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Biogenic Synthesis of Cerium Oxide Nanoparticles (CeONPs) Using *Ocimum basilicum* leaves (Basil) and its Photocatalytic Applications

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ABSTRACT

Nanotechnology is most recent, and emerging science based upon physical and chemical methods and green synthesis. The formal methods of makings nanoparticles (NPs) are costly, toxic, and not environmentally friendly, so the need of time is to adopt non-convectional approach for the synthesis of NPs. The alternate and most comprehensive approach is “Green Synthesis” due to its superiority on conventional. Green synthesis involves the use of green materials such as, plant extracts, microorganism etc. to synthesis nanoparticles. This technique avoids usage of hazardous reagents, solvents, and harmful technologies; therefore, it is environment friendly and even economically beneficial. It even eliminates the costs associated with purification stages. Green synthesis is generally performed in aqueous medium, due to its biocompatibility and other advantageous features. Thus, green synthesis is most eco- friendly, non-toxic, and economic synthesis of nanoparticles. In this study, cerium nanoparticles (CeONPs) were prepared by using leaves extract of *Ocimum basilicum* and its photocatalytic activities were determined. Moreover, NPs were characterized by different analytical techniques like Infrared Spectrometry (FTIR), Scanning Electron Microscopy (SEM), and X-ray diffraction (XRD). Characterization of the NPs revealed that FTIR spectrum transmission peak at 3690 cm^-1 corresponding to the stretching mode of O-H bonds, indicating the presence of alcohol functional groups. A peak at 2250 cm^-1 indicated weak bonding of CΞN, characteristic of nitrile compounds. SEM characterization demonstrated wide pore spaces on the surface of the CeONPs, making them promising for the remediation of Synozol dyes. EDX analysis confirmed the presence of carbon (C), oxygen (O), and cerium (Ce) in the CeONPs, further confirming their successful synthesis. The X-ray diffraction pattern indicated specific miller indices associated with the crystal structure of CeONPs. The spectrum analysis indicated that the highest absorbance occurred at a wavelength of 225 nm, while the lowest absorption peaks were observed at 430 nm. A minor shift in the peaks was observed at 430 nm. It was evident that the absorption at both wavelengths was reduced compared to the control and light conditions. It is suggested that the prepared CeONPs can be used for photocatalytic

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degradation/ remediation of dyes.

Keywords: Green synthesis, CeONPs, *Ocimum basilicum*, Photocatalysis, Nanoparticles characterization

Introduction

Nanotechnology is most recent, fast expending, multidisciplinary, and emerging area of research with broad ranges of applications in science, photonics, medicines, and technology (Malik et al., 2023). The linking of the unique characteristics of molecular and atomic groupings produced at the level of nanoscale , nanotechnology has attained the position of one of the essential scientific events of the start of 21st century (McNeil, 2005).

It deals with synthesis, characterizations, and applications of several of noble metals nano-particles (NPs) (Habibullah et al., 2021). Applications of nanotechnology in food production include transport and encapsulation of substances in selected places, enhancing taste, nutritional presenting by antibacterial based nanoparticles , development of lifespan, detecting impurity, better food items storing, outlining and security of brand (Chellaram et al., 2014). Nanotechnology field has also actual viable potential for the fiber industries. It is generally because of the fact that traditional approaches utilized to convey varying characteristics of textiles frequently which is not lead to enduring properties, will damage their purposes after wearing or laundering (Wong et al., 2006). Metals nano-particles are numerous small in size ranges of 1-100um (Ketchum, 2005). Numerous methods are working for the metallic nanoparticles formation, which is considered into two core kinds as top down and bottom up methods reliant on initial material of nanoparticle formation (Jamkhande et al., 2019). The distinctive characteristics exposed by the atoms at diverse crystal sites in a nanoparticle are due to their quantum effects or surface. Therefore, numerous unusual nanomaterials are being established by investigators with novel applications and properties (Jeevanandam et al., 2022).

However, these methods are expensive, laborious, and potentially dangerous to the environment and human health. Therefore, it is pressing need to develop a most comprehensive, scientific, compact, and non-conventional approach for the synthesis of nano-particles (NPs) (Anbu et al. 2020). Conventional methods have been employed for many years, but studies have demonstrated that green techniques are more effective in producing nanoparticles (NPs) due to their lower likelihood of failure, cost-effectiveness, and ease of characterization (Gour & Jain, 2019). Green synthesis called is alternative methods for the synthesis of nano-particles (Ijaz et al. 2020).

Green nanotechnology is more environmentally friendly, cost effective, safer, easy handling, and less requirement of material. Furthermore, green nanotechnology (biological approach) is evident way to decrease environmental impacts, risk impact to human, and cost for nanomaterial preparations (Khan, 2019).

The formation of composite materials by plants has more advantages in comparison to physical or chemical objects. The stable bonding between nanoparticles and organic material is very different in quality from the stable composite. Moreover, composite may also release nanoparticles during the process of adsorption, which can cause

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toxicity in water (Siddiqui et al., 2019). Nanoparticles formed by plants are more established and the amount of production is more rapidly than microorganisms. Moreover, nanoparticles synthesized through plant-based methods exhibit a greater variety in terms of size and shapein contrast to those made by other living things (Iravani, 2011).

