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Green Synthesis of Zinc Oxide nanoparticles using *Cocos nucifera* water: Characterization and study of its organic dye removal capacity by adsorption.

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ABSTRACT

Zinc Oxide (ZnO) nanoparticles are drawing attention of researchers because of its wide applications in medicine, food, agriculture, cosmetics, pharmaceutical, textile, rubber and electronic industries. Current research represents green synthesis of ZnO nanoparticles using *Cocos nucifera* water and its detailed characterization with respect to UV-Visible spectroscopy, X-ray Diffraction studies and Field Emission Scanning Electron Microscopy. The UV-Visible spectra of synthesized ZnO nanoparticles revealed the absorption maxima at a range between 340 to 380 nm which confirms the synthesis of ZnO nanoparticles. The XRD pattern of ZnO NPs shows sharp peaks which indicate crystallinity of ZnO NPs. The Surface morphology of the synthesized ZnO NPs was examined through FESEM analysis showed ZnO nanoparticles with both round and hexagonal shapes. These nanoparticles have size ranged from 22.97 to 61.18 nm. Adsorptive activity of ZnO nanoparticles with respect to organic cationic dye Malachite Green was observed after 31 hrs incubation as evidenced by decrease in the absorbance indicating their potential for efficient dye removal in wastewater treatment process.

Keywords: Green Synthesis, ZnO nanoparticles, *Cocos nucifera* water, FESEM analysis, Adsorptive activity.

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INTRODUCTION

Nanotechnology is an expanding discipline of research which has gained significant momentum in recent years. There is tremendous demand of nanoparticles in the field of chemistry, medicine as well as biotechnology [1]. "Nano" means one- billionth, that means, objects which belong between 1 -100 nm in size. Nowadays, different types of metals and metal oxides are used to produce nanoparticles such as silver, copper, gold, zinc, titanium, magnesium and alginate [2]. Out of different metal oxide nanoparticles, Zinc oxide nanoparticles exhibit unique physical, chemical and biological properties such as high electron mobility, wide bandgap, high piezoelectric property and thus these nanoparticles have large number of applications in laser devices, optoelectronic devices, electromagnetic coupled sensors etc. ZnO nanoparticles are reported to possess antibacterial, anticancer, antidiabetic, antioxidant, antifungal, antiparasitic, anti-inflammatory and wound healing properties and thus have potential biomedical applications. ZnO nanoparticles has been listed by US FDA as "Generally Recognized As Safe" (GRAS). Thus, Zinc oxide nanoparticles are drawing attention of researchers for its wide applications in medicine, food, agriculture, cosmetics, pharmaceutical, textile, rubber and electronic industries [3] [4].

In literature, synthesis of ZnO nanoparticles have been reported from conventional physicochemical methods such as chemical reduction, solvothermal, inert gas condensation, sol-gel method etc. but these methods require some toxic chemicals, high pressure, laser radiation etc. Few of these methods are expensive, require skilled labour and special instruments [5]. Green synthesis of ZnO nanoparticles has many advantages viz. cost-effective, non-toxic nature, biodegradability, non-hazardous to environment and use of natural resources like plants and microorganisms. Synthesis of ZnO nanoparticles using plant extracts is highly recommended due to the presence of phytochemicals in the plant material which acts as a good reducing agent for ZnO nanoparticles synthesis.

Utilizing natural extracts for synthesis of ZnO nanoparticles has gained much attention of researchers over the globe. Recent studies reported the use of various plant extracts viz. *Ocimum Tenuiflorum* (Tulsi) leaves [5], Olive[6], *Rosa indica* leaves [7], *Lawsonia inermis* (Mehendi) extract [3], *Brassica oleracea* (Broccoli) extract [8], *Cassia fistula* and *Melia azedarach* leaf extracts [9], orange fruit peel extract [10], *Myristica fragrans* (Nutmeg) [11], *Palmata* leaf extract [12], Sea lavender extract [13], Garlic bulb and ginger extracts [14], *Cymbopogon citratus* (Lemongrass) extract [15], Cinnamon and Bay leaves [16], *Salvadora persica* leaf extract [17], *Monoon longifolium* leaf extract [18], *Allium cepa* waste peel extract [19] for green synthesis of ZnO nanoparticles.

