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Frankia consortium extracts high-value metals from e-waste

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ABSTRACT

The microbial extraction method can be improved by adopting *Frankia* consortium native isolates with unique features for extracting high-value metals from e-waste. The goal of this research would be how native *Frankia* consortium isolates retrieved high-value elements from e-waste. Analytic techniques such as AAS, SEM with EDX, and XRD were used to examine the morphological arrangements and composition of high-value metal element resources in e-fine particles of e-waste. The detection reveals that high-value metal components such as Ag, Au, Cu, and Zn are present. The *Frankia* consortium was utilized in two leaching processes to bioleach high-value metal element resources from e-fine particles. Since the two-step bioleaching approach yielded the most effective high-value metal recovery when compared to the one-step bioleaching process. While comparing the two-step method to the one-step method, the synthesis of secondary metabolites, phosphatase enzyme, and organic acids by *Frankia* consortium was shown to be higher in the two-step method. The bioleaching process is mostly influenced by the above-mentioned *Frankia* consortium by-products. The FT-IR spectrum confirmed the existence of organic acids functional groups during the e-fine particle and *Frankia* consortium interactions. Leaching of high-value metal elements and control e-fine particles were found by AAS spectra analysis. As a result, the starting concentrations of high-value metal elements are Au (0.04 mg/g), Ag (0.04 mg/g), Cu (0.12 mg/g), and Zn (0.10 mg/g), respectively. As a consequence, the e-fine particles' two-step at 0.2 percent density demonstrated that they are capable of leaching metal concentrations such as Au (0.11 mg/g), Ag (0.09 mg/g), Cu (0.17 mg/g), and Zn (0.15 mg/g). Analytical techniques such as XRD, SEM with EDX, AAS, and FT-IR were used to validate high-value metal resources recovered.

1. Introduction

According to recent reports, e-waste printed circuit boards (PCBs), a major component of e-waste, have become increasingly prevalent in modern electronic technology, resulting in a large volume of e-waste globally due to their short lifespan (Marappa et al., 2020b). As per the United Nations Environment Programme (UNEP), e-waste dumped a record 53.6 million tonnes in 2020 (Narayanasamy et al., 2021b). According to a report by the Platform for Accelerating the Circular Economy (PACE) and the UN E-waste Association, global e-waste generation is on track to reach 120–130 million tonnes by 2050 if not effectively managed (Diagne et al., 2013). With a total of 10.1 million tonnes of e-waste produced, China is the world's greatest producer. Conversely, with a total of 6.9 and 3.2 million tonnes of e-waste created, the United States and India were the second and third-largest contributors in the global e-waste production, respectively (Ardente

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et al., 2019). Approximately 25%–30% of worldwide e-waste gets recycled each year, implying that 70–75 percent of e-waste is disposed of in landfills, burned, or improperly processed (Bajestani et al., 2014). Relays, capacitors, resistors, and integrated circuits, among other electric components, are all contained on PCBs. Metals are divided into two categories: dangerous and poisonous metals, and precious metals. Gold, silver, and platinum, which account for 0.3 percent, 0.2 percent, and 0.1 percent of all precious metals, are the most common. Copper, aluminium, nickel, tin, and lead are the toxic or dangerous metallic elements, accounting for 19.19%, 7.06%, 5.35%, 2.03%, and 1.01 percent, respectively (Narayanasamy et al., 2017).

The most important reason for this view is that nearly all e-waste contains toxic compounds that are both unpleasant and environmentally harmful. Lead, mercury, cadmium, chromium, and nickel are the most dangerous metals, as they have a detrimental impact on humans and can cause tumour death as a result of their exposure. Furthermore, this can pollute the entire ecosystem, posing health risks to both wildlife and humans who live in that environment (Hu et al., 2021; Narayanasamy et al., 2020). E-main waste's goal is to pollute the land, where it can become a source of toxic wastes in groundwater and soil, as well as pollutants and heavy metals, if not treated appropriately (Narayanasamy et al., 2021a). As a response, e-waste recovery and recycling are critical in order to meet the demands for raw materials and, more importantly, the issue of harmful contaminants. Several alternative technologies for e-waste recovery and recycling are already available, including hydromentallurgical, pyrometallurgical, and recently updated developing bio-based systems. Bioleaching, bioremediation, bioaccumulation, and other bio-based procedures are recommended by the majority of current e-waste researchers (Narayanasamy et al., 2021a; Wang et al., 2018, 2019).