Basil (*Ocimum basilicum*), which is the ‘‘king of the herbs’’, is very significant aromatic plants and vegetables which grown-up globally for its industrial properties and medicinal, flavoring. The economically valuable segments of the plant containing seed, flowers and leaves (Abbasifar et al., 2020). Numerous studies are synthesized nano-particles by using various plants extract (Chand et al., 2020). However, the use of Basil (*Ocimum basilicum*) is very limited. It is a very vital crop that contains various natural metabolites such as, essential oils, polyphenols, flavonoids, phenols, and phenolic acids. This forever green plant belongs to the mint family. Basil leaves also have enormous pharmaceutical interests. Against some infections that are caused by fungi, bacteria and viruses, basil has been shown to have beneficial effects. It is utilized for the treatment of coughs fevers ,bronchitis ,asthma, and flu diarrhea (Shahrajabian et al., 2020).

CeONPs (cerium oxide nanoparticles) are gaining significant attention within the research community due to their growing applications in catalysis. The biological synthesis of CeONPs holds great potential for unveiling their remarkable photocatalytic properties in the removal of various industrial pollutants, including dyes (Walkey et al., 2015). Another contribution factor in CeONPs toxicity is the shape. Calculated consequences have resolute that the quantity of nanoparticles transportation can speed up to 60 orders of degree according to their surface charge and shape (Milani et al., 2017). Numerous approaches for the production of cerium oxide nanoparticles have been advanced such as sol-gel, hydrothermal, and thermal hydrolysis, and spray pyrolysis, ball milling vacuum deposition techniques, thermal decomposition and solvotherma, precipitation (Fifere et al., 2021).

Textile industries are using various types of toxic dyes, but Synozol dyes are the most hazardous, cancer-causing, and fatal due to presence of two functional reactive groups, including triazine, and vinyl sulfon group. The presence of the vinyl sulfone functional group increases its reactivity, toxicity, and solubility in water. Synozol dyes, also called azo reactive dyes, are a group of dyes that are most commonly present in conventional textile wastewater because of their reactiveness and solubility; As a result, they exhibit minimal affinity for adsorption onto biomass and typically pass through an activated sludge system (Alsukaibi, 2022).

The objectives of our study are:

Biosynthesis of cerium oxide nanoparticles (CeONPs) using anhydrous cerium salt and leaves extract of *Ocimum basilicum*

Photocatalytic application of synthesized NPs

Characterizations of nanoparticles with different analytical techniques (UV-Vis, FT-IR, SEM and XRD)

Methodology

The experiments were conducted at COMSATS University Vehari Campus for the synthesis of nanoparticles and its application for sorption of textile dyes in

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wastewater.

Biosynthesis of CeONPs

The Cerium oxide nanoparticles (CeONPs) were made by the leaves of *Ocimum bacillum* (basil plant) which were air dried at room temperature. The leaves were collected from nursery and washed carefully with double ionized water to eradicate the sticky dirt, particulate matter. Dried leaves were crushed into fine powder and soaked into 1:10 DW. Solution was placed onto the magnetic stirrer at 80 ^{o}C for 4hours on continuous stirring. Then cerium sulphate solution (3g/100ml) will be added into the plant extract after that solution was centrifuged to separated particles at 3000 rpm for 20 minutes. The centrifuged solution was concentrated until dark colored paste formed. The paste was dried in an oven and then placed in ceramic crucible and roasted in a furnace for specific period of 2 hrs. At 450^{o}C to obtain CeONPs.