Recent study explains on the synergistic effect of coconut milk and coconut water on synthesis of ZnO nanoparticles and use of *Cocos nucifera* leaf extracts for the green synthesis of ZnO nanoparticles [20] [21] [22].

Cocos nucifera Linn commonly known as coconut belongs to the family *Arecaceae* (*Palmae* Family). It is a monocotyledon tree with pinnate leaves of dense canopy and having the height of approximately 25 m [23]. India ranked third in production of coconut after Indonesia and Philippines with 11.4 million tons per year. Coconut water is one of the best sources of delicious hydration. The chemical composition of coconut water as it contains 95.50% of water, 6.60% of potassium, 2.80% of total sugar, 0.80% of reducing sugar and 0.62% of ash, sucrose and vitamins B complex including nicotinic acid, pantothenic acid, biotin, and folic acid. Phytochemicals present in the coconut water includes flavonoids, gallic acid, tannins, saponins, tocopherols, nicotine, caffeine, oxalic acid etc. Flavonoids and terpenoids present in the coconut water act as reducing and stabilizing agents which further assist in the synthesis of ZnO nanoparticles [24]. There are many health benefits of coconut water reported such as it helps to protect against free radicals, reduces blood sugar, blood pressure, balance electrolytes, may prevent kidney stone formation, relief from muscle cramps, strengthen bones, hair provides healthy skin etc.

Thus, the present study reported green synthesis of ZnO nanoparticles using *Cocos nucifera* i.e. coconut water and its detailed characterization with respect to UV-Visible spectroscopy, X- ray Diffraction studies and Field Emission Scanning Electron Microscopy. Furthermore, the adsorptive property of ZnO nanoparticles for the removal of organic cationic dye Malachite Green from aqueous solution was studied.

MATERIALS AND METHODS

Sample Collection

Cocos nucifera (coconut) water was collected from young coconut bought from local fruit vendors of Pimpri, District Pune, State Maharashtra, India.

Chemicals and reagents

All chemicals and reagents used in the present study were of analytical grades, and Reagent-grade (purity $\geq 98\%$) in particular Zinc acetate dihydrate ($\text{Zn}(\text{CH}_3\text{CO}_2)_2 \cdot 2\text{H}_2\text{O}$), nutrient broth and Dimethyl Sulfoxide (DMSO) were purchased from HiMedia Laboratories Pvt. Ltd., Mumbai, India. Malachite green was purchased from SRL Chem, Pune.

Synthesis of zinc oxide nanoparticles (ZnO NPs)

For green synthesis of zinc oxide nanoparticles, 20 ml fresh *Cocos nucifera* (coconut) water was taken and filtered through Whatman's filter paper no.1. This water was heated to 60°C for 15 minutes and then kept on heating magnetic stirrer for 2 hrs. 1gm of zinc acetate was dissolved in 50ml of distilled water and this solution was added drop wise to coconut water placed on heating magnetic stirrer for 2 hrs with continuous stirring. Formation of white precipitate after incubation confirmed the synthesis of zinc oxide nanoparticles. The solution was centrifuged in bench top centrifuge (R-8C DX Laboratory Centrifuge, REMI, REMI ELEKTROTECHNIK Ltd.) at 8000 rpm for 20 minutes till the white precipitate gets settled down as a pellet. The white precipitate of zinc oxide nanoparticles was washed with distilled water for 3 times and was allowed to dry for 2 hrs in the oven set at 100°C [25].

