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Mimosa pudica PLANT EXTRACT-MEDIATED GREEN SYNTHESIZED CERIUM DIOXIDE NANOCATALYST FOR SONOLYTIC DEGRADATION OF METHYL ORANGE FROM WASTEWATER

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ABSTRACT
In this present study, cerium oxide nanoparticles were synthesized using *Mimosa pudica* leaf extract and characterized. The UV-visible absorption spectrum of *Mimosa pudica* plant extract mediated cerium oxide nanoparticles showed a sharp peak at 315 nm with the band gap energy of 2.7 eV, which indicates the formation of Cerium oxide. Surface morphology of cerium oxide nanoparticles was analyzed using scanning electron microscopy, which showed the presence of spherical particles with agglomeration. The crystalline structure of nanoparticles was determined by X-ray diffraction analysis, indicating a cubic crystal phase with a particle size of 12.3 nm, and it was confirmed in the particle size determined by the dynamic light scattering method. Sonocatalytic efficiency of cerium oxide nanoparticles was also tested for the degradation of methyl orange dye. The experimental observations reveal that maximum degradation of methyl orange dye was obtained at pH 3.0 in the presence of 0.1 mg of catalyst cerium oxide nanoparticles by exposing to ultrasonic waves for 120 min. The efficiency of degradation of the dye in the presence of cerium oxide nanocatalyst was found to be six times greater than in the absence of a catalyst.
Keywords: Sonacatalyst, Cerium Oxide, Methyl Orange, Plant Extract, *Mimosa Pudica*, Dye Removal.
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INTRODUCTION

Discharge of textile, tanning, cosmetics, and pharmaceutical industrial effluent brings inorganic and organic pollutants such as dyes, heavy metals, etc, into the water resources. Organic dyes are non-degradable, toxic, carcinogenic chemicals and cause serious health problems such as skin rashes, allergies, cancer in human beings and animals.[1] There are numerous physicochemical techniques available to remove organic dyes from wastewater. However, those techniques are not eco-friendly, cost-effective, and leave sludge contaminants in the environment.[2] As it is known, the chemical transformations of different species can be performed through ultraviolet radiation (UV-Vis), as well as suitable catalysts. Cerium oxide (CeO_2) is one of the metal oxides of lanthanide, which can participate in redox reactions. CeO_2-NPs have received enormous attention in nanotechnology due to their viable applications, such as drug delivery, biocatalysts, photo-catalyst, optical devices, oxygen sensor, fuel cells, and ultraviolet absorber.[3] The manufacturing of these CeO_2-nanoparticles by various methods such as hydrothermal, solvothermal, co-precipitation, sol-gel, and microwave has been reported in the literature.[4] However, recently, green synthesis has been reported, which involves the preparation of nanoparticles using natural sources, such as plant extracts, bacteria, fungi, and algae that do not create any harmful by-products. The phytochemicals such as flavonoids, terpenoids, glycosides, alkaloids, and amino acids present in plant leaf extracts have massive potential to reduce metallic salts into nanoparticles in a much shorter time as compared to fungi and bacteria, which require longer incubation times. Therefore, plant leaf extracts are an excellent and benign source for metal as well as metal oxide nanoparticle synthesis. Additionally, plant leaf extract plays a dual role by acting as both a reducing and stabilizing agent in the nanoparticles synthesis process to facilitate nanoparticles.[5,6]

Mimosa pudica L. (Mimosaceae) is one of the traditionally used medicinal plants, commonly found in arid and semi-arid regions of America, Africa also in India. Phytochemical studies on *Mimosa Pudica (M.*

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Pudica) revealed the presence of alkaloids, non-protein amino acid (mimosine), flavonoid C-glycosides, steroids, tannins, and fatty acids.[7] In this present study, green synthesis of CeO_2 -Nps from cerium ammonium nitrate using *M. Pudica* leaf extract. The *M. Pudica* leaf extract mediated cerium oxide nanoparticles (*MP*-CeO_2-Nps) are characterized by UV-vis Spectrophotometer, DLS, and SEM analysis. The present study also aimed to evaluate the sonocatalytic efficiency of *MP-CeO2-NPs* to degrade methyl orange (MO) dye.

