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Reprocess of Copper from Worn Printed Circuit Boards

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Abstract: The objective of the study is to reuse printed circuit boards which are mainly made of copper. Most top down and bottom up approaches for the synthesis of Copper nanoparticles (CuNPs) needs pure copper (II) sulfate or any other copper salts as a raw material. For the expensive non-ecofriendly reasons, here we used wasted Printed Circuit Boards (PCBs) as an alternative source of copper and *Ocimum Sanctum* leaf extract was used as a redox agent. Equally distributed, spherically shaped CuNPs around 14.6-19.7nm size were obtained by this process. Further characterization of synthesized nanoparticles was done with UV-Vis spectrophotometer, AFM, FT-IR, SEM and EDAX. Essential physical parameters like pH, Temperature, Reaction Speed were examined for the CuNPs synthesis optimization process. These CuNPs were reused for assessing dye decolorisation promisingly 70% of dye got decolorized. Based on this study, we conclude that wasted PCBs can be an alternative raw source for CuNPs synthesis and the synthesized nanoparticles can be used for environmental pollution control.

Keywords: Copper, E-Waste, Leaf Extract, Nanoparticles, Bioleaching

Introduction

Electronic waste popularly known as ‘E-waste’ broadly comprises of Waste from Electrical and Electronic Equipment (WEEE). E-waste is being generated two to three times faster than other waste streams like solid municipal wastes, hazardous wastes etc., (Grossman, 2010). Recycling of E-waste is one of the important processes which involve the conversion of used electronic devices to usable raw materials. Every year, millions of tons of E-waste get distributed from countries like U.S and Australia to developing countries like India and China, where it is dumped at landfills, which can cause pollution to the environment owing to their toxic nature (Baldé *et al*., 2015). Printed Circuit Boards (PCBs) represent 3% of the mass of global WEEE generated (Dalrymple *et al*., 2007). Waste PCB contains nearly 28% metals and the purity of metals is more than 10 times higher than that of rich-content minerals (Li *et al*., 2007). Due to significant risks to human health and the environment, recovering waste PCBs has now become increasingly important, which is mostly carried out in poor working conditions using crude technologies. Serious pollution has been generated in the recovering process such as exposure to polybrominateddiphenyl ethers, dibenzofurans and polychlorinated dibenzo-dioxins (Duan *et al*., 2011). With so many physical and chemical methods are available for recovering waste PCBs, precious metals were lost by both these recovering processes (Li and Xu, 2010; Ruan *et al*., 2013). Therefore, environment-friendly technology is immediately required for improving the additional value of waste PCBs recovery. On the other hand, copper nanoparticles have gained unique physical and chemical properties and synthesis of CuNPs is less expensive and has created great interest in medical research recently. Copper nanoparticles have shown wide applications in the field of medical and industrial use such as gas sensors, catalytic processes, solar cells etc., (Li *et al*., 2007). At the present time biological approaches using microorganisms and plant extracts for the synthesis of metal nanoparticles have been recommended as appreciated replacement to traditional synthetic methods (Krumov *et al*., 2009; Mishra and Rhee, 2010; Zare *et al*., 2017).

Science Publications

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The aim of the present study is to lay on bioleaching as an ecologically safe process to remove heavy metals from e-waste, and to synthesize copper nanoparticles from worn out mobile phone printed circuit boards using the plant extract *Ocimum sanctum* (Tulsi). Additionally, the study focuses on the characterization of the nanoparticles and the dye discoloration activity of synthesized copper nanoparticles.

About the Plant

Ocimum sanctum (Tulsi) is considered as Holy basil in India. It is a traditional medicinal plant and belongs to the family Lamiaceae. *Ocimum sanctum* remains as an active area of research, and recent innovation on *Ocimum sanctum* has reported the inhibitory activity against HIV-1 reverse transcriptase (Anuya *et al*., 2010) also it has been widely acknowledged for the treatment of headaches, coughs, diarrhea, constipation, warts, worms, kidney malfunctions etc., (Simon *et al*., 1999). Recently *O. sanctum* leaf extracts (Fig. 1) have been used in the synthesis of different nanoparticles like iron oxide, silver (Mallikarjunaa *et al*., 2011; Singhal *et al*., 2011), platinum (Soundarrajan *et al*., 2012) and gold from readily available chemicals. But to the best of our knowledge, the use of *Ocimum sanctum* leaf extract for the biosynthesis of copper nanoparticles using e-waste has not yet been reported. Hence, the present study is dealt with the biosynthesis of copper nanoparticles using *Ocimum sanctum* leaf extract from worn Printed Circuit Boards (PCB).

