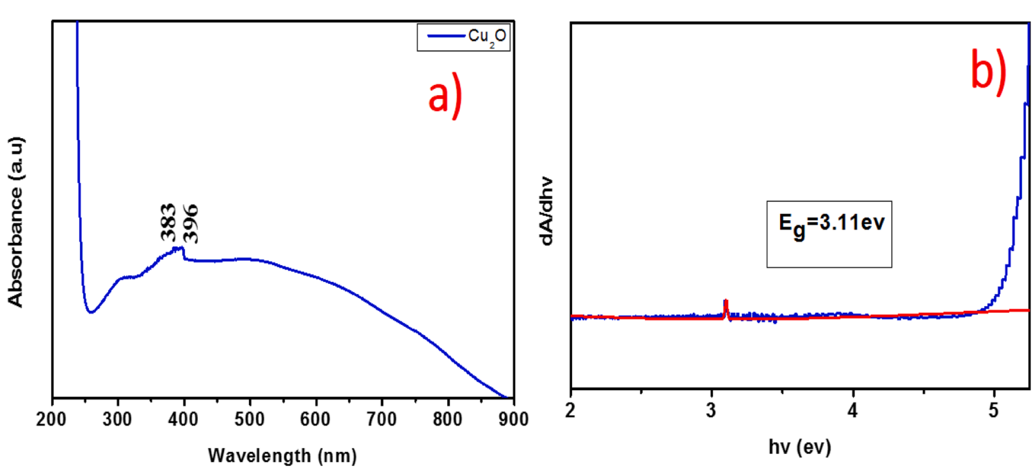

Fig. 3. (a) UV–visible absorption spectrum and (b) Bandgap energy of Cu_2O NPs.

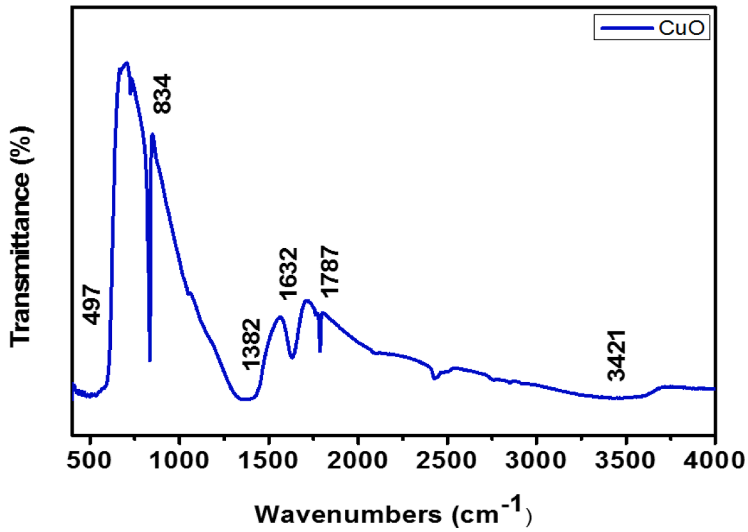

Fig. 4. Shows FTIR Spectrum shows of Cu_2O NPs.

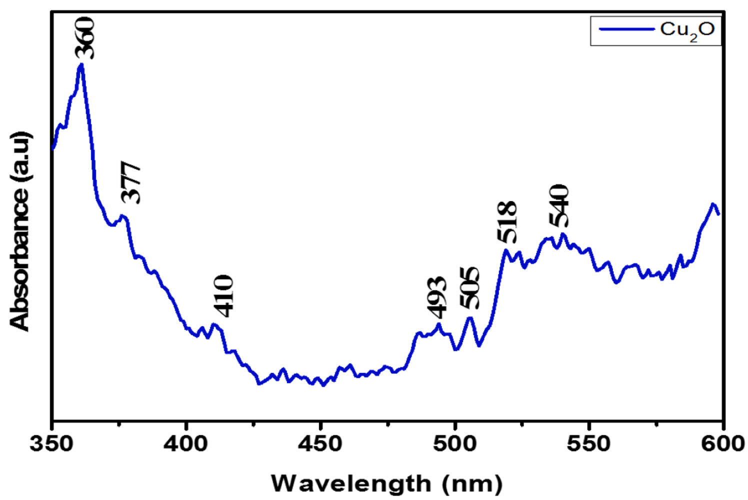

Fig. 5. Shows the PL study of Cu_2O NPs.