It has successfully formed microbial consortiums based on each isolate's capacity to grow in crude oil, diesel fuel, and lubricating oil substrates, and tested their ability to breakdown diesel in liquid culture in the past (Banat et al., 2010). Numerous studies have detailed the use of microbial consortia for crude oil degradation and other uses around the world over the years, but studies extracting high-value metals from e-waste using indigenous bacterial consortia from this native isolate region are few and far between Liu et al. (2017), Herrero and Stuckey (2015), Jin et al. (2013). In this regard, the current work focuses on a method for increasing the level of high-value metal extraction from e-waste using native isolates of the *Frankia* consortium.

Bioleaching is a long-term extraction method that employs a collection of microorganisms to transform hazardous substances into less toxic or non-toxic constituent elements while also extracting high-value metals. Features of microbial consortia or bacterial consortia, such as microbial assembly, functional connections, and the involvement of member species, have facilitated this technology's advancement in recent decades when compared to individual microbes. Different researchers recently conducted e-waste studies employing distinct individual microbes to extract precious metals and detoxify toxic metals from e-waste. As a result, recovering high-value metal resources from e-waste is incredibly difficult and a big subject of concern for scientists all around the world. The study's goal was to employ the *Frankia* consortium to recover high-value metals from e-waste in order to achieve this goal.

2. Materials and Methods

2.1. E-waste collection

The e-waste printed circuit boards (PCBs) were gathered from an industrially polluted environment in Hosur, Tamil Nadu, India, which is earmarked to be the 13th Top 20 developing city in the world by 2025, according to a Visual Capitalist report (Latitude: N 12° 76″, Longitude: E 77° 82″). E-waste sample preparation; the obtained PCB board was made as fine particles with a size of (120 m) using the method of (Narayanasamy et al., 2021b). E-waste fine (e-fine particles) was used as a bioleaching innovation along with the *Frankia* consortium.

2.2. Frankia consortium

In the *Frankia* consortium, we've isolated and identified two *Frankia* sp. and two *Frankia casuarinae* isolates so far. For *Frankia* sp. and *Frankia casuarinae*, we used the accession codes MH79558, MH79559, and MH797560, MH797561 in the NCBI database. We keep all of the *Frankia* isolates in a deep freeze in the Bioprocess Technology Lab, Department of Microbiology, Bharathidasan University, Trichy-24, Tamil Nadu, India. The maintained *Frankia* isolates were acquired and subcultured in the standard modified DPM medium (Defined Propionate Minimal Medium) to create the *Frankia* consortium for bioleaching approach, as described by Marappa et al. (2020a).

2.3. Acid digestion removes e-waste plastic and other contaminants

The e-fine particles were acid digested using 18 mL of newly prepared acids in a 1:2 ratio (6 mL HNO3 + 12 mL HCL) to eliminate any undesired plastic or debris. Following that, as detailed by Narayanasamy et al. (2018, 2021b) we used Atomic Absorption Spectroscopy (AAS) for further high-value metal resource discovery (SHIMADZU-AAS, Japan).

2.4. SEM with EDX and XRD investigation of e-fine particles morphology and metal elements composition

The morphology arrangements of e-fine particles were validated using a scanning electron microscope (SEM) (VEGA3 TESCAN Bruker, USA). Similarly, we used Energy Dispersive X-ray (EDX) and X-ray Diffraction (XRD) spectrum analysis (Rigaku Ultima all, Japan) to corroborate the high-value metal element resources composition identified in the e-fine particles. The methodologies of (Marappa et al., 2020b), have already been used to explain the full above process in detail.

2.5. Leaching process for high-value metals

Frankia consortium conducted a high-value metal element leaching experiment using two distinct leaching procedures and varying concentrations of e-fine particles. The *Frankia* consortium inoculums, as well as various concentrations (0.2, 0.4, 0.6, 0.8, and 1.0 percent) of e-fine particles, were tolerated in the modified DPM medium in the one-step procedure. Similarly, the *Frankia* consortium was allowed to develop for 2–3 days before being added the same concentration of e-fine particles for bioleaching operations in the two-step procedure. After that, the *Frankia* consortium treated with e-fine particles in both phases was incubated at 28 °C for 25–30 days for 100% metal leaching. As an upshot, both positive and negative controls were maintained (Narayanasamy et al., 2018, 2021b; Marappa et al., 2020c).

2.6. Explorations of pH and biomass changes during the leaching process

The pH and biomass variations were estimated using the Narayanasamy et al. (2021b) method at every 5 day interval during both steps of the bioleaching process. As a condition, the replication trials for the pH and biomass monitor proposals were also kept. As a response, a digital pH device was used to track the pH variations (Analab Scientific Instruments Pvt. Ltd., India). The digital measurement instrument also estimated wet weight changes in biomass variants throughout the trial.

2.7. AAS evaluations of high-value metal element leaching

Following that, the *Frankia* consortium's leaching of high-value metal elements was discovered utilizing the AAS spectrum (SHIMADZU-AAS, Japan), followed by Narayanasamy et al. (2018, 2021b), (Marappa et al., 2020b) methodologies. Additionally, the above-mentioned articles have already disclosed significant details regarding sample preparation for AAS analysis as well as other vital information.