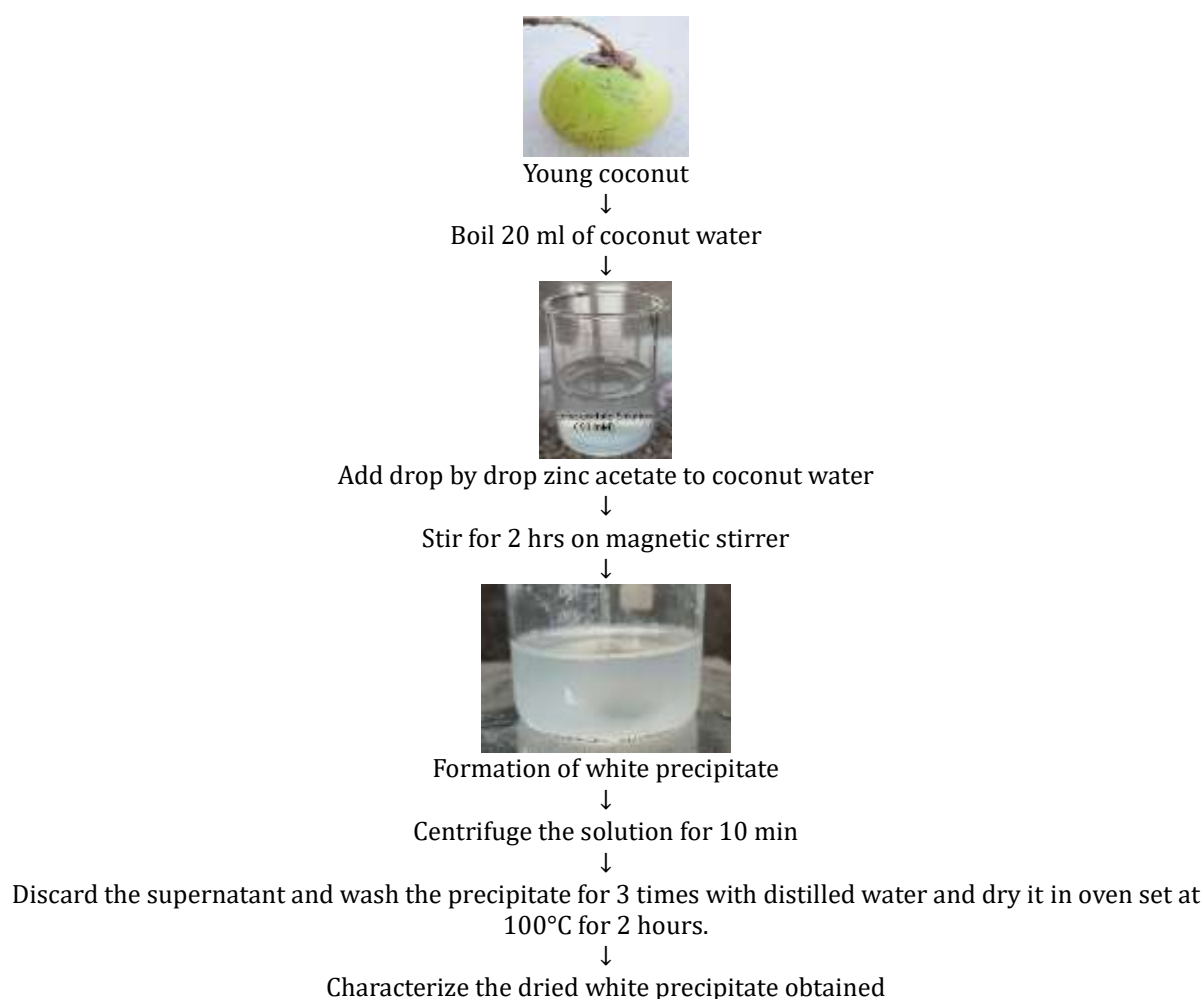


Figure 1: Overview of Synthesis of ZnO nanoparticles using *Cocos nucifera* (Coconut) water

Characterization of ZnO nanoparticles

For the confirmation of synthesis of ZnO NPs nanoparticles using *Cocos nucifera* water, the synthesized nanoparticles were characterized by using different techniques such as UV-Visible spectroscopy, X-ray Diffraction analysis and Field emission Scanning Electron microscopy.