3.3. UV–vis spectra studies

An essential characterization technique in the photophysical study of the produced samples is ultraviolet-visible (UV–Vis) spectroscopy. Hence, the green synthesised Cu_2O NPs were investigated using an absorption spectrum with a wavelength range of 200-900 nm. The obtained result is depicted in Fig. 3(a). The sequence of absorption peaks was observed at 383 and 396 nm in the UV–Vis spectra (Fig. 3a) and which were indicative of the non-uniform chains of unequal length polydisperse nature of Cu_2O NPs [21,22].

Fig. 3b) shows the band gap energies of 3.11 eV The curves- show the pure Cu^{2+} phases of Cu_2O at 383 nm, respectively. The peaks in the derivative spectrum on the lower energy side serve as a gauge for the optical band gap value. [24,25], and Fig. 3(b) contains the acquired result. The Cu_2O NPs' predicted energy range is between 3.11eV

3.4. FT-IR spectra studies

FT-IR can quickly identify the structural features responsible for Cu bioreduction and acting as a capping and reducing and stabilizing agent when metal particles interact with molecule. [22,27].

Fig. 4 displays the FTIR spectra of *Sargassum wightii* extract after the production of Cu_2O NPs. The presence of alcohol in solution is indicated by certain important peaks at 3421 cm^{-1} for O—H O—H vibrational stretching, 1787 cm^{-1} for C = O stretching for carbonyl composite, 1632cm^{-1} for C—C and C—N stretching, and 1382 cm^{-1} for C—C and C—N stretching [23]. The presence of the Cu ion required for the production of Cu_2O was verified by the 834 and 497 cm^{-1} peaks [23]. Additionally, the additional functional groups support the presence of Cu_2O NPs in *Sargassum wightii* nanoparticles (Cu_2O NPs) were found to be surrounded by alcohol and carbonyl groups in the FTIR analysis, providing strong binding sites that supported this finding [23,24].

3.5. Photoluminescence (PL) analysis

Performance can be measured by analysing the band structure of Cu_2O NPs and the presence of defect-related levels within the band structure in photoluminescence (PL) spectroscopy. Studies on photoluminescence (PL) were carried out to highlight its emission characteristics. The Cu_2O sample's photoluminescence revealed 7 emission bands at 360 nm, 377 nm, 410 nm, 493 nm, 505 nm, 518 nm and 540 nm (Fig. 5).

The Cu_2O crystal's default configurations coincide with the blue band at 410 nm. A shift between oxygen vacancy and interstitial oxygen may be related to the blue-green band seen at 505 nm [18].