2.8. SEM-EDX analysis of Frankia consortium interactions with e-fine particles and metals leaching

SEM analysis revealed interactions between the *Frankia* consortium and e-fine particles throughout the leaching process. Similarly, the leaching of high-value metal components was confirmed by EDX (VEGA3 TESCAN Bruker, USA). We previously described the entire process in detail using the methodologies of Narayanasamy et al. (2018, 2021b), and Marappa et al. (2020b).

2.9. Metal leaching and organic acid functional groups XRD and FT-IR spectrum analysis

The XRD spectrum also demonstrated the leaching of high-value metal elements during the bioleaching process (Rigaku Ultima all, Japan). Based on that, the XRD spectrum reveals the two intensities of peaks, and a literature search identified the high-value metal elements. Similar manner, the FT-IR spectrum analysis (Perkin Elmer, Spectrum TWO, Llantrisant, UK) confirms the presence of organic acid functional groups during the leaching process. Authors it has earlier highlighted in detail the entire above system using the methods of Narayanasamy et al. (2018, 2021b), and Marappa et al. (2020a).

3. Results and discussion

3.1. Mechanism of Frankia consortium and e-fine particles leaching

The mechanism of high-value metal element leaching has occurred through both direct and indirect bioleaching methods. The *Frankia* consortium's main role is to extract metal elements from e-fine particles during leaching processes. During biochemical and metabolic interaction activities, *Frankia* consortium along with e-fine particles of raw metal elements capable of transforming their refined forms. As a result, the *Frankia* consortium created by-products of secondary metabolites, phosphatase enzyme, and organic acids, which are primarily recast with high-value metal elements of e-fine particles. The growth of the *Frankia* consortium shows less turbidity at the e-fine particles pulp density of 0.2, 0.4, 0.6, 0.8, and 1.0 percent in the one-step leaching process. It is due to a lack of *Frankia* consortium by-products as well as the toxic effects of e-fine particles. The growth of the *Frankia* consortium in the two-step leaching method demonstrates a

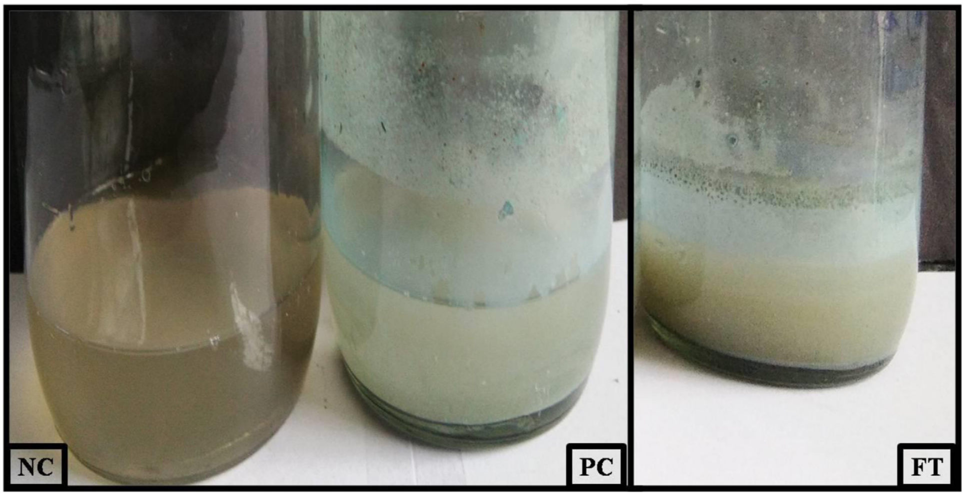

Fig. 1. Shows the interactions between the *Frankia* consortium and e-fine particles, as well as PC-Positive and NC-Negative controls (NC e-fine particles and media without culture) (PC medium with *Frankia* consortium cultures without e-fine particles).

high level of turbidity at above the same pulp density of e-fine particles. The *Frankia* consortium has previously produced by-products capable of tolerating the toxic effects of e-fine particles.

When compared to a one-step leaching process, Marappa et al. (2020c) demonstrate that a two-step gold bioleaching process is highly effective for gold leaching by *Frankia casuarinae* and *Frankia* sp. They conclude that the previously synthesized active metabolites tolerated the toxicity of e-waste and enhanced the random leaching of precious metals. Similarly, the prior e-waste gold bioleaching researchers such as Argumedo-Delira et al. (2019), Arshadi and Mousavi (2015), Narayanasamy et al. (2018), and Liu et al. (2017) have reported one and two-step bacterial growth turbidity formation or precipitation velocity. As a result, two-step gold bioleaching is more effective for bacterial growth turbidity formation than one-step due to the earlier generation of bacterial by-products. The above study was also parallel to the report Narayanasamy et al. (2021b) which described the mechanism of the one-step and two-step processes of e-waste bioremediation by *Frankia casuarinae* and *Frankia* sp. As a result, the current findings were strongly related to the preceding gold leaching storey. The two-step bioleaching process has also been described by Khaing et al. (2019), Li et al. (2015), and Narayanasamy et al. (2018) researchers.