Figure 2.1: (a) Basil Plant (b) Basil Plant Powder**.**

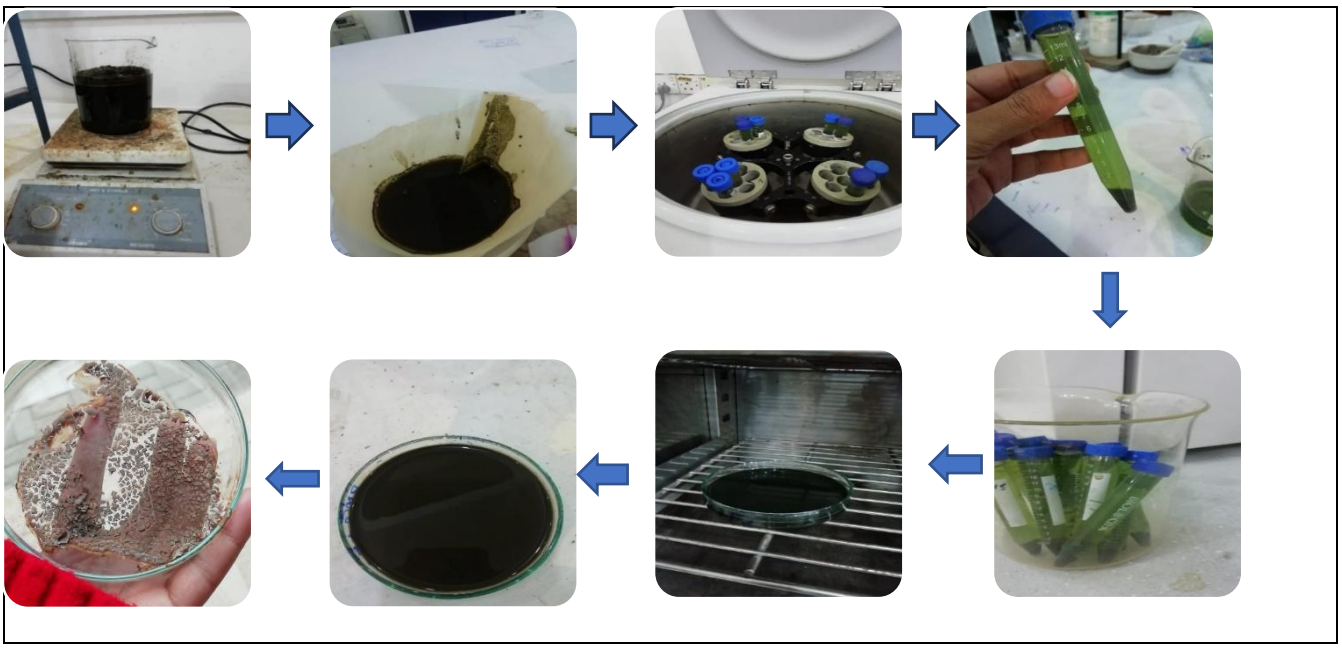

Figure 2.2: Experimental Steps for the pre

Characterization of CeONPs

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paration of OB/CeONPs and CeONPs
Following analytic techniques were adopted to characterize the synthesized CeNPs.

UV visible spectroscopy
This method is based on Beer-Lambert law in this surface plasmon resonance (SPR) is formed between the wavelength (abscissa) and absorbance (ordinate). The SPR is the properties of nanomaterial as well as the molecular entity. The copper oxide nanoparticles have SPR band at 531 nm which is characteristic property of this material.

FT-IR spectroscopy
The surface functional groups of the biosynthesised nanoparticles will be investigated using FT-IR spectroscopic analysis. The possible biomolecules that act as reducing, stabilising, and capping agents during the synthesis of cerium oxide nanomaterial are identified with the aid of this analysis. During the production of phyto-mediated CeONPs, the phytochemicals obtained from the leaf extract are essential as reducing, capping, and stabilising agents.

Scanning Electron Microscopy
A key technique will be used to determine the size and surface shape of the nanoparticles. To create a picture, the surface of the nanomaterial will be exposed to an electron beam. The beam's electrons interact with the sample to produce various signals that reveal important details about the composition and topography of the surface.

X-Ray Diffraction
XRD patterns of the prepared CeNPs will be evaluated with XRD. To find the size of the CeONPs crystals, the Debye-Scherer condition equation (Eq. 1) will be utilized.

$$D = K \lambda/\beta \cos\theta \quad \ldots\ldots\ldots\ldots\ldots (1)$$

Where D = Crystalline size of NPs.
K= Scherer Consistent with an incentive 0.9 to 1.
Λ=Wavelength of X-beam (0.1541) used in XRD.
β =Full width which is a half limit of the diffraction pinnacle.
Cosθ = Bragg's

Photocatalytic Activities
The photocatalytic activities of synthesized (CeONPs) under visible light irradiation will be done for the degradation of Synozol dyes. A photochemical reactor made up glass will be used for the experiment, attached with UV lamp and sample will be provided atmospheric oxygen with help of an oxygen pump. The entire experiment will be conducted at 25°C. A 300 ml dye solution will be combined with 0.3 g of the sample. To achieve adsorption/desorption equilibrium between the sample and the dye, the photo catalyst and dye suspension will be agitated prior to irradiation without the use of UV light. A 5 ml piece of the mixture will be taken out of the reactor at

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regular intervals after 15 minutes, and the absorbance will be measured to track the dye's decolorisation. The following formula will be used to determine the deterioration efficiency. Degradation efficiency (%) = $\left(\frac{C_0 - C_t}{C_0}\right) \times 100$

In the above equation
Initial concentration = C_0 C_t = Represent the concentration at time‘t’.

Results

Characterization

Figure 3.1 displays the Fourier Transform Infrared (FTIR) spectra that were acquired in the wavenumber range of 4,000 cm-1 to 300 cm-1. The stretching mode of O-H bonds was found to be responsible for the transmission seen at wavenumber 3690 cm-1 in the Fourier-transform infrared (FTIR) spectra of CeONPs. This peak's sharp form suggests that alcohol functional groups are present. The stretching mode linked to the weak bonding of C–N, which is typical of nitrile compounds, is represented by the peak at wavenumber 2250 cm^-1. The bending of C-H bonds in the alkane group can be linked to the observed peaks at wavenumber 1451 cm^-1, indicating medium bond strength.The spectra found at the wavenumber of 995 cm^-1 are indicative of the presence of functional groups associated with C=C bending and alkane groups, while the peaks observed at the wavenumber of 1185 cm^-1 can be linked to the stretching of the C-O bond in the ester group. Figure 3.2 shows the SEM characterisation of CeONPs. The figure clearly shows that the prepared particles had wide pore spaces on the surface of the CeONPs, suggesting that the materials may be useful for the remediation of Synozole dyes.Energy-Dispersive X-ray (EDX) analysis was used to determine the chemical composition of CeONPs; the findings are shown in Figure 3.3. As shown in Figure 3.3, the EDX analysis of CeONPs indicated the existence of the elements carbon (C), oxygen (O), and cerium (Ce). The presence of carbon (C), oxygen (O), and cerium (Ce) in the produced nanocomposites attests to the successful green synthesis of CeO nanocomposites employing Ocimum bacillum.