Materials and Methods

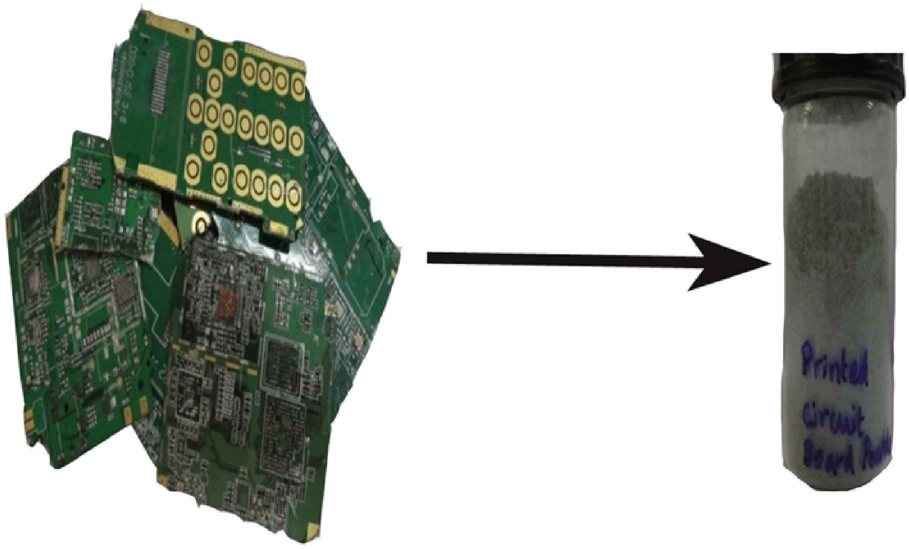
Printed Circuit Boards (PCB) Collection and Copper Content Analysis

A modified protocol of Sheng and Estell (2007) was followed for PCB collections where in discarded mobile phone circuit boards were collected from mobile phone service centers. The mobile phone circuit board was cut into pieces and ground to a minute powder using the mechanical grinder. The PCB powder was sieved, autoclaved and used for further experiments (Fig. 2a and b). About 100 mL of aqua regia was added to 1g of PCB powder and refluxed in a round bottomed flask at 100°C for 1 hour using heating mantle. The solution was cooled and the volume was made up to 100 mL using distilled water. The solution was filtered and stored at 4°C for further analysis.

Preparation of Ocimum sanctum Extract

About 10 g of fresh *Ocimum sanctum* leaves was surface sterilized, finely chopped and mixed with 50 mL of distilled water. The mixture was stirred at 60°C for 1 hour, cooled and then filtered through a 0.45 μm membrane filter and stored at 4°C for further experiments.

Fig. 1: *Ocimum sanctum* plant



(a) (b)

Fig. 2: (a) Mobile phone PCBs (b) Powdered PCBs

Bioleaching Process

In the bioleaching process 1g of autoclaved Printed Circuit Board (PCB) powder and 1mL of *O. sanctum* extract was added to the flask containing 99 mL of sterile distilled water and incubated in an incubator shaker at 37°C for visible color change. The bioleaching of copper ions was monitored by periodic sampling of aliquots of the suspension and measured using UV-Vis spectroscopy.

Characterization of the Synthesized Copper Nanoparticles

Optical absorption measurements of nanoparticles have been carried out by SPECORD 210 PLUS UV-Vis spectrophotometer. The morphology of the synthesized nanoparticles was studied using Atomic Force Microscope (NANOSURF EASY SCAN 2) and Scanning Electron Microscope (SEM- EDAX FEI QUANTA 200). The FT-IR (NICOLET 380), analysis have been used to receive information about the element to element bounding or possible functional groups attached to the nanoparticles that are responsible for capping and efficient stabilization of the synthesized copper nanoparticles. The elemental analysis or the chemical characterization of the synthesized nanoparticles was determined by Energy-dispersive X-ray spectroscopy.

Effect of Physical Parameters on the Synthesis of Copper Nanoparticles

The essential factors like solution pH, temperature, reaction mixing speed, concentration of the extracts used etc., are the altering factors for nanoparticles synthesis. Meager changes in the above mentioned parameters will affect the nanoparticle size, shape and synthesis. In our study, we have taken three important parameters (pH, Temperature, Reaction Speed) for the optimization process. Different mixing speed range from 100 rpm to 1000 rpm, reaction mixture pH ranging from pH 2 to pH 12 and reaction temperatures ranging from 40°C to 100°C were examined to observe the optimum conditions for CuNPs synthesis. All experiments were performed in triplicates and for each parameter, respective controls were maintained.