The recent study was significant when compared to previous work because the native isolates of *Frankia* consortium produce a higher level of secondary metabolites and organic acid during the two-step metal extraction process, and there is no more toxic outcome when screening the one-step metal extraction and previously accessible method of specific microorganisms approach in both steps of e-waste bioleaching. When compared to other available bioleaching approaches, the native isolates of the *Frankia* consortium were able to tolerate the maximum level of e-waste toxicity. Correspondingly, the *Frankia* consortium was able to produce more secondary metabolites and organic acids during the two-step metal extraction process than previously available e-waste bioleaching methods. The preceding statements confirm the study's unique approach.

3.2. Confirmation of changes in pH and biomass during metal leaching

The pH and biomass profiles of both controls and e-fine particles treated trials were documented at 5-day intervals throughout the bioleaching process (Fig. 1). The changes in pH and biomass during metal leaching were demonstrated using one-step and two-step pH and biomass profiles. The first day of pH and biomass is exposed very little at all concentrations of e-fine particles treated trials in a single step. The pH decreased as the biomass value increased, depending on the density of the e-fine particles occupied (Fig. 2). When compared to a one-step method, the two-step pH and biomass profiles show the normal level at all concentrations of e-fine particles treated trials. As a result, during the day-to-day leaching process, the biomass was arbitrarily improved, and the pH was arbitrarily reduced, depending on the density of the e-fine particles in use (Fig. 3). The positive control pH and biomass profiles of the first day (pH 8.0, Biomass 0.5 g/L) were subsequently reduced and the biomass concentrations increased on the final day of leaching trials (pH 7.3, Biomass 6.9 g/L). Similarly, the negative control shows that there are no pH changes on the final day of leaching trials because pH changes are caused by the *Frankia* consortium, not an e-fine particle (Fig. 4). Researchers Marappa et al. (2020a), Narayanasamy et al. (2018, 2021b), and Khaing et al. (2019) previously provided explanations for pH and biomass variations during precious metals bioleaching.

As an upshot, they conclude that the two-step method produces more organic acids than the one-step method, which is primarily responsible for the pH level decrease due to the mass level of biomass production. When compared to the

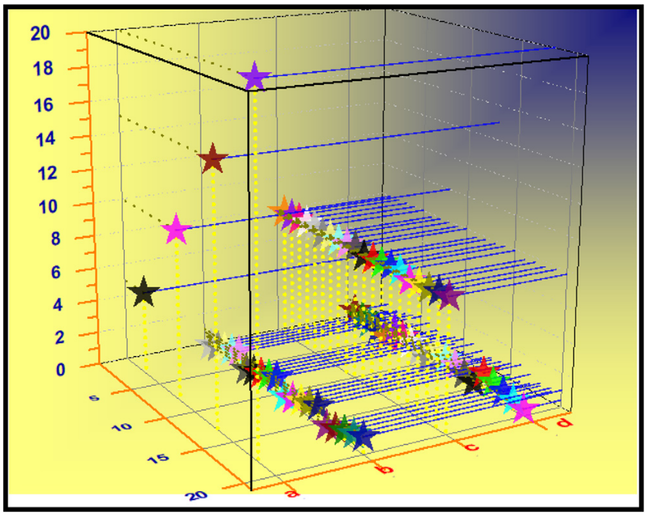

Fig. 2. Depicted a three-dimensional image of the pH and biomass profile of *Frankia* consortium one-step bioleaching. (a) Intervals of a single day. (b). varying concentrations of tiny particles (c). Bioleaching pH fluctuation. (d) Biomass changes as a result of bioleaching.