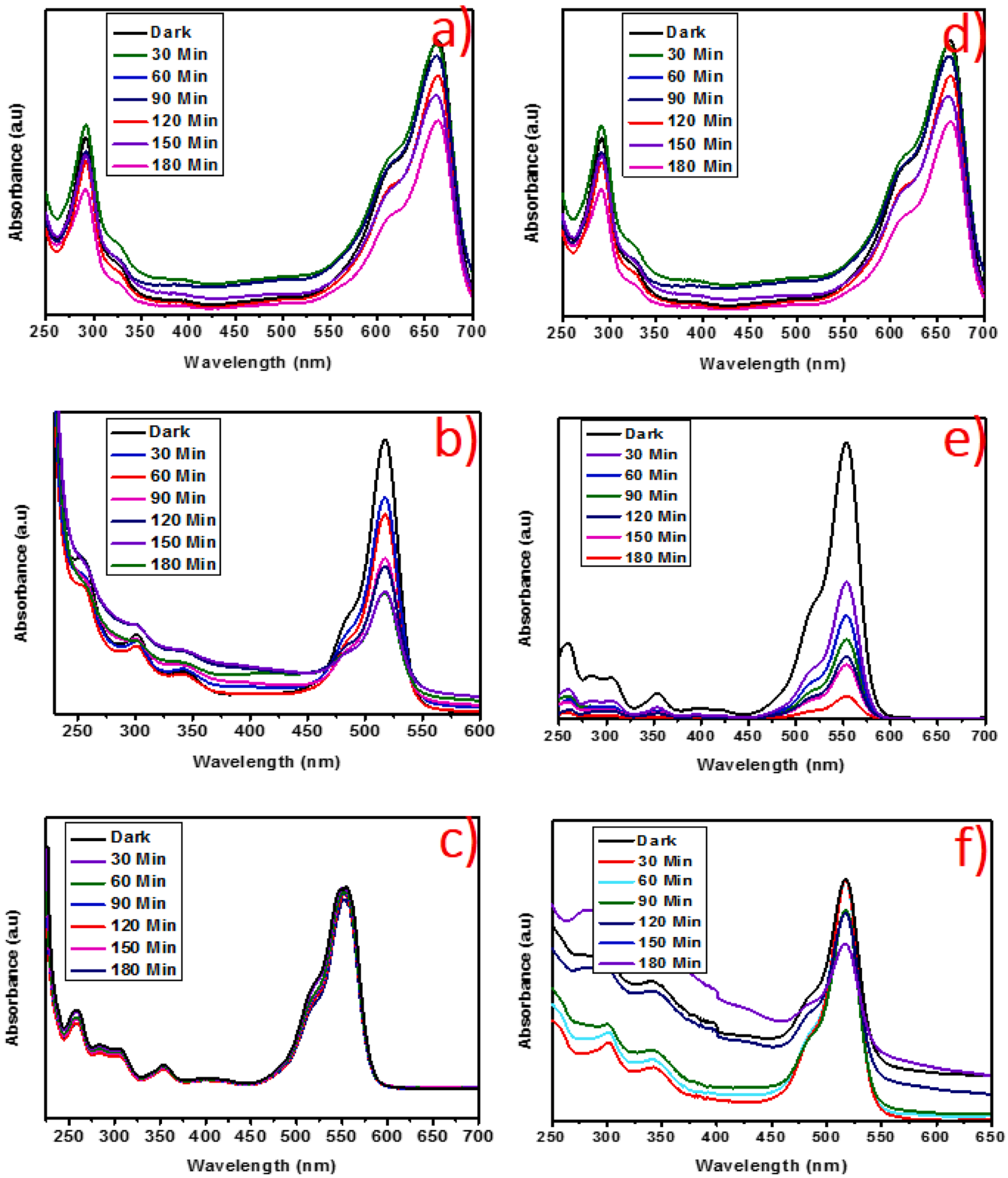

Fig. 6. a). MB UV Light b) MO UV Light c) RB UV Light d) MB Sun Light e) MO Sun Light f) RB sun Light assisted photocatalytic degradation.

3.6. Photocatalytic activity

The Cu_2O nanoparticles can exhibit strong photocatalytic activity against MB, MO, and RB dyes due to their larger surface area and smaller crystal size. The UV absorption spectra of MB, MO, and MR dye were shown in Fig. 6a, b, c, d, e and f over 180 min of different light irradiations. In particular Fig. 6 Sunlight and UV light irradiated over the as the photocatalytic reaction was shown. For hydrophilic substances like azo dyes, the main reaction routes involve the bulk liquid, where they may be efficiently eliminated by oxidative destruction by hydroxyl radicals [24]. The presence of one or more azo bonds (-N@N-) in combination with aromatic systems and auxochromes often identifies azo compounds (–OH, –SO_3, etc.) [26]. By combining photocatalysts with electron-scavenging species such as organic molecules, metals, and metal oxides, numerous methods have been developed to date to suppress the recombination of photo-induced charge carriers, a frequent process of photocatalytic processes [27].