Dye Discoloration Activity of Synthesized Copper Nanoparticles

The discoloration of Methyl Orange (MO) in the absence and presence of CuNPs was studied spectrotometrically by using SPECORD 210 PLUS UV-Vis spectrophotometer determining the decrease in the absorbance at 464nm. To a mixture containing 300μL of MO (30μg) 1mL of CuNPs containing 50μg was added and made up to 3mL with distilled water. The 5 discoloration reaction was studied spectrophotometrically at room temperature after two to three hours.

Results and Discussion

Determination of Copper in Mobile Phone Printed Circuit Board (PCB)

PCBs from personal computers and mobile phones contain the highest amounts of valuable metals (Cui and Zhang, 2008). Previous studies reported that the amount of copper present in the PCBs ranges from 12% to 35% which were determined by Atomic Absorption Spectroscopy (AAS) (Chehade *et al*., 2012; Willner *et al*., 2013). This difference in the concentration of copper may also be attributed to the analytical methods (Pradhan and Kumar, 2012). In the present study chemical analysis of ground powder of mobile phone printed circuit boards was carried out to determine the concentration of copper present in it. 76.6% of Copper was present in the ground powder of mobile phone printed circuit board which was also confirmed by AAS. Based on the previous findings, mineral content and recovery percentage varies based on the types of PCBs that we use for our studies (Xiang *et al*., 2010; Gu *et al*., 2017). In our study, we have got 76.6±9.5 of copper from mg/g PCBs and recovered 68.6% copper with *O. Sanctum* leaf extract bioleaching process that was conformed with the help of AAS.

Synthesis of Copper Nanoparticles from Mobile Phone Printed Circuit Board

Visual Inspection

The synthesis of nanoparticles is preliminarily confirmed by the visual color change due to the excitation of the surface plasmon resonance. Subhankari and Nayak (2013) reported that the synthesis of copper nano particles by the aqueous extract of *Syzygiumaro maticum* is indicated by the color change from dark brown to sea green after the addition of copper sulphate solution to the aqueous extract. A similar color change has been reported by other workers by the addition of 5 mm copper sulphate solution to the *Morganella* sp. culture led to the appearance of a dark green color solution indicating the formation of nanoparticles (Ramanathan *et al*., 2013). Electroplating industry wastewater when treated with *Pseudomonas stutzeri* biomass turned red due to the surface Plasmon resonance of the copper nanoparticles formed (Varshney *et al*., 2011). In the same way the visual color change was observed from the aqueous extract of *Ocimum sanctum* showed the color variation from yellow to brown after the addition of PCB (Fig 3a and b). The formation of copper nanoparticles is primarily confirmed by the visible color change from yellow to brown.

Characterization of Synthesized Nanoparticles

UV – Vis Spectroscopy

Next level of confirmation was determined by UV-Vis spectroscopy, it is used to study the size and shape of nanoparticles, the plasmon peak positions and shapes are sensitive to particle size in aqueous suspensions. The absorption peaks arise from the localized Surface Plasmon Resonance (SPR), which is predicted by the well-known resonance condition. The exact position of the SPR band may shift depending on the individual particle properties including shape, size, and capping agents (Galkowski *et al*., 2007). The UV-Vis spectra of reaction solution containing the PCB waste treated with aqueous extract of *Ocimum sanctum* shows the broad peak from 350-450 nm with the λmax at 390 nm as indicated in Fig. 4. A red shift in the wavelength from 325 to 450 nm was observed with the increase in amount of precursor. The shift is due to the generation of greater amount $Cu2^{+}$ ions, which increases the nucleation rate and also indicates the generation of smaller nanoparticle in the solution. Based on our work, UV- plasmon peak was seen around wave length of 390 nm, which indicates the presence of small separate CuNPs average particle size less than 20 nm. This results specifies the high concentration of capping agents and then smaller CuNPs (Xiong *et al*., 2011).

(a) (b)

Fig. 3: Aqueous extract of *Ocimum sanctum* without Printed Circuit Board (PCB) waste (b) Aqueous extract of *Ocimum sanctum* after incubation for 24 h with PCB waste