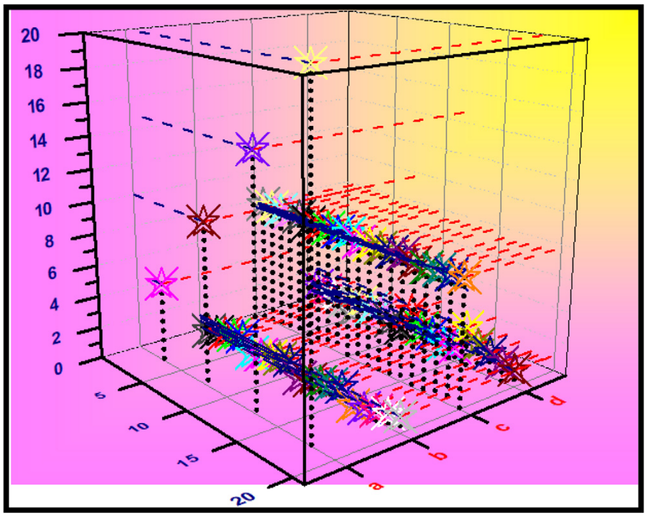

Fig. 3. Showing the *Frankia* consortium two-step bioleaching pH and biomass profile from a three-dimensional perspective. (a) Intervals of a single day. (b). varying concentrations of e-fine particles (c). Bioleaching pH fluctuation. (d) Biomass changes as a result of bioleaching.

above-mentioned gold and precious metal leaching proclamation, the pH of the *Frankia* consortium e-waste bioleaching process was greatly reduced and the biomass value increased, depending on the density of the e-fine particles occupied. As a result of the high level of pH reduction at the same time, the *Frankia* consortium produced more organic acid when compared to the previous specific microbes approach.

3.3. AAS spectrum evidence of high-value metal element leaching

AAS spectrum analysis detected high-value metal elements leaching and control e-fine particles. The unique light absorption capacity of each leaching and control metal revealed the initial and final leaching concentration of high-value metal elements. As a result, the initial concentrations of high-value metal elements are as follows: Au (0.04 mg/g), Ag (0.04 mg/g), Cu (0.12 mg/g), and Zn (0.10 mg/g). Similarly, when compared to control e-fine particles, the two-step metal leaching validates the gradually increased high-value metal element concentration. Similarly, the concentration of high-value metal elements was increased depending on the density of the e-fine particles (from 0.2 to 1.0 percent) and leaching methods. As a result of this, the two-step at 0.2 percent density of the e-fine particles revealed that they are capable of leaching metal concentrations such as Au (0.11 mg/g), Ag (0.09 mg/g), Cu (0.17 mg/g), and Zn (0.15 mg/g) (Fig. 5). Some

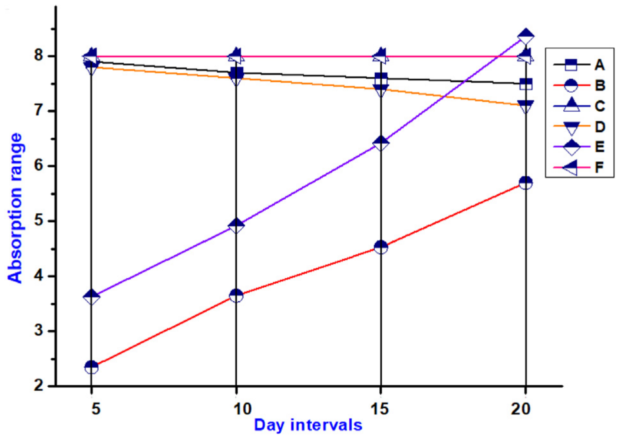

Fig. 4. Positive and negative controls for both steps are depicted in graphs. (A). One pH step with a positive control. (B). Control that is positive One step's biomass. (A, B, C, D, E, G, H, I, J, Both steps have negative pH controls. (In the line, all graphs are identical.) (D). Two-step pH positive control (E). Control that is positive Two-step biomass.

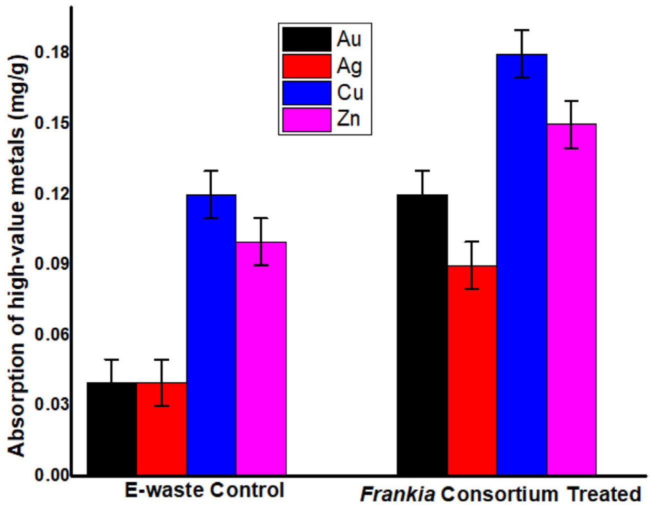

Fig. 5. The graphs depict the leaching of valuable metals by the *Frankia* consortium and the AAS spectrum's control of e-fine particles.

other e-waste researchers, including Sethurajan et al. (2012), Huang et al. (2019), and Narayanasamy et al. (2021b), have already clarified the above parallel reports (Marappa et al., 2020c). They already work with similar bacteria and other bacterial populations, as well as e-waste, and the AAS spectrum confirmed their main focus on precious metals and other trace elements. In comparison to the above-mentioned declaration, the metal leaching of the *Frankia* consortium e-waste bioleaching process was much more efficient, and the biomass value increased depending on the density of the e-fine particles occupied. As a result of the superior level of high-value metal elements leaching, the Frankia consortium leached the high-value metal elements when compared to the previous specific microbe's approach, which was confirmed by AAS spectrum analysis.