CeONPs' X-ray diffraction pattern was examined between 0° and 80° (Figure 3.4).The recorded peaks at 22°, 27.50°, 34.50°, 53.5°, and 74° were associated with the miller indices (hkl) values of (111), (220), (311), (400), and (422), respectively.

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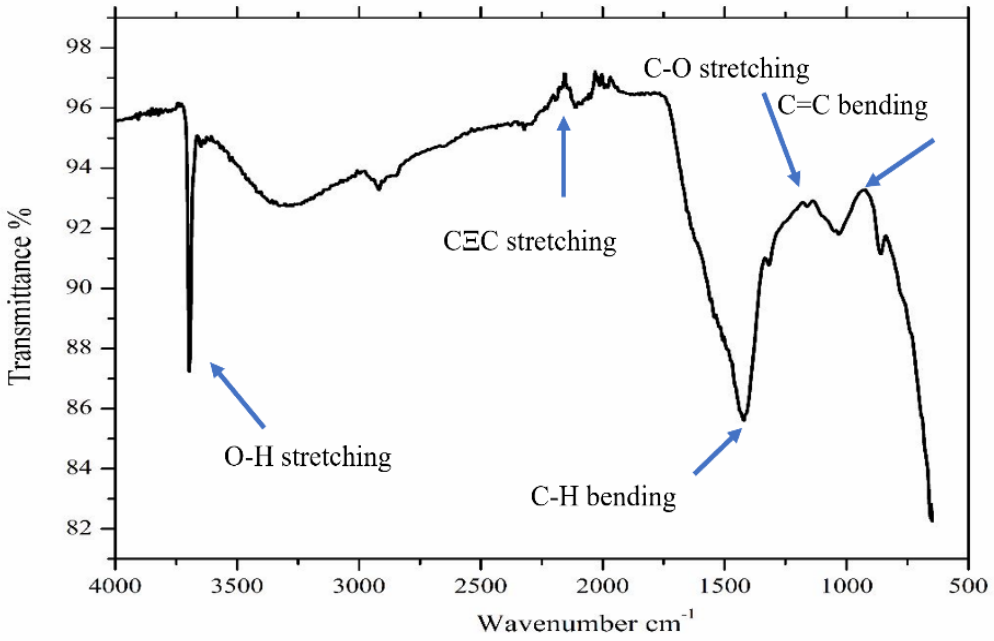