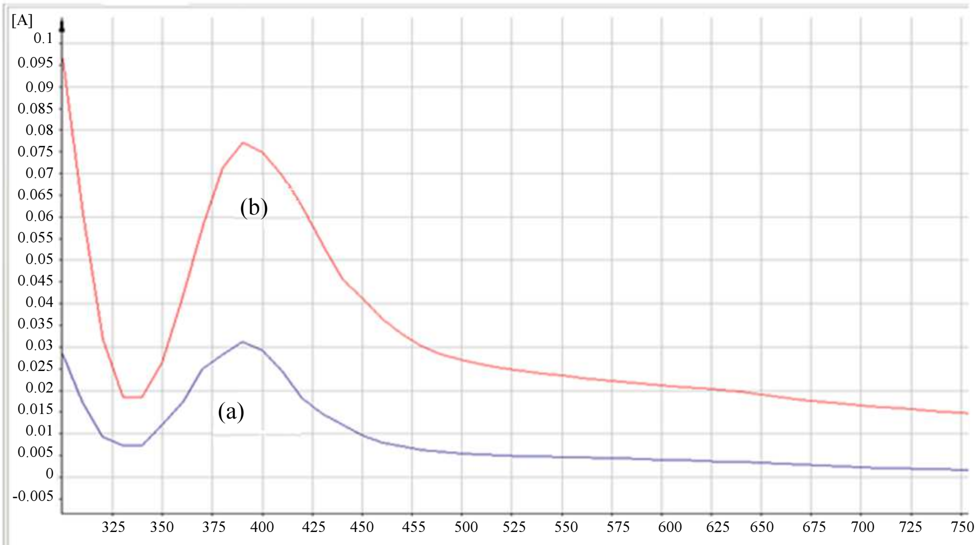


Fig. 4: UV-Vis spectrum of aqueous extract of *O. sanctum* (a) Aqueous extract incubated with Printed Circuit Board waste (b) Aqueous extract incubated with standard Copper solution

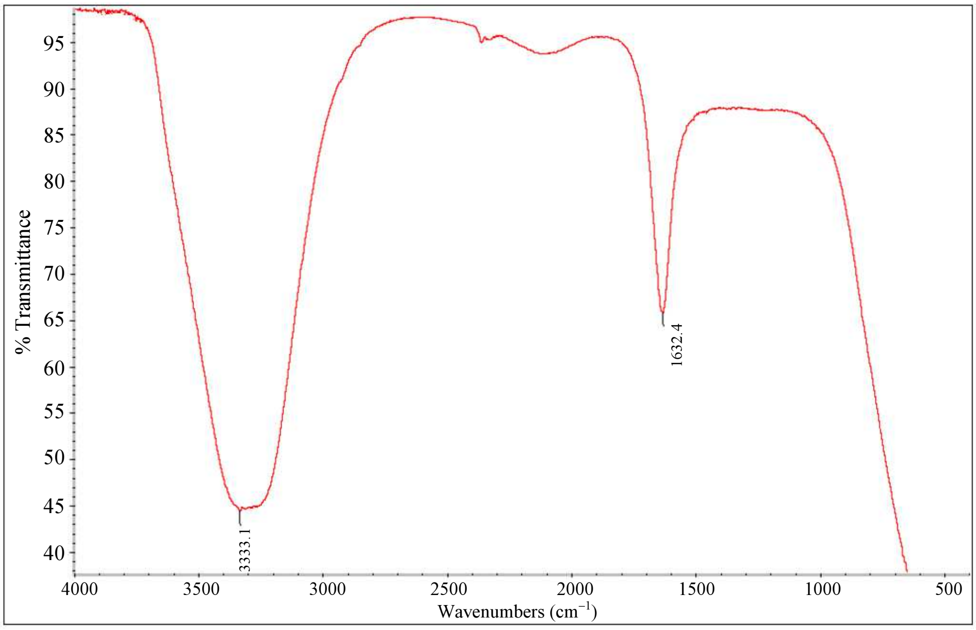


Fig. 5: FT-IR spectrum of copper nanoparticles synthesised using *O. sanctum* aqueous extract

Fourier Transform Infrared Spectroscopy

FT-IR spectroscopy is conducted to identify the possible interactions between reducing agents and the functional groups present on nanoparticles. The size of peak in the spectrum is directionally proportional to the number of functional groups that present in the nanomaterial (Chauhan *et al*., 2012). Peaks were located at about 1632.4 and 3333.6 cm^{-1} for copper nanoparticles synthesized from *O. sanctum* (Fig. 5). The peak at 1634.7 characterize the bending vibrations of O- H bonds in OH groups and the peak at 3333.6 characterize the stretching vibrations of O-H bonds in H_2O molecules. Similar peaks have been reported by (Petrov *et al*., 2012; Majumder, 2013) for copper nanoparticles. The peaks 1634.7 is close to that reported for native proteins (Macdonald and Smith, 1996) which suggests that proteins are interacting with synthesized nanoparticles

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and also their secondary structure were not affected during reaction with copper ions or after binding with copper nanoparticles (Fayaz *et al*., 2010).