EXPERIMENTAL

Material and Methods

M. Pudica leaves were collected from the local area of Tiruchirappalli town. Double-distilled water has been used for preparing the plant extract. Laboratory-grade Sodium hydroxide, cerium nitrate, ethanol, and other chemicals were purchased from Nice Chemicals Pvt. *MP*-CeO_2-Nps was characterized using a UV-Visible spectrophotometer (ELICO Model: SL-159) in the range of 200 to 500 nm. Surface morphology of the *MP*-CeO_2-Nps was examined using scanning electron microscopy (SEM-Hitachi (Model S-3000H). Elemental composition of *MP-CeO2-NPs* was determined by Energy Dispersive X-ray (EDX) analysis. The dynamic light scattering (DLS) method was used to determine the average size of the *MP-CeO2-NPs*. X-ray diffraction analysis of *MP-CeO2-NPs* was done using a Bruker X-RAY diffractometer (Model XPERT-PRO) with monochromatized CuKα1 radiation of wavelength λ = 1.5406 Å.

Preparation of Plant Extract and Cerium Oxide Nanoparticles

Plant extract of *M. Pudica* leaves was prepared by taking 25 g of the powdered dry leaves in 500mL of double-distilled water and boiling at 80ºC for half an hour. The colour of the aqueous solution turned light green. The extract was cooled to room temperature and filtered through Whatman No. 1 filter paper. 100 mL of light green coloured leaf extract was taken in a conical flask, and the pH of the solution was adjusted to 8. To the content of the flask, 5 mL of 0.01M aqueous solution of ammonium cerium (IV) nitrate was added with constant stirring. The mixture yields dark yellowish brown colour crystals of cerium oxide nanoparticles. After 20 minutes, the mixture was centrifuged and filtered. The yellowish-brown powder of cerium oxide nanoparticles was collected and dried in an oven at 60ºC for one hour and stored at ambient temperature.[8]

Evaluation of Sonocatalytic Activity of Cerium Oxide Nanoparticles

Sonocatalytic efficiency of *MP-CeO2-NPs* was examined for the degradation of methyl orange (MO) dye in aqueous medium under ultrasonic irradiation. For this study, 0.01mg of the catalyst *MP*-CeO_2-Nps was added to 100mL of an aqueous solution of 10ppm concentrated MO dye taken in an Erlenmeyer flask with constant stirring in a dark room. The solution was adjusted to pH 5.0. Then the flask is kept in a Citizen digital ultrasonic bath at 40 kHz and 120W. The concentration of undegraded MO dye remaining in the solution was determined at different process times using an Elico UV-vis spectrophotometer (Model: SL 159) at 592 nm. The effect of contact time and pH of the medium on the sonocatalytic ability of *MP-CeO2-NPs* on MO dye degradation will also be evaluated. The percentage degradation of dye in the aqueous solution has also been determined using the following eqn.-1.

$$\% \text{ Degradation of the dye} = \left(\frac{C_0 - C}{C_0}\right) \times 100 \qquad (1)$$

Where C_0 And C is are initial and equilibrium concentration of dye in the solution, respectively.[9]

RESULTS AND DISCUSSION

UV-Visible Spectrum of MP-CeO_2-Nps

UV-vis spectral data provide quantitative information about the nature of the oxidation state of the cerium ion in Cerium oxide nanoparticles. A sample absorbs light in both the UV and visible regions corresponding to Ce_2O_3 nanoparticles, while a sample having cerium (IV) ion shows the absorption peak only in the UV region and has higher transparency in the visible region. Figure-1 is the UV-Vis spectrum of *MP*-CeO_2-Nps. This shows an absorption peak at 325 nm, which is attributed to the charge transfer from O_{2p} to Ce_{4f} orbital. This indicates the presence of Ce (IV) ion in *MP*-CeO_2-Nps.[8] Band gap energy (E_g) for the direct allowed transition for *MP*-CeO_2-Nps *has* been calculated using eqn.-2.

$$(\alpha h\vartheta)^{1/2} = B(h\vartheta - E_g) \qquad (2)$$

Where α is the absorption coefficient, B is a constant which is 2 for the direct allowed transition of CeO_2 nanoparticles, $h\vartheta$ It is the energy of a photon.