UV-Visible Spectroscopy

The optical properties of the prepared ZnO NPs were studied using UV-Visible spectroscopy. The white precipitate of ZnO NPs obtained in the synthesis process was dissolved in 1ml butanol and the UV-spectra for this sample was scanned between the range of 200-700 nm on SHIMADZU UV-Spectrophotometer (Serial No- A11455009292) to observe the characteristic peak confirming ZnO NPs formation. 1ml butanol without sample was used as the blank.

X-ray diffraction (XRD) analysis

In order to characterize the crystallinity of the ZnO nanoparticles formed, the ZnO NPs synthesized using *Cocos nucifera* water were analysed for XRD using Single crystal X-Ray Diffractometer (Bruker D8 Venture), using Cu/40kV/40mA as the X-Ray source with K-beta filter.

Field Emission Scanning Electron Microscope (FESEM) analysis

To analyse the surface morphology of the ZnO nanoparticles formed, FESEM was performed using Nova NanoSEM 450 and EDS: Bruker XFlash 6130.

Adsorptive activity of ZnO nanoparticles

Here, we report the adsorptive activity of ZnO nanoparticles formed using *Cocos nucifera* (Coconut) water against organic cationic dye Malachite Green. 5 ml dye solution of Malachite green having concentration 0.5 mg/ml was mixed with ZnO NPs having concentration 1mg/ml. Initial absorbance was obtained at time zero using colorimeter (620 nm). The solution was kept at incubation for 24 hours at room temperature. After 24 hours, the test solution showing decolourization was used to check the absorptive trend of ZnO nanoparticles formed by measuring the absorbance after every hour.

RESULTS AND DISCUSSION

Synthesis of zinc nanoparticles using *Cocos nucifera* water

There are different physicochemical methods for synthesis of ZnO nanoparticles but to avoid the drawbacks of these methods, green synthesis of ZnO nanoparticles is employed using *Cocos nucifera* water as a stabilizing as well as reducing agent. Recent studies reported the green synthesis of ZnO nanoparticles using *Cocos nucifera* leaf extracts [21] [22] and also reported the process optimization of green synthesis of ZnO nanoparticles mediated by coconut water using response surface methodology (RSM) [26]. Phytochemicals such as flavonoids and terpenoids present in the coconut water act as reducing and stabilizing agents which further assist in the synthesis of ZnO nanoparticles. Thus, *Cocos nucifera* water was selected for the green synthesis of ZnO nanoparticles. Fig. 1 shows the flow of synthesis of ZnO nanoparticles using *Cocos nucifera* water. The ZnO nanoparticle formation was examined by the white precipitate formation. The white precipitate formed during the synthesis of nanoparticles is used for further studies.

Characterization of ZnO nanoparticles

Ultraviolet-Visible spectroscopy analysis

In order to study the optical absorption property of the synthesized ZnO nanoparticles using *Cocos nucifera* water, the UV-visible absorbance spectra was monitored at room temperature in the wavelengths of 200 to 700 nm. The UV-Visible spectra of synthesized ZnO nanoparticles showed the absorption maxima at a range between 340 and 380 nm, which is specific for ZnO NPs. This confirms the green synthesis of ZnO

nanoparticles from *Cocos nucifera* water. Our results correlate with the earlier studies [12] [13] [15] and [21].

X-ray diffraction (XRD) analysis

XRD method is one of the non-destructive analytical method for recognizing and quantifying the various crystalline forms found in both powder and solid materials. Fig. 2. describes the XRD pattern of the ZnO nanoparticles. The XRD pattern of ZnO NPs reveals sharp peaks that indicate the purity and crystallinity of green synthesized ZnO NPs using *Cocos nucifera* water. The peaks in the graph shows that the dominated peaks are at the orientation of nearby 30 degrees / 2θ for ZnO nanoparticles. The peaks were identified from 30-60° which confirms the formation of crystalline ZnO nanoparticles.

