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PHOTOCATALYTIC PROPERTIES OF BIOLOGICALLY SYNTHESIZED UNCOATED AND CALCIUM COATED ZnO NANOPARTICLES USING CUCUMBER JUICE

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ABSTRACT

Uncoated ZnO and calcium-coated ZnO nanoparticles (Ca-ZnO) were biologically synthesized using cucumber juice as a reducing and capping agent. Cucumber belongs to *Cucurbitaceae* family, is rich in water content and possesses antioxidant activity. The chemical composition and morphology of the synthesized NPs were determined by scanning electron microscopy (SEM), energy dispersive X-ray spectrophotometer (EDS), high-resolution transmission electron microscopy (HR-TEM), X-ray diffraction (XRD)and FT-IR. The spherical as well as hexagonal-shaped ZnO nanoparticles were obtained in the size range of 4-70 nm. Coated and uncoated ZnO nanoparticles were evaluated for their photo degradability of methylene blue (MB) and rhodamine B (RB) dyes. They showed nearly 100% photodegradation of methylene blue in lesser time, however, showed minimum degradation of rhodamine B dye. Calcium coating over ZnO NPs enhanced their photocatalytic properties.

Keywords: Cucumber Juice, Methylene Blue, Rhodamine Blue, Biological Method, ZnO NPs, Photocatalysts.

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INTRODUCTION

These days world is facing two major challenges associated with "P" i.e., Population explosion and Pollution. Both Ps are in the hands of human beings. Although population explosion has been controlled to some extent by awareness campaign programs, but pollution seems to be difficult in the era of rapid industrialization. Several agricultural practices as well as industrial activities like the use of pesticides, herbicides, dyes, pharmaceuticals, etc.[1-3] are the matter of concern for the scientific world and environmentalists. These anthropogenic activities lead to water and soil pollution. Organic pollutant effluents in water, textile and pharmaceutical industries, in particular, are the biggest challenges that require attention to maintain a healthy environment. Textile industries discharge dyes and pigments loaded water into natural water resources. These organic pollutants adversely disturb the ecological balance in aquatic life due to their toxicity, complexity of structure and perseverance. However, industries have been using various physical techniques such as coagulation, ion exchange chromatography, activated carbon, reverse osmosis, and ultrafiltration for their disposal.[4-5] In this regard, the development of efficient technology for water as well as soil remediation is imperative to safeguard the environment. In recent years, several nano-based technologies have been adopted due to wide applications from solar fuel, cosmetic, medicaland photocatalytic properties. Several metal/metal oxide nanoparticles such as Ag, Au, TiO_2, CuO, ZnO, etc. have been examined for their photocatalytic properties in the decolorization of textile wastewater.[6-9] These nanoparticles have frequently been used in the treatment of wastewater as they lead tothe complete mineralization of contaminants due to redox reactions.[10-11] Nanoparticles of rare earth metals have also been gaining interest because of their good optical and catalytic properties.[12-14] Among all, Titanium oxide (TiO_2)

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and zinc oxide (ZnO) nanoparticles are well known for dye degradation in sole capacity as well as in composite form. Zinc oxide nanoparticles are the most studied after titanium oxide due to their comparable physical &chemical properties such as wide band gap (3.37eV), stability, inexpensive, oxidation-reduction potential, the non-toxic and high binding energy of excitons, moreover can easily be prepared.[15-17] The main challenge that affects the efficiency of the photocatalyst is the fast recombination of photogenerated electron-hole pairs in the semiconducting material. The scientific world has come up with many ideas to address this issue such as coupling with another semiconductor or doping with a suitable atom (N, C),or addition of an electron acceptor etc.[18-20]Coating of noble metals on nanoparticles have also been investigated to reduce the recombination rate and increase the photocatalytic properties of the nanomaterials. Heterostructured NPs show better properties in dye degradation under solar light.[21-22]The photocatalytic activity of nanomaterials is enhanced due to the photoreduction of noble metal ions to nanoparticles. Zn/Ca mixed ferrites NPs are effective photocatalysts. CaO nanoparticles have been explored in degrading organic dyes under optimum conditions. The addition of calcium in ZnO reportedly improves the properties of ZnO nanoparticles as compared to the neat ZnO NPs.[23-24]Micro rods of calcium carbonate have been used to degrade methyl violet.[25]Although nanoparticles have been synthesized by various physicochemical techniques, but biosynthesized NPs have several advantages over chemically synthesized NPs due to low toxicity, involve one-pot reaction and environment benign.[26] Cucumber is a popular crop used in ayurvedic medicine since ancient times. Cucumber juice contains several potent bioactive compounds such as vitexin, orientin, apigenin, cucurbitacins, etc. which can act as reducing agents and also aid in the stabilization of NPs. Little attention has been given to studying the effect of calcium coating on ZnO NPs in dye degradation. In this paper, we have proposed the biosynthesis of cucumber juice-mediated ZnO nanoparticles and calcium-coated ZnO nanoparticles (Ca-ZnO). Synthesized nanomaterials were evaluated for their catalytic activity in the degradation of Methylene blue (MB) and Rhodamine B (RB).