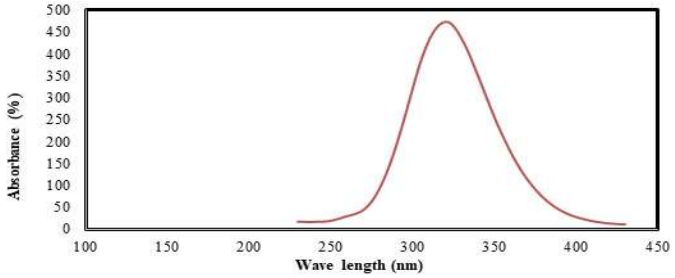

Fig.-1: UV-Visible Spectrum of Cerium Oxide Nanoparticles Synthesized using *M. pudica* Leaf Extract

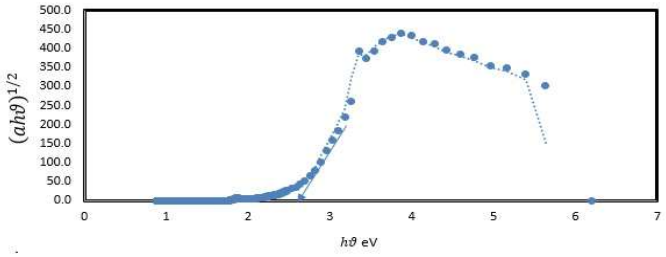

Fig.-2: Tauc Plot of Cerium Oxide Nanoparticles Synthesized using M. pudica Leaf Extract

From the Tauc plot (Fig.-2) of $(\alpha h\vartheta)^{1/2}$ vs $h\vartheta$ The Band gap energy of *MP*-CeO_2-Nps was calculated as 2.7 eV, which is less compared to the band gap energy of bulk CeO_2 (3.19eV). These results revealed that the above synthesized *MP-CeO2-NPs* can be used as a sonocatalyst for the degradation of organic dye.[9]

Morphological Analysis

Surface morphology of *MP-CeO2-NPs* was characterized by using SEM and EDX analysis. Figure-3 shows the SEM morphological image of *MP*-CeO_2-Nps. The SEM image reveals that the presence of spherical shape *MP*-CeO_2-Nps with agglomeration may be due to capping and stabilization of nanoparticles by phytochemicals present in the plant extract. Figure-4 shows the EDX spectrum of *MP*-CeO_2-Nps consists of an intense peak corresponding to 67.42 % cerium and a strong peak corresponding to oxygen. Weak signals of C, S, and Cl elements in the EDX spectrum may be due to phytochemicals of the plant extract. These may be due to the emission of light by phytochemicals present in the plant extracts. Elemental composition shown in the EDX analysis supports the formation of *MP*-CeO_2-Nps, and is capped with phytochemicals.[10]

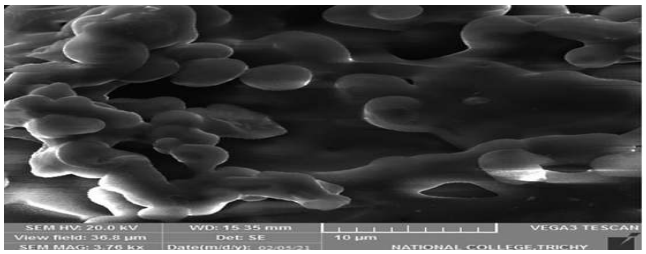

Fig.-3: SEM Image of Cerium Oxide Nanoparticles Synthesized using *M. pudica* Leaf Extract

Particle Size Determination

Figure-5 shows the XRD pattern of the *MP*-CeO_2-Nps. Structure and size of the *MP-CeO2-NPs* have been calculated using the Debye Scherrer eqn.-3.

$$D = \frac{k\lambda}{\beta \cos\theta} \qquad (3)$$

Where D is the crystalline size of the particles, K = 0.9, which is a dimensionless constant.; λ is the wavelength of the X-ray used for this analysis, β can be calculated from (FWHM), and Θ is the Bragg angle.

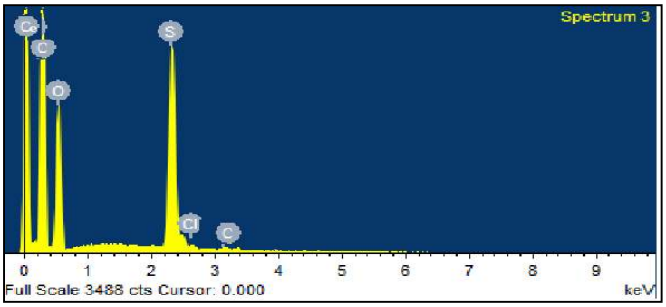

Fig.-4: EDAX Image of Cerium Oxide Nanoparticles Synthesized using M. pudica Leaf Extract