Atomic Force Microscopy

Atomic Force Microscopy (AFM) is primarily technique for studying the shape, size 2, 3- Dimensional views and height distribution of nanoparticles. Previous finding explain that the copper oxide nanoparticle have a notable propensity to form uniform size and shaped 7 agglomerates at less concentration because they are very stable molecules, air and water and did not convert nanoparticles into any other associated compounds (Fan *et al*., 2003). Honary *et al*. (2012; Sampath *et al*., 2014) also studied the synthesis of copper nanoparticles by *Penicillium spp.,* wherein green chemical reduction method was followed and observed spherical, Jasmin bud-like copper nanoparticles respectively. Ansilin *et al*. (2016) reported that the size of CuNPs are found in different sizes range between 49-324 nm using *Azadirachta indica* (neem) leaf aqueous extract. In our study, Fig. 6 displays the three dimensional AFM image of synthesized copper nanoparticles by PCB waste using *O. sanctum* leaf extract. Most of the copper oxide nanoparticles were found to be spherical in shape with smooth surface and compact structure. Based on the observation, current study results imply that the synthesized CuNPs are equally distributed with spherical in shape.

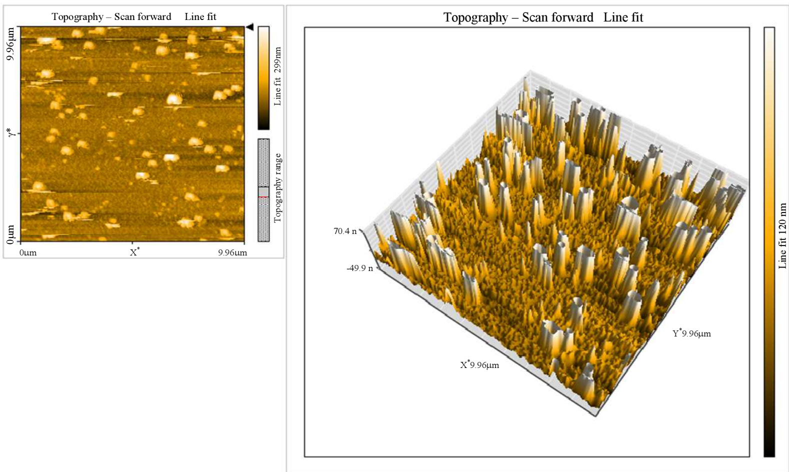

Fig. 6: AFM image of copper nanoparticles synthesised from PCB waste using *O. sanctum*

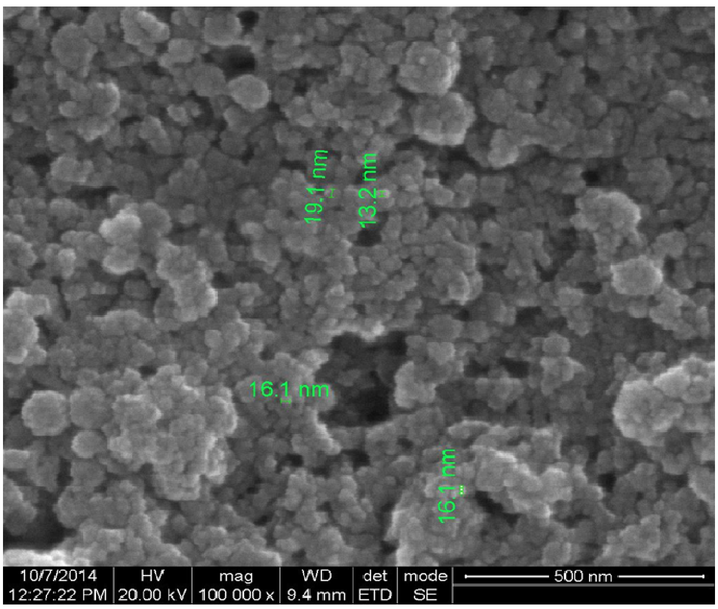

Fig. 7: Scanning electron microscopic image of synthesized copper nonparticles

Scanning Electron Microscopy and Energy Dispersive Spectroscopy (SEM-EDAX) Analysis

SEM is another visualizing technique used to observe the morphology of synthesized biomolecules. SEM produce 2D images based on two electron scattering (Gupta and Kant, 2013). Figure 7 illustration the copper nanoparticles synthesized by the plant extract *O. sanctum.* Copper nanoparticles found to be spherical and relatively uniform shape of the copper nanoparticles was confirmed in the range of 14.6-19.7nm. The average particle size of the Cu nanoparticles is around 17 nm. Energy Dispersive Analysis of X-Rays (EDAX) provides data on the surface atomic distribution and chemical composition of the synthesized product. EDAX analysis of the synthesized product from PCB waste using *O. sanctum* shows elemental signals of copper at 1.00, 8.00 and 9.00 keV (Fig. 8). Synthesized CuNPs showed strong copper signals in the EDAX image along with P and C peaks, which may indicates the biomolecules that were attach to the surface of CuNPs (Chung *et al*., 2017).