EXPERIMENTAL

Materialand Methods

$Zn(CH_3COO)_2.2H_2O$ (Zinc acetate dihydrate), Calcium Chloride and sodium hydroxide were purchased from SRL (India). Methylene blue and Rhodamine B were bought from Sigma-Aldrich (USA). Chemicals were used as such in the research and did not purify any further. Double distilled water was used in the synthesis. Fresh cucumber was purchased from the local market for extract preparation.

Preparation of Cucumber Juice Extract

Cucumber was washed with tap water to remove dirt particles followed by peeling. Peeled cucumber was grated and pressed to get the juice. Fresh juice was used every time for the biosynthesis of uncoated and coated ZnO NPs.

Synthesis of Cucumber juice mediated ZnO nanoparticles (CJA)

Cucumber juice-mediated ZnO nanoparticles were synthesized via a modified co-precipitation method.[27]Approximately 50 ml of fresh cucumber juice was taken in a beaker containing a magnetic bead. It was heated to about 60°C at pH~8 on a magnetic stirrer for 15-20 minutes. The pH of the cucumber juice/extract was maintained by adding sodium hydroxide. Weighed quantity of zinc acetate was added to a resulting solution with continuous stirring for 15-20 minutes. Stirring was continued for another one hour at room temperature. The formed precipitate was filtered and washed with deionized water (Fig.-1). ZnO NPs were first dried in the oven and then calcinated at 500°C in a muffle furnace for one hour. The sample was stored in an air-tight container for further analysis.

Synthesis of Calcium-coated ZnO nanoparticles (CJ11 and CJ21)

Calcium-coated ZnO nanoparticles in two different weight ratios (1:1 and 1:2) were synthesized similarly as mentioned above. The weighed amounts of zinc acetate were dissolved in 50 ml of fresh cucumber juice taken in two beakers maintained at 60°C and pH~8. Calcium salt in two weight ratios (1:1 and 1:2) w.r.t. to zinc salt was added in respective beakers under continuous stirring conditions for 1 hour at room temperature. The formed precipitate was drained and calcinated at 500°C for one hour. The resulting samples were named CJ11 (Zn: Ca=1:1) and CJ21 (Zn: Ca=2:1). The samples were stored in air-tight containers for further analysis.

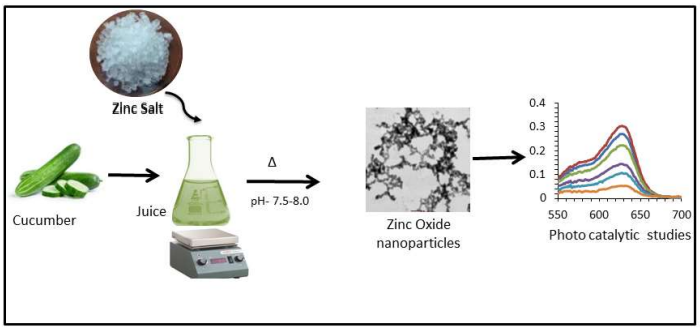

Fig.-1: Schematic representation of biosynthesized ZnO Nanoparticles

Characterization

Synthesized ZnO nanoparticles were characterized by HR-TEM, FT-IR, SEM coupled with EDAX and XRD. The absorbance of the synthesized nanomaterials was determined by a UV-Vis spectrophotometer (Motras) in the range of 200-800 nm. X-ray diffraction patterns (XRD) were recorded on a Bruker, D8 Discover diffractometer with CuKα radiation (λ=1.540598Aº), set at 45kV and 40 mA. Scans were performed with a detector size of 0.02º with 2θ range from 10º-70º. The morphology of the prepared ZnO nanoparticles was analyzed by Scanning Electron Microscope (SEM) with energy dispersive X-ray analysis (EDAX), (JEOL JSM-6610LV) and High-Resolution Transmission Electron Microscope (HR-TEM) (Thermo Fischer Scientific Talos L120C). FT-IR was recorded on Nicolet iS50. The FT-IR samples were prepared by grinding a small sample (app. 2mg) with KBr salt. The prepared disk was then placed in a holder and spectra were recorded in the range of 4000 to 400 cm^{-1} to determine the biomolecules involved in the stabilization of synthesized nanomaterials.