Although the holes in the valence band (VB) change water molecules (H_2O) into hydroxyl radicals (OH-) and dye molecules into oxidised dye radicals, the electrons in the conduction band (CB) reduce oxide molecules (O_2) into superoxide radical ions (O_2-). As a result, the dye is broken down into CO_2, H_2O, and a few mineral acids by the photo-generated superoxide, hydroxyl, and dye-free radicals [28]. Because of their increased surface area, bio-synthesised Cu_2O NPs exhibit a more pronounced photocatalytic activity [29].

$$\%\mathbf{D} = (\mathbf{A_0} - \mathbf{A_t})/\mathbf{A_0}\mathbf{X100}$$

Where D is the% degradation, A_0 is the absorbence of the initial dye solution, and A_t is the absorbence after illumination.

The photocatalytic degradation efficiency of prepared samples was shown by the observed steady reduction in absorbence intensity at max = 670 nm from MB, 554 nm from MO and 552 nm from RB Cu_2O nanoparticles had a 70% degradation effectiveness against MO dye within 150 min of UV light irradiation (Fig. 6e). Fig. 7 shown the

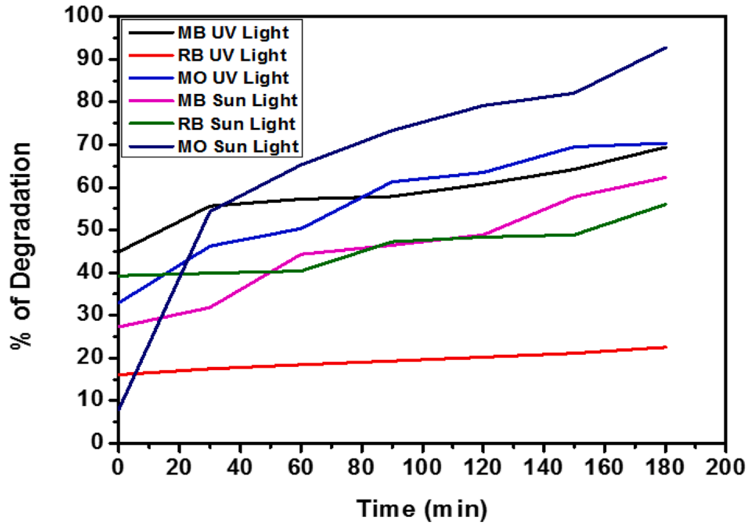

Fig. 7. photocatalytic degradation efficiency of Cu_2O nanoparticles.

photocatalytic degradation efficiency of different dye light source. Due to its high UV light absorption, it shows high photocatalytic degradation efficiency under Sunlight irradiation. [18,3032].

Conclusion

Green synthesis of cuprous oxide was effectively synthesised using an extract from the medicinal *Sargassum wightii* plant. The *Sargassum wightii* extract, which is inexpensive and eco-friendly, was tested as a potential stabilising and reducing agent. The cuprous oxide was initially synthesised in a transparent brown colour, and their optical characteristics for the bandgap energy are $Eg = 3.11$ eV FTIR spectroscopy was used to confirm the existence of functional molecules, and XRD analysis showed that Cu_2O NPs are in crystalline nature. The SEM showed that the green synthesised Cu_2O nanoparticles had spherical shape morphology. According to the photodegradation study, Cu_2O under Sunlight MO degrades by 92% after 180min of exposure. Finally, this work shows that the Cu_2O nanoparticles is an efficient activity that is advantageous to water purification and environmentally favourable applications.

Research data

All Data Included in Manuscript file.

CRediT authorship contribution statement

P. Ramesh: Conceptualization, Formal analysis, Investigation, Writing – original draft. **A. Rajendran:** Supervision, Validation.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

No data was used for the research described in the article.

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