Figure 3.1: FTIR spectra of CeONPs

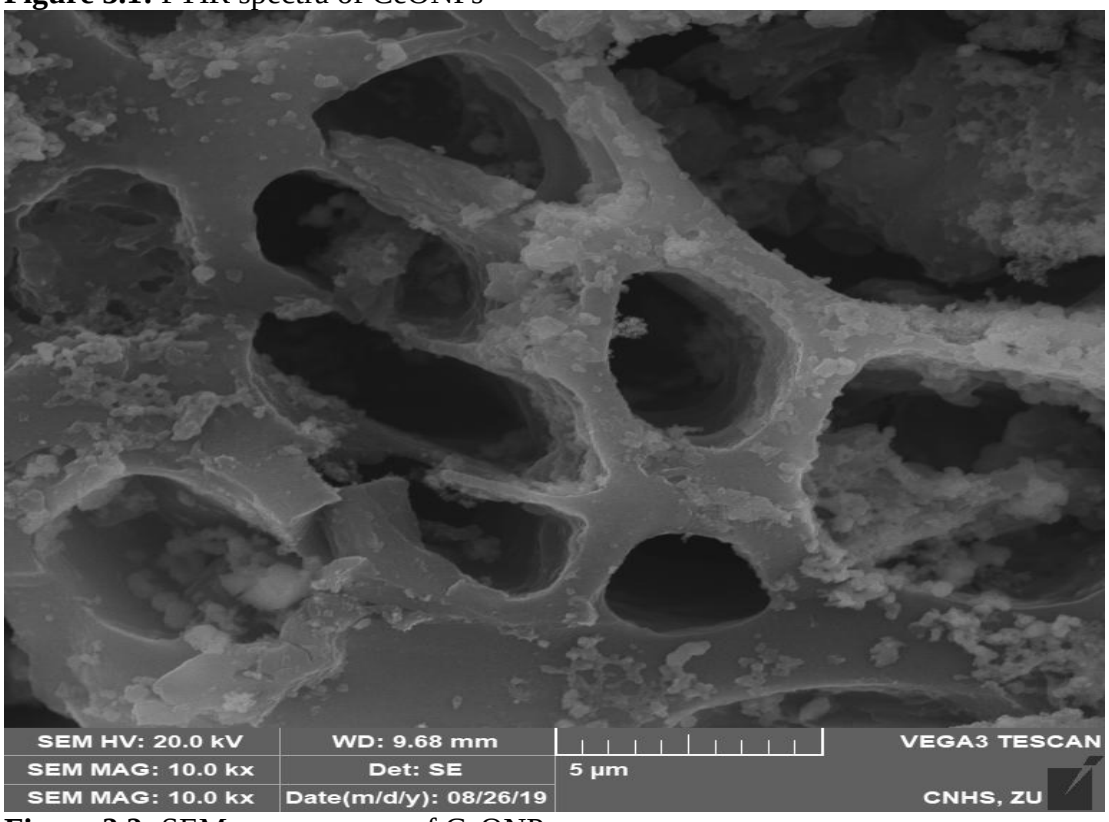

Figure 3.2: SEM spectroscopy of CeONPs

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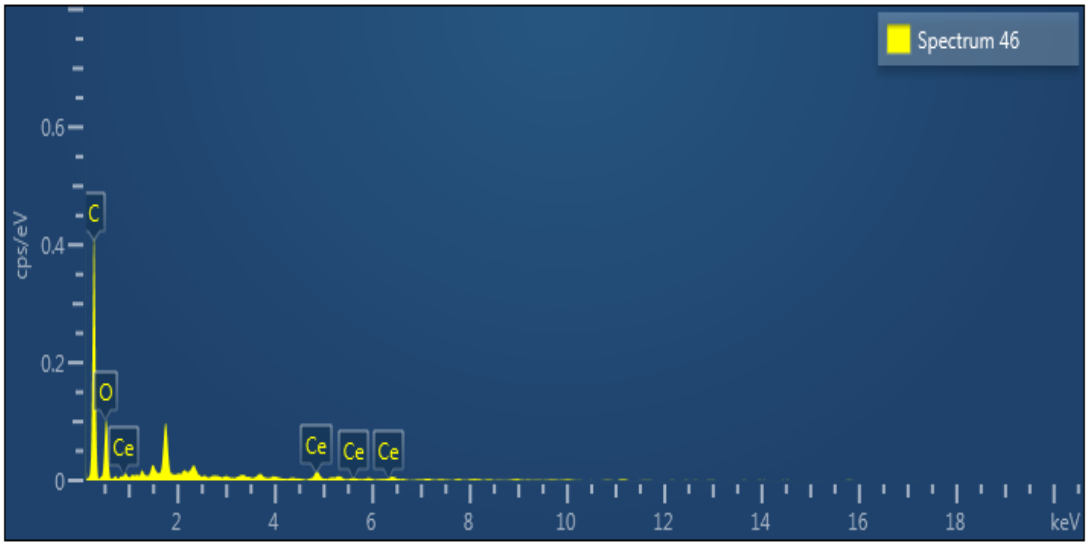

Figure 3.3: EDX spectra of CeONPs

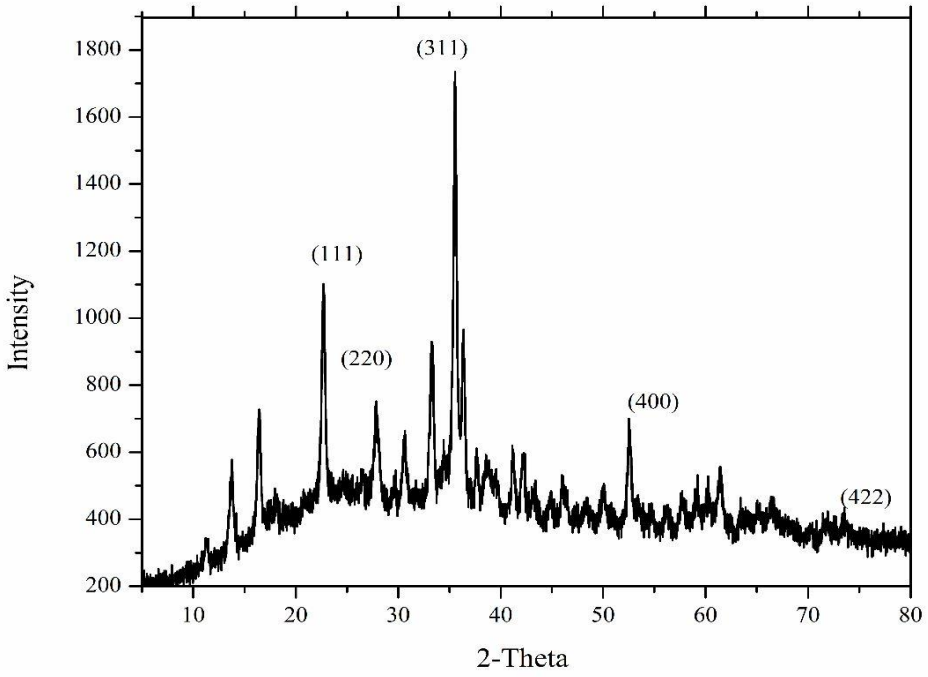

Figure 3.4: XRD spectra of the of CeONPs

Energy Dispersive X-ray analyzer (EDX)

Energy Dispersive X-ray spectroscopy (EDS), also known as EDX or EDX, is a well-established electron microscopy technique used for elemental analysis. Despite some attempts at improvements, the practice and application of EDS have remained largely unchanged in comparison to electron energy loss spectroscopy (EELS). EDS is widely regarded as the standard and reliable method in the field of analytical electron

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microscopy (Shindo et al., 2002). The technique utilizes the generation of characteristic X-rays to identify and analyze the elements present in the specimens, making it a valuable tool for elemental analysis in electron microscopy.