Photocatalytic Degradation

A stock solution of 10mg/L of rhodamine B (RB) and methylene blue (MB) was prepared in double distilled water. For photocatalytic activity, a certain amount of the prepared ZnO sample (CJA, CJ-11, and CJ21) was added to a 100 mL stock solution contained in a 250 C.C. conical flask. The solution was shaken thoroughly in dark at room temperature to ensure adsorption/desorption equilibria before solar light irradiation. Control of both dyes was also kept without the samples. 2-3 ml aliquots were taken out after a fixed time interval and photocatalytic degradation activity was determined using a UV-Visible spectrophotometer in the different wavelengths. The concentration of the dye at different time intervals was calculated by measuring the absorbance value of methylene blue at 627 nm and rhodamine B at 554 nm. To explore the photocatalytic activity of synthesized nanoparticles, the experiments were replicated.[28]

The percentage of dye degradation was determined by the formula given as follows:

$$\% \text{ of dye degradation} = \frac{(C_i - C_f)}{C_i} \times 100$$

Where, C_i is the initial concentration of the dye (mg/L), C_f is the concentration of dye after a fixed time interval (mg/L).

RESULTS AND DISCUSSION

XRD

To determine the phase evolution of the prepared uncoated ZnO and Ca-coated ZnO nanoparticles (CJ11 and CJ21), XRD characterization was done at room temperature. Fig.-2 displays the XRD pattern of CJA, CJ21 and CJ11. X-ray diffraction pattern of uncoated (CJA) shows 2θ values at 31.82º, 34.53º, 36.39º, 47.81º, 56.64º, 63.04º, and 68.19º. The strong and broad pattern of diffraction peaks of uncoated ZnO confirms the presence of a hexagonal wurtzite structure (JCPDS data card no: 79-0208). Although ZnO exists in hexagonal wurtzite and cubic zinc blend forms. But wurtzite structure is most stable under ambient conditions and thus commonly found. This also shows that synthesized ZnO nanoparticles do not contain any impurity as no other peaks are present.[29] Coating of calcium into ZnO structure introduced additional

peaks of low intensities at an angle of 29.72º, 43.69º, 45.87º and 75.70º respectively. It may be noted further that intensity of the peaks diminished with a coating of calcium ions on the surface of ZnO NPs and also there is a slight shift of peaks towards the lower 2θº side. The difference in ion size causes the local distribution of lattice.[30] Furthermore, it is important to note that orientation on the (002) atomic plane remains the same in all the synthesized samples. This revealed that the hexagonal wurtzite symmetry of ZnO is maintained even after Ca coating.[31-33]

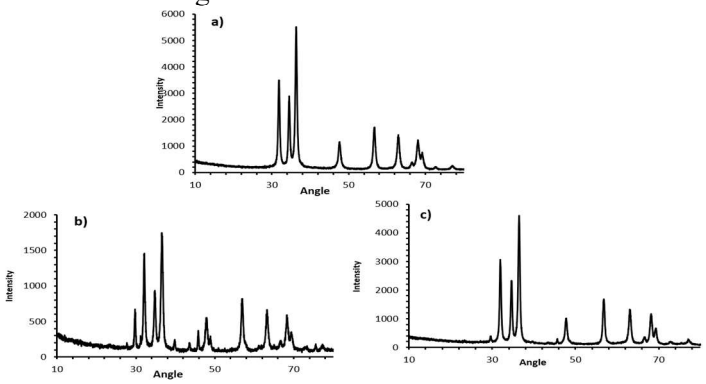

Fig.-2: XRD Patterns of (a) CJA, (b) CJ21, and (c) CJ11

FT-IR

Functional groups present in the cucumber juice that aid in the bioreduction and capping of the ZnO NPs were identified using FT-IR analysis. Polyphenolic compounds like flavonoids are potent biomolecules that are involved in biosynthesis.[34-35] The FT-IR spectra of cucumber juice, ZnO nanoparticles and calcium-coated ZnO nanoparticles are depicted in Fig.-3. The sharp peaks in the range of 880-1380 cm^{-1}, 1527-1700 cm^{-1}, and broad peaks at 3452 cm^{-1} are due to C-O, C=O, and O-H mode of vibrations related to polyphenolic compounds and alkaloids respectively.[36-37] Functional groups present in the cucumber juice are the source of electrons that can reduce Zn^{2+} to Zn^{1+} and eventually to ZnO NPs. Moreover, negatively charged functional groups might have a stabilizing effect. The Zn-O bending vibration appeared around 526 cm^{-1} in the FT-IR spectra.[38]