Table-1 gives the XRD data of *MP*-CeO_2-Nps. From the XRD data average size of the nanoparticles was calculated as 14.3 nm, which is in good agreement with the average size of *MP*-CeO_2-Nps in the DLS method as 12.9 nm (Fig.-6). X-ray diffraction pattern shows the peaks at 22.83°, 28.42°, 39.27°, 46.57°, 48.93° and 58.44°. Those peaks have been indexed to the crystal planes (110), (111), (211), (220), (311), and (222), which confirmed the face-centered cubic lattice (a= 54.113) with fluorite crystalline structure (standard data JCPDS 34-0394) of the *MP*-CeO_2-Nps.[11,12]

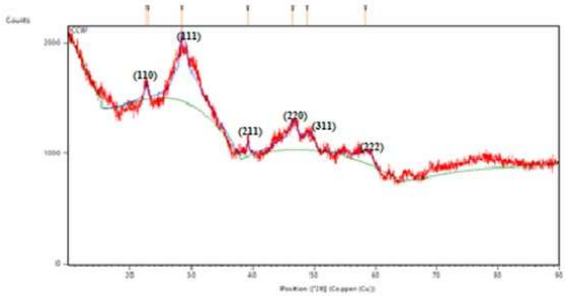

Fig.-5: XRD Pattern of Cerium Oxide Nanoparticles Synthesized using *M. pudica* Leaf Extract

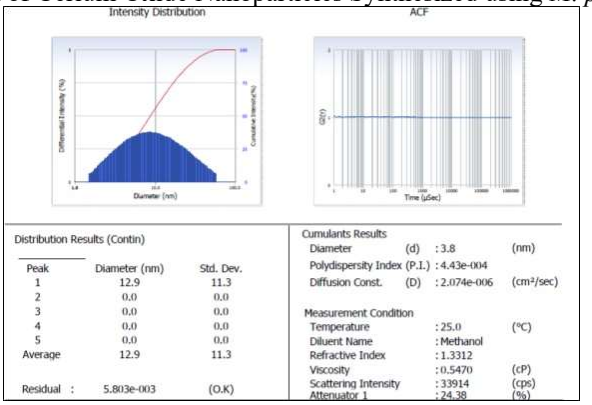

Fig.-6: DLS Analysis of Cerium Oxide Nanoparticles Synthesized using *M. pudica* Leaf Extract

Table-1: Cerium Oxide Nanoparticles Synthesized using *M. pudica* Leaf Extract

Pos. [°2θ]	Height [cts]	FWHM [°2θ]	d-spacing [Å]	Rel. Int. [%]	β(Ran)	Crystalline Size -D (nm)	Miller Indices (hkl)
22.8328	168.59	0.5816	3.8916	33.58	0.50	28.40	(110)
28.4216	502.08	1.2905	3.1378	100.00	1.10	13.10	(111)
39.2794	99.9	0.8217	2.2919	19.90	0.70	21.00	(211)
46.5774	184.47	2.0878	1.9483	36.74	1.79	08.40	(220)
48.9351	130.09	2.6329	1.6598	25.91	2.25	06.80	(311)
58.4404	98.84	2.5038	1.5779	19.69	2.14	07.40	(222)

Sonocatalytic Efficiency of MP-CeO2-NPs for the Degradation of Dye

The degradation of a 10 ppm concentrated aqueous solution of MO dye by ultrasonic irradiation was studied in the presence and absence of the catalyst *MP*-CeO_2-Nps under the same reaction conditions, and the

outcome of the experiments is shown in Fig.-7. The observations of the above experiments indicate that about 91% MO dye has been degraded within 120 min in the presence of 0.1 mg of *MP*-CeO_2-Nps at a solution pH of 5.0. Whereas in the absence of MP-CeO-NPs, only 13 % of the dye has been degraded within the reaction. Degradation of MO dye by sonication alone may be due to the chemical effect of ultrasonic sound, leading to the splitting of the water molecules and producing hydroxide radicals. The hydroxide radicals were able to degrade the organic dye by oxidative decomposition. But the number of hydroxide species produced by sonication alone is less. Formation of reactive oxygen species (ROS) such as oxide, hydroxide, and peroxide radical has been enhanced by sonication of water in the presence of a suitable semiconducting catalyst. This may be due to the growth of a great number of cavity bubbles on the semiconductor surface of the nanoparticles.[13,14,15] As per the MKY mechanism, the organic pollutants were oxidized on the surface of cerium oxide nanoparticles using the lattice oxygen, and hence oxygen vacancy is created in cerium oxide nanoparticles.[16,17] This phenomenon increases the number of active sites on the surface of the cerium oxide and enhances the splitting of water to produce a greater number of ROS pieces. Therefore, the degradation efficiency of the MO dye in the presence of *MP*-CeO_2-Nps is enhanced than in the absence of the catalyst.