Effect of pH on Copper Nanoparticle Synthesis

Copper reduction reaction depends on the various factors like aqueous media pH, Particle size, shape, type of solvent etc., (Zhang *et al*., 2009) (Fig. 9) shows the UV–vis absorption spectra of effect of pH on nanoparticle formation for the pH ranging from 2 to 12. In which Plasmon resonance was not clearly visible at pH2. Different peak values were observed at 382,380,379 and 378 nm for pH 2 to 12. Synthesis of copper nanoparticles using *Ocimum sanctum* extract was pragmatic only after pH 4 with the peak at 382 nm.

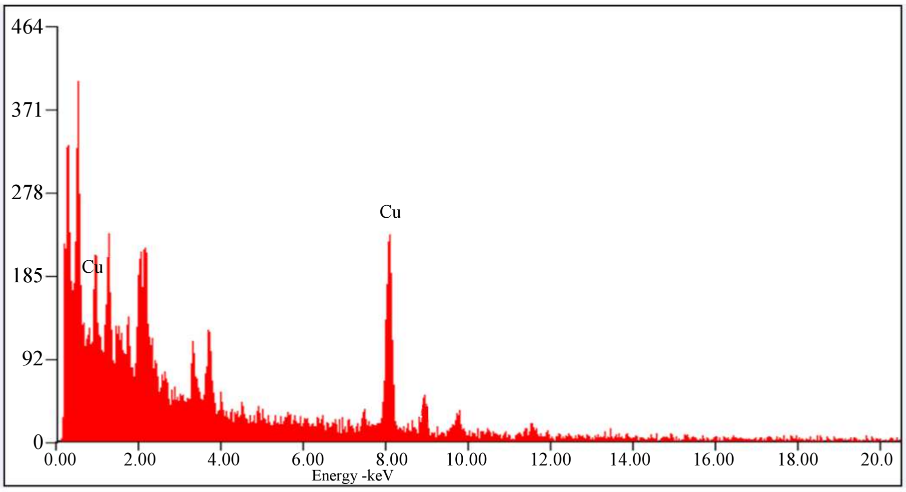

Fig. 8: EDAX spectrum of nonparticles synthesised form PCB waste using *O. anctum* aqueous extract

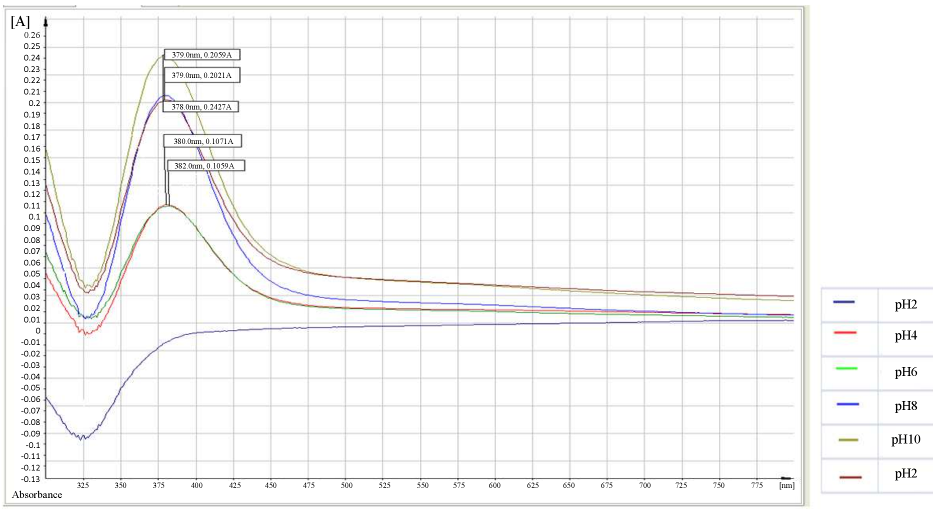

Fig. 9: UV-Vis spectra showing the effect of pH on the synthesised of copper nonparticles form PCB waste using aqueous extract of *O. anctum*