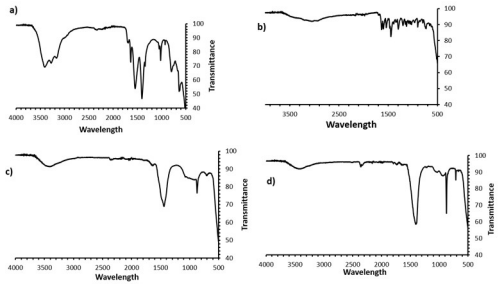

Fig.-3: FT-IR Spectra of a) Cucumber Juice, b) CJA, c) CJ21 and d) CJ11

Morphology and Composition of Uncoated and Calcium-Coated ZnO NPs

SEM micrographs and elemental composition of uncoated (CJA) and calcium-coated ZnO NPs (CJ21 & CJ11) are depicted in Fig.-4. The SEM micrograph of uncoated ZnO nanoparticles revealed clusters of small particles having spherical shapes whereas coating of calcium on ZnO nanoparticles slightly changes its morphology from spherical to irregular hexagonal shape (HR-TEM images). The SEM images clearly show the occurrence of agglomeration and also established a similitude in the morphology of the coated ZnO nanoparticles. It is a result of the uniform distribution of Ca on the surface of ZnO NPs. As per HR-TEM images (Fig.- 5), the ZnO NPs are spherical in shape having a small particle size accumulated in aggregate. The grain size of ZnO nanoparticles is in the range of 4-70 nm. Particle size got increased with calcium coating over the surface of ZnO NPs which is due to the replacement of large-size Ca^{2+} into Zn^{2+} sites. Although less concentration of calcium did not affect the shape of ZnO NPs (CJ21) higher concentration led to the modification of shape closer to hexagonal shape.[39]