According to earlier reports of [13] [15] and [21], the XRD pattern of ZnO nanoparticles produced revealed different diffraction peaks appearing at 2θ angles of 31.83°, 34.46°, 36.28°, 47.58°, 56.62°, 62.91°, 66.46°, 68.06° and 69.10° corresponding to the Miller indices of 100, 002, 101, 102, 110, 103, 112, 200 and 201, respectively confirms the formation of ZnO hexagonal wurtzite structure, which agrees with the JCPDS Data Card No. 36-1451 ZnO nanoparticles.

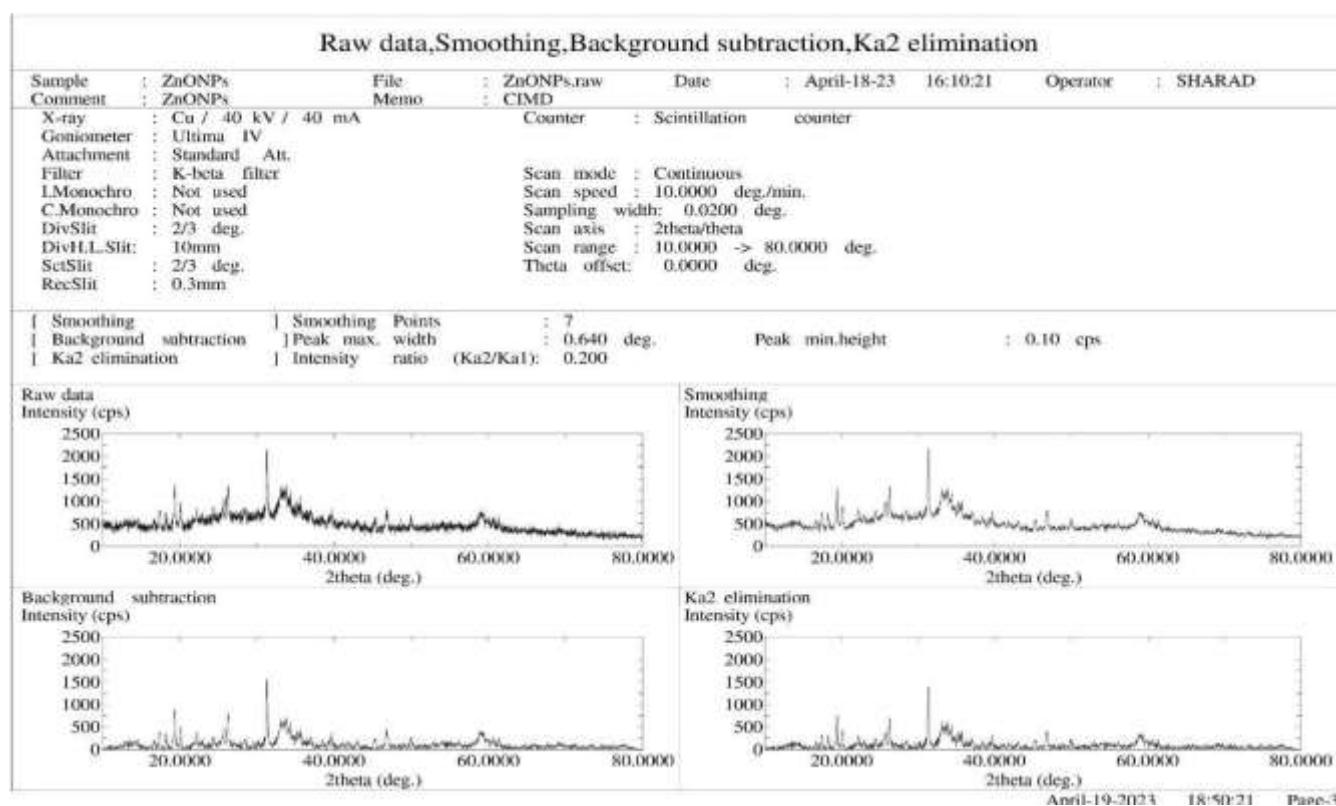


Figure 2: XRD pattern of ZnO NPs prepared using *Cocos nucifera* leaf extract.