EDX microanalysis is widely utilized in various biomedical fields by researchers and clinicians; however, its potential applications are not fully recognized by the scientific community. The spectrum obtained from EDX microanalysis provides both semi-qualitative and semi-quantitative information. This technique proves valuable in drug-related studies, such as investigating drug delivery systems where EDX serves as a crucial tool for detecting nanoparticles commonly used to enhance the therapeutic efficacy of certain chemotherapeutic agents. Additionally, EDX finds application in studying environmental pollution and characterizing mineral accumulation in tissues. In summary, EDX is a valuable tool for determining both endogenous and exogenous elements in tissues, cells, and other types of samples, making it useful in a wide range of scientific investigations (Scimeca et al. 2018).

Micro-analysis of the elemental composition of OB/CeSO4 were performed by using energy dispersive X-ray spectroscopy (EDX). We calculated that the weight percentage of the carbon was highest in OB/CeSO4 61.45%, weight sigma was 2.92% and atomic percentage was also highest as 70.55% on K series. Oxygen weight percentage 33.61%, weight sigma 2.96% and atomic percentage 28.97% were reported on K series in OB/CeSO4.Cerium weight percentage on L series4.93%, weight sigma percentage 1.16% and atomic percentage 0.49% on L series in OB/CeSO4 were detected by EDX as shown in figure 3.8.

Fourier Transform Infrared Spectra(FTIR)

Fourier Transform Infrared (FTIR) spectroscopy is an analytical method that is used to determine functional groups and chemical composition, which involves measuring the absorption of infrared radiation using a sample. The spectrum obtained gives qualitative and quantitative data of the structures and bonds of molecules (Dutta, 2017).

FTIR spectrum of OB/CeSO 4 was measured within the wavelength of 500-4000 cm -1(Fig. 3.3). The wide peak at 3700 cm to -1 is a representation of O -H groups of proteins, polyphenols and carbohydrates. That NH stretching of amines represents N–H stretching of amines by the peak close to 2800 cm -1. The 1400 cm-1 is the absorption of C-O and C-C bonds whereas, the 1500-1900 cm -1 of absorption is C=C bonds of unsaturated compounds. The highest peak of about 800 cm -1 is associated with alkyl amines.

X-ray diffraction

X-ray diffraction (XRD) is a non-destructive method of analysis of crystalline material that is usually used. It gives useful data regarding crystal structure, phase composition, grain size, strain, crystallinity, and crystal defects. Constructive interference by certain crystal planes gives a number of diffraction peaks when a beam of monochromatic X-rays interacts with a crystalline sample. The formation of these peaks forms a typical structure to show the arrangement of atoms in the material (Bunaciu et al., 2015).

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XRD finds wide use in identifying unknown crystalline material and establishing structural characteristics in many disciplines including materials science, pharmaceuticals, geology, environmental science, and engineering. It is also applicable in deciding unit cell sizes, determining minerals, describing crystalline phases, and determining the purity of the sample (Fatimah et al., 2022).

Scanning Electron Microscope (SEM)

Scanning Electron Microscopy (SEM) is an effective method of observation of the surface morphology and microstructure in substances. SEM is the method where a beam is focused on the surface of the sample and the interaction between the sample and the beam causes the scattering of the electrons. These are picked up and translated into high-resolution images that display the structure and composition of the surface (Mahltig & Grethe, 2022). The intensity of the detected electrons of distinct points on the sample surface is linked to the brightness of each pixel in the SEM image. SEM finds extensive application in materials science to examine the surface properties, particle size, and structural information (Inkson, 2016; Ural, 2021).

Photocatalytic Activities

Absorption in control

Absorption peaks of Synozol dyes were determined prior to the addition of catalyst and UV light ranging from 200 – 700 nm wavelength. The peaks were measured after 15 mins shown in figure 3.5. The spectrum reveals that the highest absorbance was observed at 495 nm wavelength. However, other peaks were observed at 355 nm. After each time interval there was no significant change was observed in the Spectra.

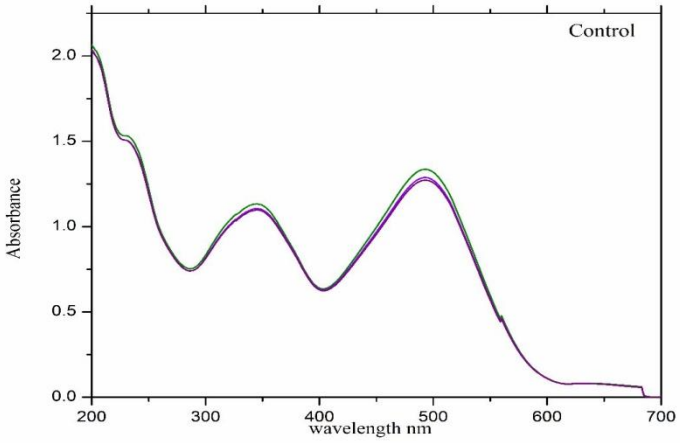

Figure 3.5: Absorbance spectroscopy of Synozole dye at 200-700 nm without any treatment

Absorption of dyes in the presence of light

Absorption peaks of Synozole dyes were determined in the presence of light. The peaks were measured after 15 mins shown in figure 3.6. The spectrum reveals that the highest absorbance was observed at 225 nm wavelength. and the loest absorption

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peaks were observed at 430 nm. After each time interval there was no significant change was observed in the Spectra at 225 nm of wavelength. However, a minor shift in peaks was observed at 430 nm.