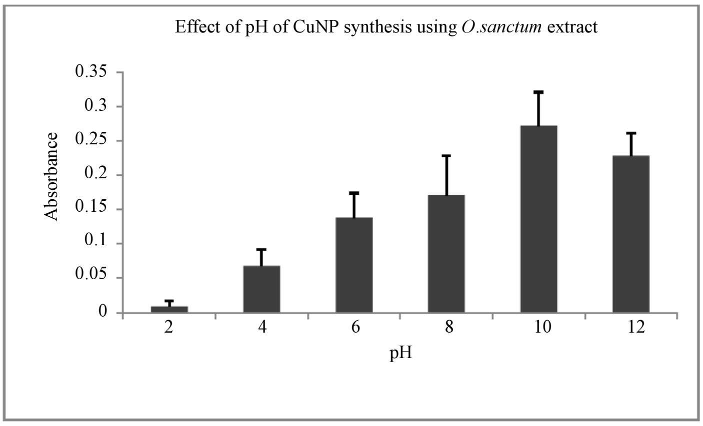

Fig. 10: Effect of pH on the synthesised of copper nonparticles form PCB waste using aqueous extract of *O. anctum*

While increasing the pH there was increase in nanoparticle synthesis up to pH 10. At pH 12, the intensity of peak decreased around the maximum pH indicating the decrease in particle synthesis. According to the literature, the alkali concentration and the pH value should play a vital role in controlling the size distribution of finally synthesized nanoparticles (Ji *et al*., 2007) (Fig. 10) represents the graphical representation of mean absorbance of triplicates measured with different pH range.

Effect of Temperature

The variations in the reaction temperature will unquestionably influence the morphology and structure of nanomaterials. In this cause, temperature of a solution is a major dependable factor. The processes nanoparticles synthesis needs to lower their Gibbs free energy for their nucleation and the growth of particles formation events. This can be achieved with the help of diluting or reducing solution supersaturation because absolutely a high supersaturated solution has high Gibbs free energy (Gaber *et al*., 2014). For nanoparticle synthesis by physical methods need more or less than 350°C but green synthesis or natural methods requires less than 100°C (Patra and Baek, 2014). A range between 40 to 100°C temperatures were used for copper nanoparticles synthesis using *O. sanctum* leaf extract. Figure 11 indicates that increasing reaction temperature induces the production of copper nanoparticle synthesis by this method. Up to 80°C the production rate shows gradual increase after that it shows a decline state. Generally, for Gibbs free energy change, low supersaturation, nucleation and high growth rates are the responsible factors for green nanoparticle synthesis. This was obtained in this method at 80°C, which means that the optimum temperature for copper nanoparticle synthesis by this method is less than 90°C.

Effect of Speed

Increasing the reaction speed leads to the more energy release and rapid monomodal dispersion of nanoparticles occur in the polymeric organic phase. Copper nanoparticle prepared at lower homogenization speeds (100, 250, 500, 750, 1000 rpm) resulted in less absorbance Fig. 12 demonstrate that the gradual increase in reaction speed will be an inducing factor for green particles synthesis process. A recent study results reported that the encapsulation efficiency increased when the speed of emulsification was increased from 800 rpm to10, 000 rpm respectively. Due to the high degree of agitation, anomalous diffusion of particles can be a reducing factor for nanoparticle production (Sharma *et al*., 2016). In our study we observed the nanoparticle production rate alone. Based on our results 750-1000 rpm can be an optimum reaction speed for green nanoparticle synthesis process.

Dye Discoloration Activity of the Synthesized Copper Nanoparticles

In the dye discoloration activity, the color of the reaction mixture started to fade immediately after an hour indicating the degradation of methyl orange (Fig. 13). After 24 h, 68.4% of the dye have been decolorized by the copper nanoparticles synthesized by aqueous extract of *O. sanctum*. The absorption of the visible band in UV-Spec at 464 nm decreased rapidly, which indications the aromatic fragment degradation in the dye molecule and its intermediates (Ince and Tezcanli, 2001) and reflects the discoloration effects of the synthesized nanoparticles (Li *et al*., 2015). Green synthesized CuNPs can act as catalyst and leaves extract has no role in the degradation of MO (Devi and Singh, 2014). It was thus observed that copper nanoparticles synthesized by using natural, renewable and eco-friendly, reducing agents exhibit discoloration activity against dye molecules and can be used in dye effluent treatment.