3.4. SEM-EDX analysis of bioleached structural and metal element variations

SEM predicted the assorted texture irregular sizes of metal elements in the bioleaching residue. The interaction of the *Frankia* consortium with e-fine particles was revealed by SEM examination in the two-step leaching experiment. Similarly, the *Frankia* consortium's surface and hyphae accumulate solid e-fine particles. Because of the toxicity effect of e-fine particles, SEM microphotographs of *Frankia* consortium morphology appeared wrinkled; it is not a stable structure (Fig. 6). Similarly, the EDX determines the high-value metal elements leaching during the interactions of the *Frankia* consortium and e-fine particles. As a result, the highest concentrations of high-value metal elements are Au (0.04 mg/g), Ag (0.04 mg/g), Cu (0.12 mg/g), and Zn (0.09 mg/g). Accordingly, when compared to control e-fine particles, two-step

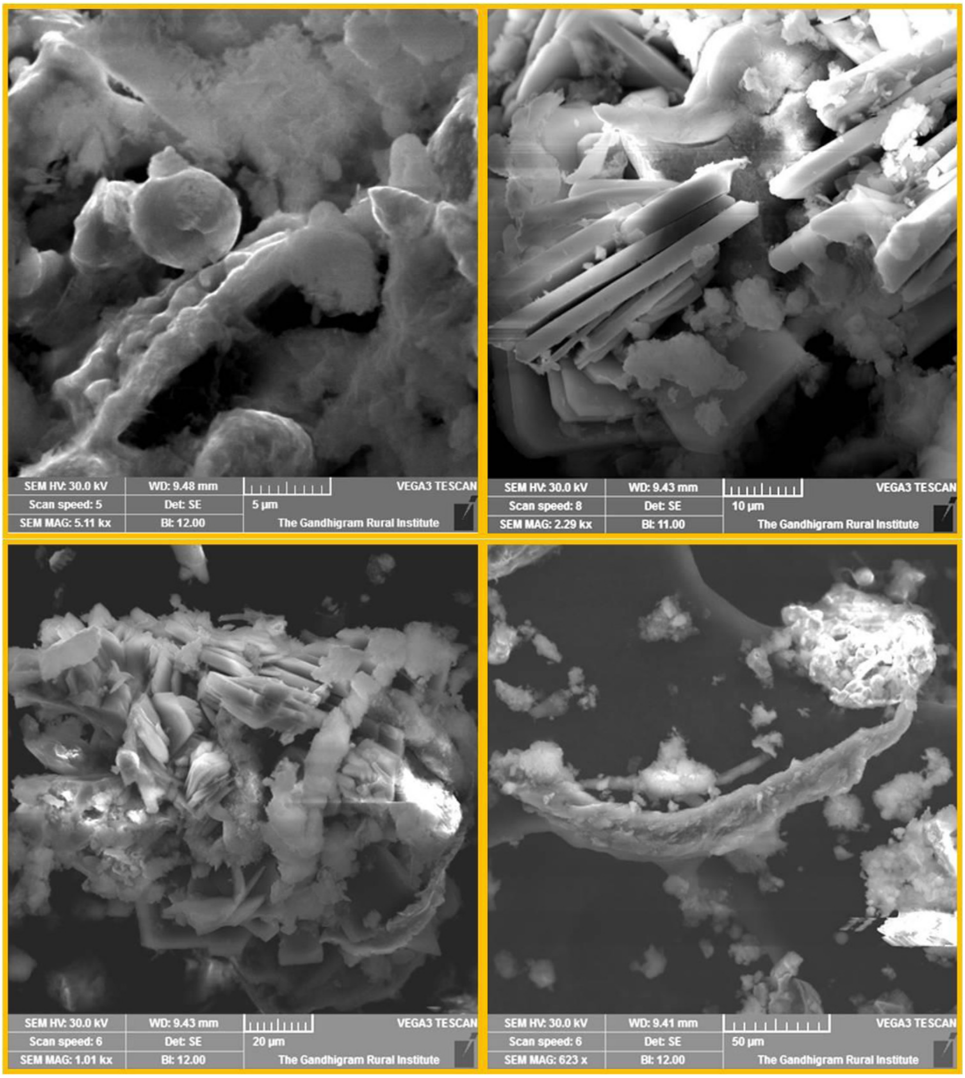

Fig. 6. Microphotographs of *Frankia* consortium and e-fine particle interactions exposed by SEM at various magnifications (5, 10, 20, 50 m).

