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GREEN SYNTHESIS AND CHARACTERIZATION OF CERIUM OXIDE NANOPARTICLES USING *Piper Betle* Leaves.

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ABSTRACT

Cerium Oxide Nanoparticles were synthesized by using *piper betle* leaf extract. The UV absorption spectrophotometer analysis of *piper betle* leaf extract and metal compounds showed absorbance spectra in the range of 300-350 nm. The UV-Vis spectroscopy shows surface Plasmon resonance of CeO₂ nanoparticles at 310 nm indicating the particles are poly dispersed. The FT-IR measurements were carried out to identify the formation of Cerium oxide nanoparticles and possible molecules such as OH, C=C, C=O, and aromatic compounds. The FT-IR analysis played a vital role in displaying the nanoparticles which showed strong absorbance in the range 671.05cm⁻¹ for Cerium oxide nanoparticles. The diffraction peaks and planes of ions are indexed. From the 'X' ray diffraction studies, the size of the Cerium oxide nanoparticles is 27 nm as calculated by using Debye Scherrer's equation. The size, shape, and structure of nanoparticles were also analyzed by scanning electron microscope which shows an almost spherical shape of nanoparticles aggregation. This Eco-Friendly synthesis method has many advantages over chemical methods because it reduces the use of toxic metals in the synthetic process.

Keywords: Piper Betle Leaves, Leaf Extract, Green Synthesis, Cerium Oxide Nanoparticles, Characterization.

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INTRODUCTION

The field of nanotechnology is one of the most modern research areas of information science. Nanotechnology field has become a very rapid development as well as the applications in science and technology are designed to create new materials at the nanoscale. Nanoscale materials exhibit unique electrical, magnetic, optical, catalytic, and chemical properties compared to conventional materials and products.¹

This is due to quantum size effects and large surface-to-volume. Biosynthesis of nanoparticles is a bottom-up process and the reactions that occur are reduction and oxidation. Phytochemicals with antioxidant and reducing properties are generally responsible for reducing metal compounds in the preparation of nanoparticles. Bio organism is an environmental protection agent that can be used as a capping agent. Nanoparticles were produced due to the presence of phenols, ethers, hydroxyl groups and amides in the leaf extract. Cerium oxide (CeO₂) nanoparticles are widely used due to their unique surface chemistry, high stability, and biocompatibility² when compared to other nanoparticles.

It is often used in the production of sensors, cells, catalysts, medical, drug delivery, and anti-inflammatory drugs. CeO₂ nanoparticles have antioxidant activity. CeO₂ nanoparticles can be used as new drugs against various microbial diseases. Current methods are used to prepare CeO₂ nanoparticles and these methods have their own limitations. It is aimed to make the connection more effective, economical, and soft by using CeO₂ nanoparticles. In India, the leaves of the *betle* are called Paan. Research on the leaf extract of *piper betle* has shown that it has many pharmacological benefits, such as antioxidant, antibacterial, and anti-inflammatory properties. There are various studies related to this research, such as the green synthesis of Fe₂O₃ nanoparticles using *betle* leaves and their characterization⁴ as well as the synthesis, characterization, and antibacterial activity of copper oxide nanoparticles.

The green synthesis process is one of the most suitable methods for the synthesis of cerium oxide nanoparticles because it is cost-effective, environmentally friendly and easy to synthesize on a large scale. This project aims to produce cerium oxide nanoparticles in a green biogenetic way. It will find important and special uses. Detailed information about the features of the new model is discussed.



EXPERIMENTAL

Materials

Piper betle leaf extract was used as a reducing agent in the synthesis of cerium oxide nanoparticles. Cerium nitrate solution is also used as a precursor for the preparation of cerium oxide nanoparticles.

Preparation of *Piper Betle* Leaf Extract

The new leaf of *piper betle* leaf is collected locally. 10 grams of fresh leaves is washed thoroughly under running tap water and chopped finely. 50 ml of distilled water is added to a 250 ml beaker and cooked for 10 minutes. The extracts were used for the synthesis of CeO₂ nanoparticles.

Green Synthesis of Cerium Oxide Nanoparticles

0.5~M cerium nitrate solution was added with 30 ml of water. 20 ml of *piper betle* leaf extract is added to the above solution and stirred for 10 minutes. Then 0.5~ml H₂SO₄ (5M) was added to the above dispersion. The suspension was transferred to a 50 ml Teflon-lined stainless-steel autoclave. The autoclave was preheated to 2000 °C and heated for 3 hours and then cooled to normal room temperature. The precipitate was centrifuged, washed with water, and calcined at 300 °C for 2 h to obtain cerium oxide nanoparticles.⁶

Characterization

Characterization of the synthesized green cerium oxide nanoparticles by XRD model using the BRUKKER binary V3 (original) model. The surface morphology and elemental analysis of the synthesized samples were examined by scanning electron microscopy using the MAKE - Zeiss model - EVO18. The absorbance spectrum of the synthesized sample is measured by a UV-visible spectrophotometer at a wavelength of 300-350 nm. FTIR spectra of synthetic samples in the range of 500 - 4000 cm⁻¹ were recorded in the FTIR Perkin Elmer model.

RESULTS AND DISCUSSION

The reduced form of cerium oxide nanoparticles was obtained and monitored by measuring UV-visible light using a UV-visible spectrophotometer. The UV-visible light absorption peaks of nanoparticles of different sizes arise from the physical process of surface plasmon resonance and record the UV-visible spectrum of the reaction. The absorbance peak was observed at 310 nm. This demonstrates the development of CeO₂ nanoparticles. UV spectroscopy can analyze cerium oxide nanoparticles of controlled size and shape. Figure-1 shows the UV spectrum recorded in the reaction medium.