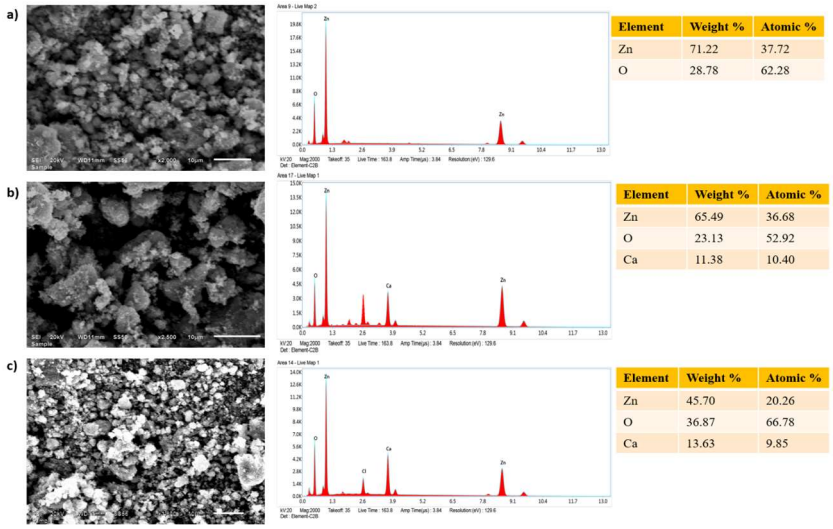

Fig.-4: SEM Images and EDX of (a) CJA, (b) CJ21 and (c) CJ11

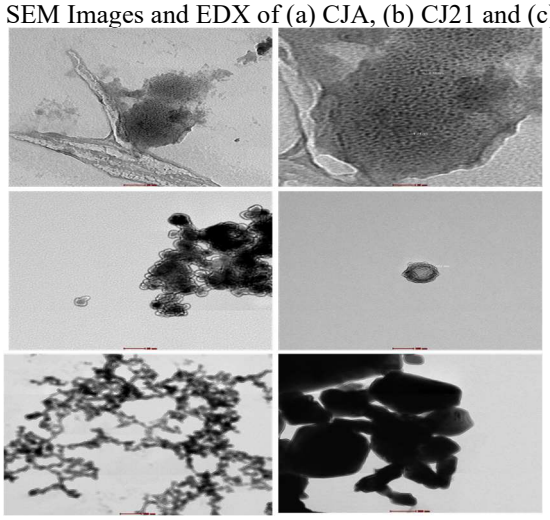

Fig.-5: HR-TEM Images of a) CJA, b) CJ21 and c) CJ11

Photocatalytic Properties

ZnO NPs are potent photocatalysts due to their higher photocatalytic and optical properties. Photocatalysis on the surface of semiconductors is the environment benign solution in the degradation of organic compounds/dyes in wastewater.[40-41] This process involves the generation of electrons/holes in the conduction band and valence band when irradiated with the source of light.[42] Produced electron and hole then migrate to the surface of the semiconductor where it reacts with water and oxygen molecules to produce reactive oxygen ($O^{\cdot}$) and hydroxyl ($OH^{\cdot}$) free radicals. Oxygen and hydroxyl free radicals are strong oxidizing agents that degrade the complex dye molecules in polluted water. The catalytic properties of the synthesized ZnO NPs in environmental remediation were determined by studying their photocatalytic degradation of the rhodamine B (RB) and methylene blue (MB) dyes under natural sunlight. Figure-6 illustrates the photocatalytic properties of uncoated and coated ZnO NPs. Synthesized ZnO NPs demonstrated the highest photocatalytic activity app. 100% degradation of methylene blue dye within 40-50 minutes. Photocatalytic activity increases with Ca coating (CJ21). Further rise in Ca concentration did not show enhanced activity (CJ11). Although prepared nano samples showed poor catalytic activity towards rhodamine B dye, here also calcium coating enhanced its catalytic activity. Higher loading of the Ca might have occupied the catalytic sites present on the surface of the semiconductor which is necessary for the adsorption of dyes hence hampering the removal of dye from the solution.[43-45] The whole process can be summarized as follows:

$$ZnO/Ca\text{-}ZnO + h\nu \longrightarrow e\ ZnO/Ca\text{-}ZnO^{-} + h\ ZnO/Ca\text{-}ZnO^{+}$$

$$e\ ZnO/Ca\text{-}ZnO^{-} + O_2 \longrightarrow OH^{\cdot}$$

$$OH^{\cdot} + Dye \longrightarrow Degraded\ products$$

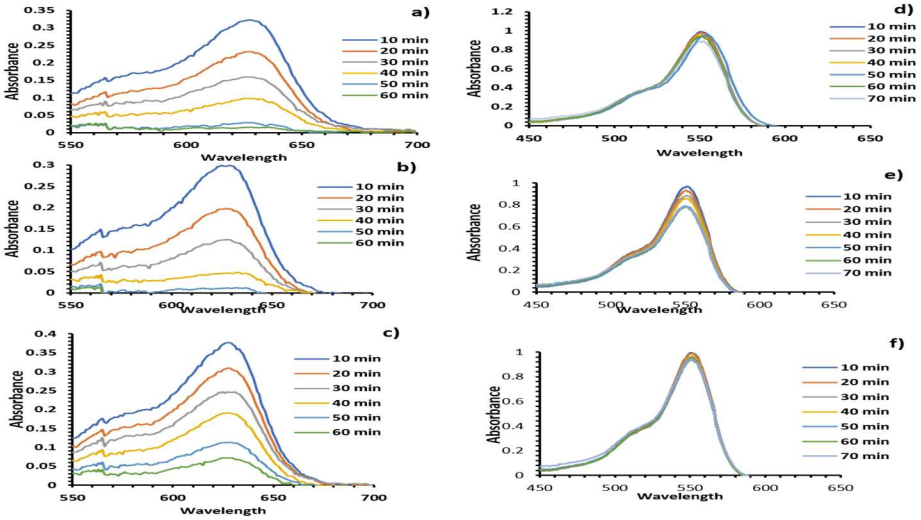

Fig.-6:UV-Vis Spectra of Degradation of Methylene Blue by (a) CJA, (b) CJ21, (c) CJ11 and UV-Vis Spectra Of Degradation of Rhodamine B by (e) CJA, (f) CJ21, (g) CJ11 at different time intervals

CONCLUSION

Herein, we report the biosynthesis of ZnO and calcium-coated ZnO NPs (Ca-ZnO) using cucumber juice as a reducing and stabilizing agent. Uncoated ZnO and coated ZnO nanoparticles were synthesized successfully and characterized by various spectroscopic techniques. All samples showed nearly 100 percent degradation of methylene blue dye and coating of calcium over ZnO NPs enhanced the their catalytic activity. However, there was minimal catalytic activity observed towards rhodamine B but here also calcium coating increased photocatalytic activity. Further rise in calcium concentration decreased the catalytic properties of the ZnO due to the accumulation/adsorption of calcium on its surface.

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CONFLICT OF INTEREST

There is no conflict of interest for the publication of the article.

AUTHOR CONTRIBUTIONS

All the authors contributed significantly to the manuscript, participated in reviewing/editing, and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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