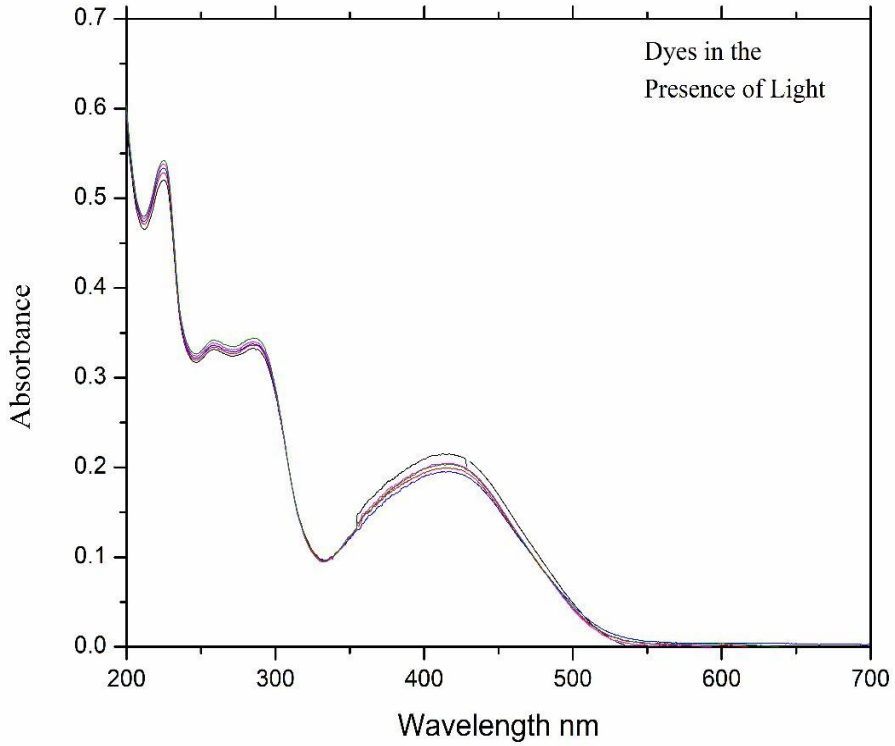

Figure 3.6: Absorbance spectroscopy of Synozole dye at 200-700 nm in the presence of light

Absorption of dyes in the Acidic Media

The absorption peaks of Synozole dyes were examined under the influence acidic media, and the measurements were taken after a 15-minute interval (Figure 3.7). The spectrum analysis indicated that the highest absorbance occurred at a wavelength of 225 nm, while the lowest absorption peaks were observed at 430 nm. Over the course of each time interval, no significant changes were observed in the spectra at a wavelength of 225 nm. However, a minor shift in the peaks was observed at 430 nm. It was evident that the absorption at both wavelengths was reduced compared to the control and light conditions.

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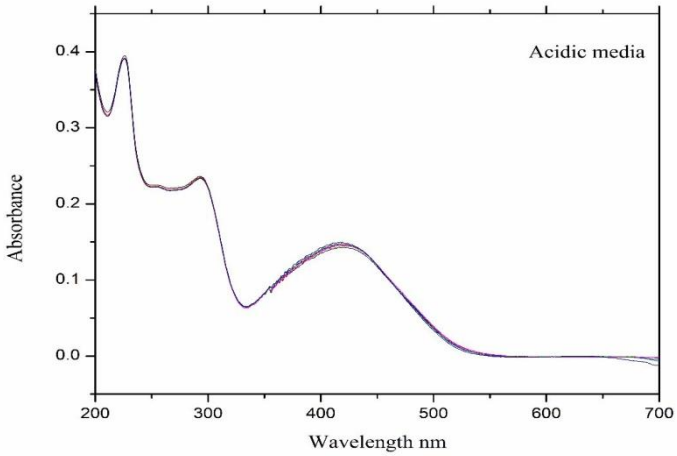

Figure 3.7: Absorbance spectroscopy of Synozole dye at 200-700 nm in the presence Acidic Media.

Discussion

The current investigation has proved that cerium oxide nanoparticles (CeONPs) can be successfully green-synthesized by utilizing *Ocimum basilicum* leaf extract and tested their photocatalytic properties towards the degradation of Synozol dyes. The findings prove that the synthesis of stable cerium oxide nanoparticle with potential catalytic efficiency can be done successfully using plant mediators. The reason why green synthesis methods have attracted much attention is that most of these methods do not rely on dangerous chemicals, is less toxic to the environment and offers a relatively inexpensive means of producing nanoparticle products than the established physical and chemical techniques (Iravani, 2011; Jeevanandam et al., 2018).