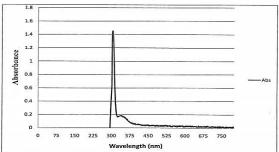


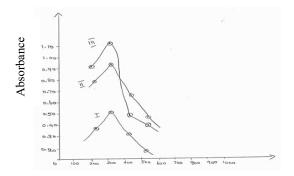
Fig.-1: the UV Spectrum Recorded by The Reaction Medium

Stability of Cerium Oxide Nanoparticles

The absorption peak of CeO₂ nanoparticles is observed at 310 nm. The absorption rate of the CeO₂ nanoparticle preparation was tested on the 1st day, 15th day, and 30th day. There was no significant change in peak area in 30 days. This confirms the stability of CeO₂ nanoparticles as shown in Table-1.

Table-1: The Stability of CeO₂ Nanoparticles

Table-1. The Stability of CeO ₂ Nanoparticles			
Wave	Absorbance		
Length(nm)	1st day	15 th day	30th day
210	0.37	0.79	0.90
310	0.50	0.93	1.10
410	0.30	0.65	0.45
510	0.20	0.45	0.40



Wavelength (nm)
Fig.-2 Stability of Cerium Oxide Nanoparticles

The FTIR spectrum of cerium oxide nanoparticles is shown in Fig.-3. The strong and broad peak near 671 is due to the vibration of O-Ce-O. Other absorption peaks are found due to other organic molecules such as OH, CH, C-O, C-C, C=O, C-N, C=C, and alkenes.

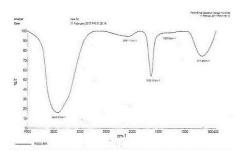


Fig.-3: The FTIR Spectrum of Cerium Oxide Nanoparticles

In the present work, the prepared CeO₂ nanoparticles were characterized by XRD studies. Particle size was determined from the width of XRD peaks using Scherrer's formula⁷,

$$d = \frac{(0.94 \ \lambda)}{(\beta \cos \theta)}$$

where β is the full-width half maximum, θ is the diffraction angle, d is the average crystalline grain size and λ is the wavelength of the 'X' rays

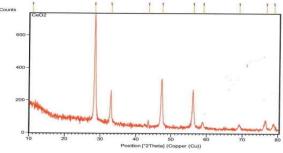


Fig.-4: The Diffraction Pattern of CeO₂ Nanoparticles

The diffraction pattern of CeO₂ nanoparticles is shown in Fig.-4. The most common crystal behavior of CeO₂ is cubic. The maximum density of CeO₂ nanoparticles increases due to their quantum size effect. The average particle size is 27 nm by the Scherrer equation. The exponential diffraction peak at 2θ for the synthesized CeO₂ sample is 5.435 A⁰. Positions of 28.43⁰, 33.62⁰, 48.38⁰, 57.74⁰, 59.03⁰, 69.37⁰, 76.69⁰ and 79.09⁰ correspond to (111), (200), (220), (311), (222), (400), (311) and (420) XRD pattern (Fig.-4) shows four main orientations (111), (220) and (311) properties of CeO₂ cubic phase structure. The unassigned peak could be due to the presence of crystallization of the bioorganic phase that occurs on the surface of the nanoparticles. The size, morphology and distribution of the synthesized green CeO₂ nanoparticles were observed by SEM. The SEM image of CeO₂ nanoparticles is shown in Fig.-5, which shows that the particles are well dispersed and almost circular. The size of nanoparticles is about 27 nm and when a nanoparticle is combined it shows large nanoparticles. This

aggregation occurs due to cellular material on the surface of the nanoparticles and acts as a capping agent. The white color of the surface indicates that the shape of the pores in the SEM image is almost spherical. Particle distribution is slightly uneven. There are very few differences between the products.

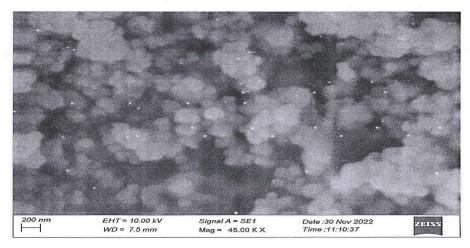


Fig.-5: SEM Image of CeO₂ Nanoparticles

CONCLUSION

Cerium oxide nanoparticles have been successfully prepared using *Piper betle* leaf extract, this method is simple, environmentally friendly, has good compatibility, provides a large-scale process, and less time. The leaf extract serves as a capping and reducing agent in nanoparticle synthesis. UV absorption spectrophotometric analysis of CeO₂ nanoparticles produced by the lobes found a band at 310 nm identified as the surface plasmon resonance band, which was identified as excited valence electrons. FTIR analysis showed high performance and stability of CeO₂ nanoparticles. The FTIR spectrum shows a strong absorption of CeO₂ at 671 cm⁻¹. Based on XRD study, the size of the nanoparticles was calculated as 27 nm using the Debye Scherrer equation. The shape of the nanoparticles was analyzed by scanning electron microscopy and the CeO₂ nanoparticles were found to be highly dispersed and nearly spherical. Green synthesis of nanoparticles is an evolution of nanotechnology. Biological CeO₂ nanoparticles have attracted interest in biomedicine and other fields due to their unique surface morphology, small crystal size, and biocompatibility. For example, it is used as an antibiotic and antioxidant in the treatment of various cancers. Therefore, the synthesis, characterization, and application of metal nanoparticles in foreign countries have become an important trend in modern science. CeO₂ nanoparticles are widely used in the medical field.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

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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