metal leaching allows for increasingly increased high-value metal element concentration. Similarly, the concentration of high-value metal elements increased depending on the solidity of the e-fine particles (from 0.2 to 1.0 percent) and leaching methods. As a result, the e-fine particles exposed to the two-step at 0.2 percent density were capable of leaching metal concentrations such as Au (0.13 mg/g), Ag (0.08 mg/g), Cu (0.19 mg/g), and Zn (0.13 mg/g) correspondingly (Fig. 7, 8). The findings presented above were consistent with those of other researchers working with various microbes. Bajestani et al. (2014) use SEM interpretation to clarify the microbe metal interaction during metal leaching by *Acidithiobacillus ferrooxidans*. Hu et al. (2021) used SEM analysis to shed light on the interactions between bacteria biofilm and e-waste metals during metal extractions. Similarly, Santhiya and Ting (2005) and Narayanasamy et al. (2018) provided explanations for *Aspergillus niger* precious metal leaching from e-waste using SEM-EDX exploration, which was strongly interrelated to the prior ending. In contrast to the above mentioned SEM interpretation of the e-waste treatment declaration, the *Frankia* consortium strongly interacted with e-waste high-value metal elements, as confirmed by SEM interpretation. Similarly, the EDX confirms the presence of significant metal leaching (see Fig. 8).

3.5. XRD spectrum evidence of high-value metal element leaching

XRD spectrum exploration revealed high-value metal elements leaching and control e-fine particles. The microstructural view of e-fine particle crystallinity revealed by XRD demonstrates the collaboration of anode and cathode catalysts 2 peaks. As an upshot, the discovery reveals catalysts 2 peaks values (44), (21), (32), (38) and their subsequent intensity

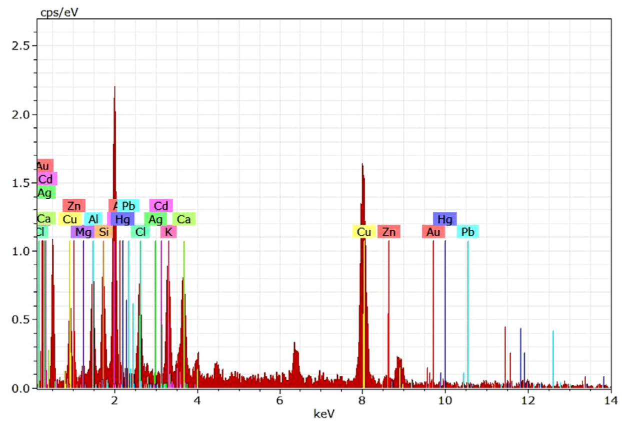

Fig. 7. EDX reveals high-value metal elements leached by the *Frankia* consortium and controls e-fine particles.

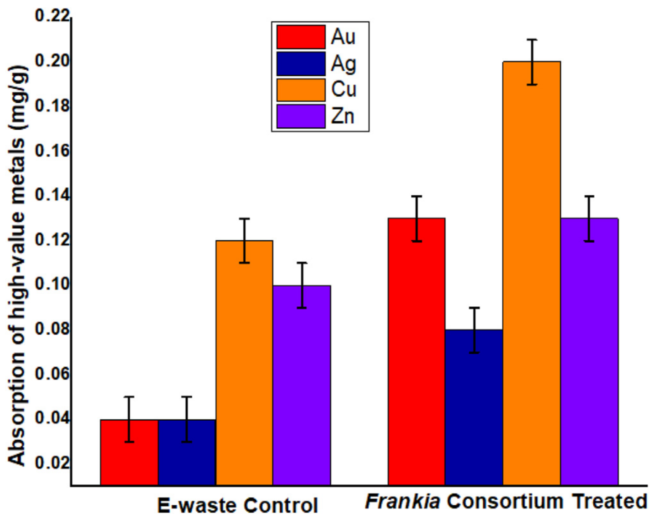

Fig. 8. EDX demonstrates that the *Frankia* consortium leaches high-value metal elements when compared to control e-fine particles.

values (1600), (14000), (1800), (1900), and (1300), respectively (Fig. 9). Correspondingly, the previous discovery reveals the presence of high-value metal elements such as Au, Ag, Cu, and Zn during the metal leaching process, as well as control e-fine particles. As a consequence, the XRD provides evidence for two-step metal leaching by gradually increasing the concentration of high-value metal elements when compared to control e-fine particles. Analogously, the concentration of high-value metal elements increased with particle density (from 0.2 to 1.0 percent) and leaching methods.