The analysis of the FTIR showed that the OH, CN, CH, CO, and other functional groups were peaks, which indicated the presence of the biomolecules formed in basil leaf extract. Such biomolecules as phenolics, flavonoids, and proteins presumably acted as reducing and stabilizing agents throughout the nanoparticle formation. The same FTIR signatures are also described when using phytochemicals in the synthesis of nanoparticles when reducing metal ions, which is required and stabilizing nanoparticles occurs (Jadoun et al., 2021). The high peak of 3690 cm -1 in the current experiment is linked to the hydroxyl groups that are commonly found in the plant metabolites, indicating that they are involved in the stabilization of the nanoparticles.

The morphology of the synthesized CeONPs was porous surface morphology as demonstrated by the analysis of the SEM. The porous or dissimilar nanoparticle structures are preferable in catalysis owing to the augmented surface area and active reaction spots. The greater the surface area, the more nanoparticles interact with pollutant molecules, which leads to the high efficiency of catalytic degradation (Zhou et al., 2020). Cerium oxide nanoparticles made with botanical extracts have been reported to possess similar porous or aggregated structures (Nadeem et al., 2020).

The EDX result confirmed the presence of cerium (Ce), oxygen (O), and carbon (C) in the produced nanoparticles, which confirmed the successfulness of the cerium

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oxide nanostructures formation. This carbon could be attributed to the presence of organic biomolecules that are still attached to the surfaces of nanoparticles as stabilizing molecules in the basil extract. Another commercially common technique to confirm the presence of nanoparticles and their composition is elemental confirmation by EDX, which is extensively applied in green nanotechnology research (Scimeca et al., 2018).

The XRD pattern was also used to verify the crystalline nature of the CeONPs and characteristic diffraction peaks with a specific Miller index were observed. These peaks show that crystalline structures of cerium oxide are formed, which corresponds to CeO2 nanoparticles that were synthesized using biological techniques previously (Corso et al., 2021). The XRD is quite commonly applied to identify the crystal structure and phase purity of nanomaterials and gives valuable data on the crystallite size and structure properties (Bunaciu et al., 2015).

Photocatalytic experiments have revealed that CeONPs had the potential of decreasing the absorbance of Synozol dye which implied that dye molecules were degraded in the presence of light. Azo dyes are known environmental pollutants because they are toxic, persistent, and biologically non-biodegradable textile dyes (Bunaciu et al., 2015; Castellanos-Henriquez et al., 2026) Due to the oxygen storage capacity and the redox mechanism between the Ce 3+ and Ce 4+ forms, cerium oxide nanoparticles have been considered as photocatalysts because of improved catalytic activities (Ahmad et al., 2020). This reduction in the dye absorbance in the current research indicates that CeONPs were able to produce reactive oxygen species that were involved in the degradation of dye molecules.

The other results showed that absorbance variations were present in varying conditions like the exposure to light and the acidic media, which implied that the environment conditions affect the photocatalytic efficiency. It has been stated that pH and light intensity have a considerable impact on the efficiency of nanoparticles during photocatalysis as previous researchers noted that these parameters influence the surface charge, electron transfer processes, and radical formation (Ahmad et al., 2020; Nadeem et al., 2018).

All in all, the results of the present research can be used to conclude that basil leaf extract may be effectively utilized as a biological agent in the synthesis of cerium oxide nanoparticles with photocatalytic properties. The successful synthesis of CeONPs was confirmed by the porous morphology, crystalline structure and elemental composition by the use of SEM, XRD and EDX analyses. Moreover, the experienced dye absorbance decreases signify that these nanoparticles can be used in environmental cleanup especially in managing textile wastewater that is contaminated with reactive dyes.

Future research must be directed at the optimization of synthesis settings, comparison between degradation rates under different environmental factors, and large-scale use of green-synthesized CeONPs in the treatment of industrial wastewater.

Conclusion

In conclusion, nanotechnology has emerged as a cutting-edge field based on physical and chemical methods, with green synthesis being a desirable alternative due to its superiority over conventional techniques. This study successfully utilized a non-

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conventional approach, namely "Green Synthesis," to prepare cerium nanoparticles (CeONPs) using *Ocimum basilicum* leaves extract. The CeONPs were characterized using various analytical techniques, including FTIR, SEM, and XRD, revealing valuable information about their structure and composition. The FTIR analysis indicated the presence of alcohol functional groups and nitrile compounds in the CeONPs. SEM characterization demonstrated the presence of wide pore spaces on the nanoparticle surface, suggesting their potential in the remediation of Synozole dyes. EDX analysis confirmed the presence of carbon, oxygen, and cerium in the CeONPs, further verifying their successful synthesis. X-ray diffraction patterns provided insights into the crystal structure of the CeONPs. Photocatalytic activity evaluation revealed a significant reduction in dye absorption at specific wavelengths, indicating the potential of the prepared CeONPs for photocatalytic remediation of dyes. Overall, this study highlights the promising application of green-synthesized CeONPs in environmental remediation processes.

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