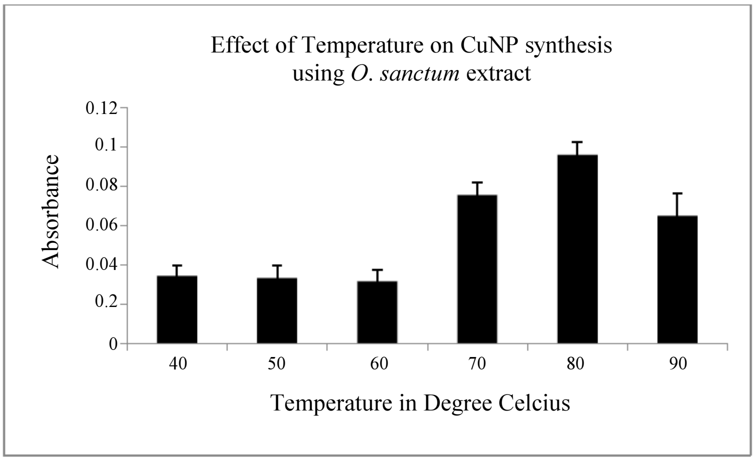

Fig. 11: Effect of temperature on the synthesise of copper nonparticles form PCB waste using aqueous extract of *O. anctum* (Values represent average of triplicates; bars indicate standard error)

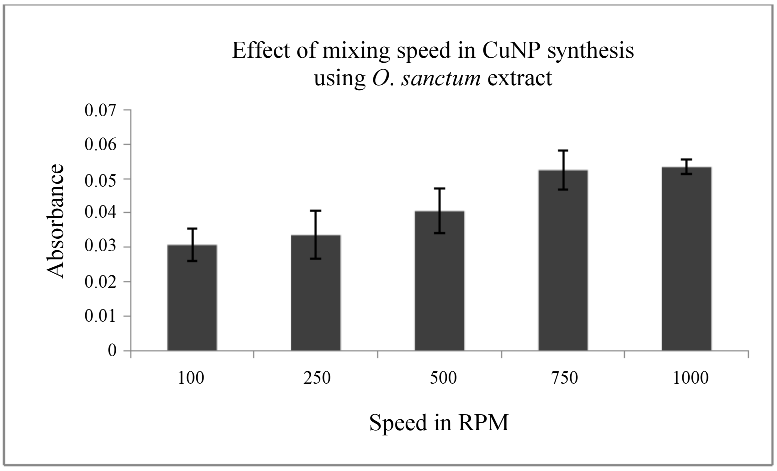

Fig. 12: Effect of reaction mixing speed on the synthesise of copper nonparticles form PCB waste using aqueous extract of *O. anctum* (Values represent average of triplicates; bars indicate standard error)

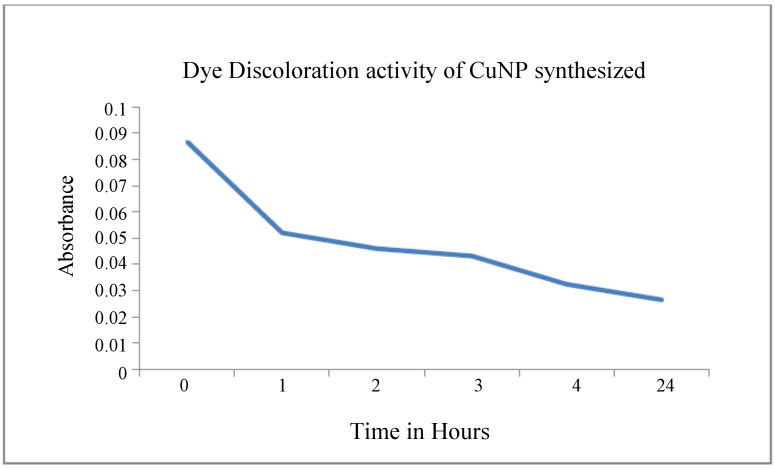

Fig. 13: Dye discoloration activity of CuNP

Conclusion

From these results we conclude that, by using E-Waste and green chemistry, the copper nanoparticles have been prepared using *Ocimum Sanctum* leaf extract. The synthesized copper nanoparticles was able to decolorize synthetic dyes. Our preliminary results suggest that copper nanoparticles can be an alternative economical source for industrial effluent treatments. Due to its good stability, copper nanoparticles could also be effectively used in various other fields like drug delivery, antimicrobials, wound healing as well as on industrial applications. Thus, we conclude that this protocol could be used in other heavy metal bioleaching process. Since it is cost effective and eco-friendly approach for the recovery of valuable metals and an alternative to the chemical synthesis protocol.

Acknowledgement

We greatly acknowledge the host institutions for providing us the space and basic chemicals.

Author's Contributions

All authors contributed equally in thepreparation and development of thismanuscript

Conflict of Interest

The authors declare that they have no conflict of interest.

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