Field Emission Scanning Electron Microscope (FESEM) analysis

Surface morphology of the synthesized ZnO NPs was examined through FESEM analysis at various magnification levels (100000 X and 200000 X) (Fig 3a and 3b). Fig. 3a and b shows the SEM images of irregularly shaped ZnO nanoparticles with both round and hexagonal shapes. These nanoparticles have size ranged from 22.97 to 61.18 nm. Earlier studies reported the size of ZnO nanoparticles synthesized from coconut milk and coconut water as 20 to 80 nm which is comparable with our reports [20]. Agglomerations of individual zinc oxide particles were observed in the images (Fig. 3), which shows the tendency of the nanoparticles to form clusters. Similar agglomeration of ZnO nanoparticles was observed in the reports of [12] and [18]. According to reports of [12], agglomeration of ZnO NPs was due to the high surface energy present on the surface of nanoparticles. The round and the hexagonal shapes of the ZnO nanoparticles were reported by [19]. Rod and hexagonal shape of ZnO nanoparticles synthesized from *Salvadora persica* leaf extract was reported by [17].

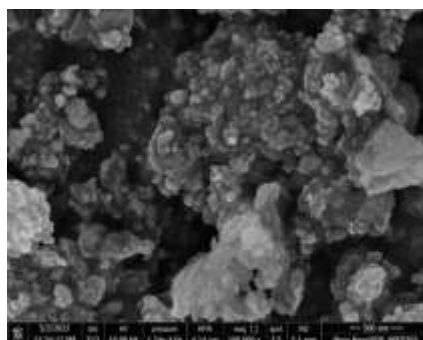


Figure 3a: FE-SEM images of ZnO-NPs. (100000x magnification)

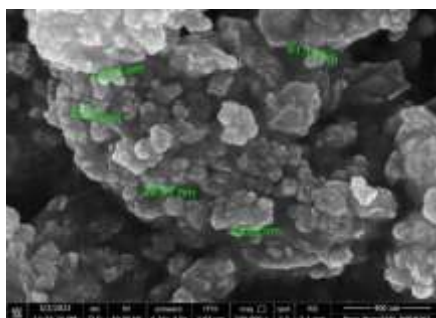


Figure 3b: FE-SEM images of ZnO-NPs. (200000x magnification)

Adsorptive activity of ZnO nanoparticles

The dye adsorptive trend of ZnO NPs with respect to organic cationic dye Malachite Green was seen after 24 hrs of incubation (Fig. 4 and Fig. 5). The removal of Malachite Green dye from aqueous solution because of its adsorption was checked using colorimeter (Fig. 5). The decrease in optical density of the dye solution was obtained after 24 hrs was used to check the absorptive trend of ZnO NPs for dye absorption against blank control. For Malachite green, the absorbance was taken at 620 nm (Fig 5). From graph, it can be seen that the absorbance of malachite green dye solution decreased with the time. The initial absorbance was recorded as 2.538 whereas after 31 hrs, the absorbance of malachite green solution with ZnO nanoparticles was recorded as 2.149. This decrease in absorbance trend showed that ZnO NPs have good absorptive property towards organic cationic dye Malachite Green (Fig. 5).



A B

Figure 4: (A) Blank with Malachite Green dye solution without ZnO nanoparticles (B) Adsorption of Malachite Green dye on ZnO nanoparticles formed using *Cocos nucifera* water after 24 hrs incubation.