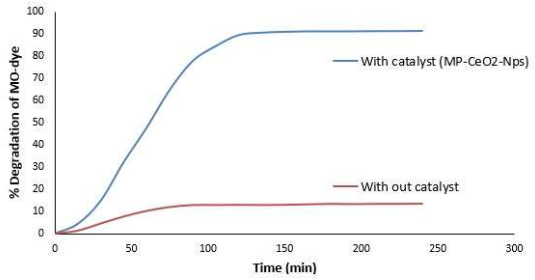

Fig.-7: Efficiency of Cerium Oxide Nanocatalyst for the Sonolytic Degradation of Methyl Orange Dye

Effect of pH on the Degradation of Dye

The adsorption capacity of the catalyst was the key factor that determined the rate of degradation of the dye by ultrasonication.[16] To determine the effect of pH on catalytic ability of catalytic efficiency of *MP-CeO_2-Nps, the reactions were carried out at* different pH levels of the medium. 100 ml of methyl orange solution of initial concentration 10 ppm was agitated with 0.1 mg of *MP-CeO-Nps*. The reaction mixture was signalized with ultrasonic waves for 120 minutes at different initial pH levels of the solution in the range 2 to 12. Figure-8 shows the effect of pH on the degradation of methyl orange dye by sonolysis in the presence of *MP-CeO-Nps*. Maximum degradation of the dye was found at pH 3.0, then it decreases with an increase in pH of the solution. Maximum degradation at low pH can be attributed to electrostatic absorption of anionic MO dye on the electropositive surface of the cerium oxide nanoparticles. A large number of dye molecules absorbed on the surface of the semiconductor nanocatalyst were exposed to a greater number of ROS species.[17] Therefore, the amount of degradation of dye molecules also increased. In this present study, about 91% methyl orange dye was degraded at Ph 3.0 in the presence of 0.1 mg of *MP-CeO-Nps* by the sonication method. While at higher pH efficiency of degradation if found to decrease to 20 % at pH12.0. This may be due increasing pH of the solution beyond pH 6.2, the surface of the nanoparticles became negatively charged. Consequently, the repulsion between the negatively charged dye and the surface of the nanoparticles reduces the absorption phenomenon, resulting in decreased dye removal through sonolysis.[16,17]

CONCLUSION

Discharge of industrial waste introduces a huge amount of toxic organic and inorganic pollutants into the freshwater, which cause serious health problems to humans and other living beings. How to remove toxic organic pollutants is the main research focus of the present study. In this work, simple, cost-effective techniques was adopted to remove carcinogenic organic dye, which is sonolysis. Semiconductor nanoparticles MP-*CeO-Nps* was synthesized by the green method using *Mimosa Pudica plant* extract and which was used as a catalyst to accelerate the degradation of dye under sonication of the solution.

The efficiency of MP-CeO-NPs as a catalyst for the degradation of MO dye at different contact times and pH of the medium was investigated. The experimental results reveal that 0.1 mg of MP-CeO-Nps can

effectively remove 91 % methyl orange dye in aqueous solution at pH 3.0 within the sonication time of 120 min. The efficiency of the MP-CeO-Nps catalyst is about 6 times greater than that of the degradation of dye without the catalyst under sonication process.

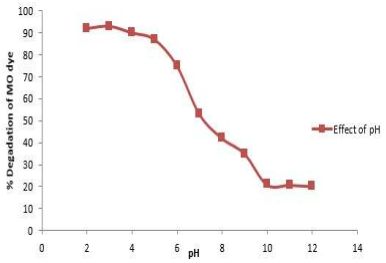

Fig.-8: Effect of pH on Sonolytic Degradation of Methyl Orange Dye in the Presence of CeO-Nps

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG 6: Clean Water and Sanitation*. All the authors contributed significantly to this 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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