Heretofore, researchers Sannigrahi and Suthindhiran (2019), Shah et al. (2015), and Yuan et al. (2018) specified the evidence for metals detection by XRD using both untreated e-fine particles and two-step leaching metal elements. Similarly, Narayanasamy et al. (2018, 2021b), and Marappa et al. (2020a) used an XRD spectrum to discuss the anode and cathode catalysts' two peaks, as well as their intensity values of precious and toxic metals from e-waste. In comparison to the previous researcher's report, the *Frankia* consortium's XRD interpretation supports the presence of high concentrations of precious and toxic metals in e-waste.

3.6. FT-IR spectrum evidence for organic acid functional groups

The existence of some transmittance bands connected to distinct organic acid functional groups can be seen in the infrared spectrum. Correspondingly, the IR spectrum of the two-step leaching residue confirms the presence of a range of organic acid functional groups. As a result, the transmittance bands at 1010.80, 533.05, 561.09, 457.61, 509.95, and 478.93 cm^1, which were associated to functional groups of C-O, C–C-N, C–C, and RCO-OH, respectively, were found to be interrelated (Fig. 10). Alike, the significance was to determine the presence of various organic acids such as citric, oxalic, and gluconic acid during the two-step technique of metal leaching. The generation of organic acids during the

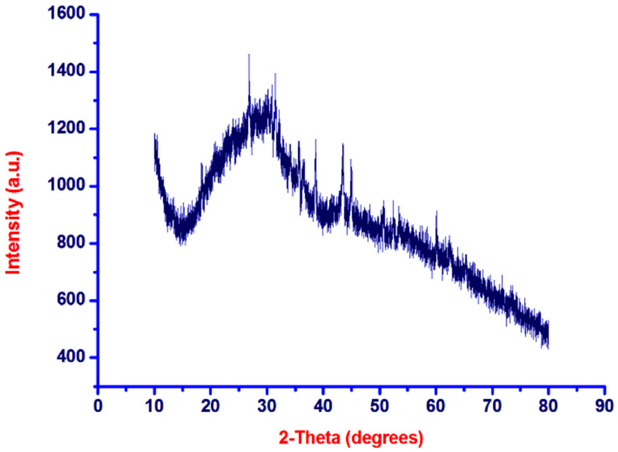

Fig. 9. During the leaching process by *Frankia* consortium and control e-fine particles, XRD revealed the existence of high-value metal elements 2-theta intensity picks.

Fig. 10. IR spectra confirming the presence of organic acid functional groups during the *Frankia* consortium's e-fine particle leaching process.

metal leaching process was a likely cause of the pH drop. IR spectrum exposure during the e-waste bioleaching process was used by Wang et al. (2018, 2019) to describe the existence of organic acid functional groups. Likewise, they are indicating the presence of aromatic metabolic substances such as lipids, proteins, carbohydrates, and nucleic acids, which are very comparable to the above-mentioned recent findings. Identical, Narayanasamy et al. (2018, 2021b), and Marappa et al. (2020b) discussed organic acid, which was principally trustworthy for fungal and bacterial bioleaching from e-waste precious metals. In compared to prior researcher's report, the *Frankia* consortium's IR spectrum exposure interpretation supports the presence of high concentrations of various organic acids functional groups such as citric, oxalic, and gluconic acid, which were primarily responsible for the high-value metal element leaching from e-waste.

4. Conclusion

The AAS spectrum was used to determine the initial concentration of e-fine particle metal elements and the escalating concentration of bioleaching high-value metal elements, according to the results. The SEM was used to verify the morphological configurations of e-fine particles that facilitated for *Frankia* consortium interactions. As an upshot, EDX and XRD spectrum analysis validated the initial concentration of e-fine particles and the escalating concentration of bioleaching high-value metal components. In comparison to control, the high-value metal gold (Au) is leached out by *Frankia* consortium at a rate of almost 90%, followed by silver (Ag) at around 75%. As an end result, when measured with other high-value metals, gold (Au) and silver (Ag) were heavily leached out by the *Frankia* consortium. The *Frankia* consortium's gathered products of high-value metal elements revival from e-fine particles of e-waste establish a potential to drain our materials valuable to mankind. It also helps to control e-waste contamination in the environment by recovering and reusing PCBs. The *Frankia* consortium's findings of high-value metal elements recovery from e-fine particles of e-waste are the unique feature of our technique, according to the current study. Since a result, the major findings were advised, as *Frankia* consortium bioleaching is a booming technique in favors of e-waste recovery, and recycling is a cost-effective and environmentally beneficial strategy.

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.

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