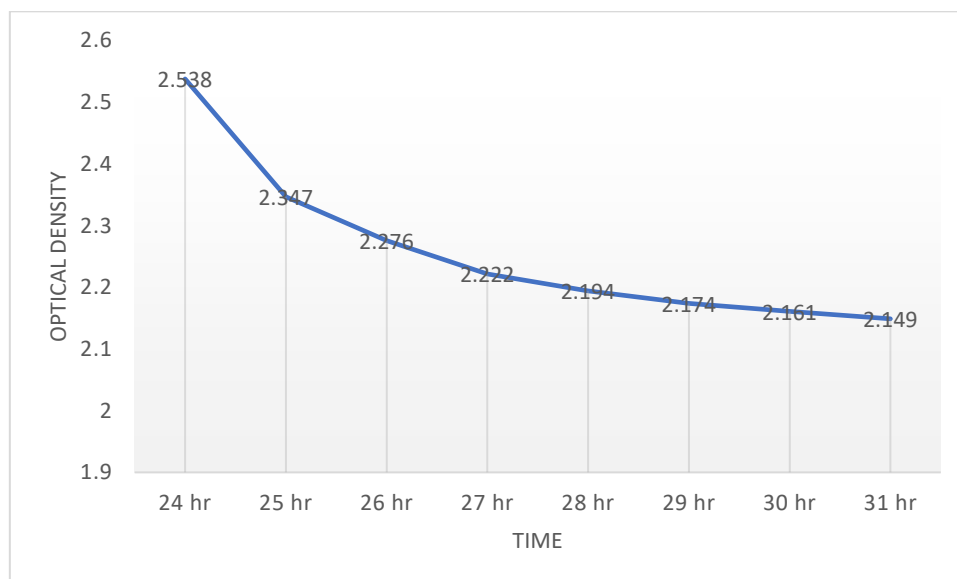


Figure 5: Adsorptive effect of ZnO nanoparticles with respect to organic cationic dye Malachite Green.

Earlier studies reported the high adsorptive capacity of ZnO nanoparticles for dye removal by using both cationic dye malachite green, and anionic dyes acid fuchsin and Congo red [27]. According to reports of [26], ZnO nanoparticles can remove both anionic as well as cationic dyes efficiently. The adsorption process was spontaneous and exothermic. Organic dyes are widely used in various sectors like textiles, rubber, paint, plastic, cosmetics etc. and thus dye containing wastewater is always a serious environmental concern. These dyes are harmful to the aquatic life, thus it is necessary to remove these organic dyes from wastewater before being released into natural water bodies. Recently, metal and metal oxide nanoparticles are used as adsorbent in removal of dyes from industrial wastewater as one of the wastewater treatment processes. ZnO nanoparticles have recently emerged as a promising adsorbent because of their high surface area, controllable surface properties, and strong dye-adsorbent interactions. Because of these properties ZnO nanoparticles can physically trap the dye molecules through various interactions like hydrogen bonding, electrostatic interactions etc. Recently, [17] also reported the methyl orange dye removal by adsorption using ZnO nanoparticles synthesized from *Salvadora persica* leaf extract. Recent review stated the current advances of ZnO nanoparticles for dye removal [28]. Thus, our study reported the decrease in absorbance trend of Malachite Green dye solution when incubated with ZnO nanoparticles which showed that ZnO NPs synthesized using *Cocos nucifera* water have good adsorptive property towards organic cationic dye Malachite Green and indicating their potential for efficient dye removal in wastewater treatment.

CONCLUSION

The present research represents green synthesis of ZnO nanoparticles using *Cocos nucifera* water i.e. coconut water and its detailed characterization with respect to UV-Visible spectroscopy, X-ray Diffraction studies and Field Emission Scanning Electron Microscopy. The UV-Visible spectra of synthesized ZnO nanoparticles showed the absorption maxima at a range between 340 and 380 nm which confirms the synthesis of ZnO nanoparticles. The XRD pattern of ZnO NPs reveals sharp peaks that indicate the purity and crystallinity of green synthesized ZnO NPs using *Cocos nucifera* water. The Surface morphology of the synthesized ZnO NPs was examined through FESEM analysis and it was observed that ZnO nanoparticles using *Cocos nucifera* water are irregularly shaped ZnO nanoparticles with both round and hexagonal shapes. These nanoparticles have size ranged from 22.97 to 61.18 nm and agglomerations of individual zinc oxide particles were observed which shows the tendency of the nanoparticles to form clusters. Adsorptive activity of ZnO nanoparticles with respect to organic cationic dye Malachite Green was observed after 31 hrs incubation as evidenced by decrease in the absorbance. Thus, ZnO NPs synthesized using *Cocos nucifera* water have good adsorptive property towards organic cationic dye Malachite Green and indicating their potential for efficient dye removal in wastewater treatment.

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