RESEARCH PAPER

Surface modifier-mediated microwave-precipitation synthesis of ceria nanostructures and their photocatalytic activities

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Abstract

Two different types of surfactants (CTAB and SDBS) and one complexing agent-citric acid (CA) were used to synthesize CeO_2 nanostructures with the support of a microwave-precipitation process. Distinct molecular interactions of the ionic surfactants or complexing agent with the ceria precursor during nucleation and growth processes, which lead to the difference in the following features such as crystalline diffraction intensity, crystallite size, molecular functional groups, and surface morphology of the prepared CeO_2 compounds. With the help of XRD characterizations, the structural properties of the surfactants and complexing agent-mediated CeO_2 material was examined and their results showed a perfect face-centered cubic fluorite structure. The interaction between Ce–O bonds and the functional groups associated with the respective surfactant or surface modifier was identified from the FT-IR spectra. The SEM images revealed the formation of nanostructured ceria particles with well-defined and controlled morphologies. The size controlled CeO_2 nanostructures were further evaluated for photocatalytic activity through the degradation of malachite green (MG) dye under visible-light irradiation.

Keywords: Cerium oxide; Nanomaterials; Microwave-precipitation process; Surface modifier; Photocatalytic activity

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Introduction

Toxic organic pollutants as well as heavy metals have been released from the industrial sector. The released hazardous chemicals were mixed into the water, which caused water contamination to occur, followed by creating a serious ecosystem. Photocatalyst technology is the cost-effective process to clean the pollutant from the water without creating any secondary pollutants [1,2]. Photocatalytic and biological activity of nanocrystalline ceria has been investigated extensively by many researchers for its potential in the era of the environment and biomedicine [1-57]. One of the reasons for selecting the ceria material for environmental applications is its excellent redox behaviour due to the easily exchanging charge transfer states between Ce^{3+} and Ce^{4+}, having effective active sites during catalytic activity, and easily generating oxygen vacancies during the synthesis of CeO_2 [55]. Meanwhile, when it is considered for biomedical applications, CeO_2 serves as an inducer of reactive oxygen species as well as being eco-friendly and free from toxicity [55]. Hence, CeO_2 nanostructured material can take the next generation of environmental and biomedical applications.

Nanocrystalline CeO_2 material is generally called a rare earth oxide and exhibits remarkable optical and luminescence properties. These characteristics directly support the catalytic and biological performance [57]. Standard chemical synthesis such as green synthesis, solution combustion, electrochemical, precipitation, hydrothermal, and microwave reactions were used to synthesize of CeO_2 nanostructures in various shapes for the photocatalytic and biological applications [1-15, 57].

Various surfactants (CTAB, SDS)-mediated hydrothermally synthesized CeO_2 nanophotocatalyst were reported by Govindasamy Palanisamy et al. [9]. They found that the photodegradation performance of the CeO_2 nanostructures have greatly tuned under the influence of surfactants. Li et al. [10] reported the catalytic activity of CTAB-mediated CeO_2 nanostructures via various chemical routes. Aurélien Vantomme et al. [40] and Özge Tunusoğlu et al. [41] reported the synthesis of CTAB-assisted CeO_2 nanostructures through a wet chemical method. Mei et al. [42] scrutinized the catalytic activity of CTAB-mediated metal-organic-framework synthesized CeO_2. Investigation on the CO oxidation response through catalytic activity of tri polymer (P123) surfactant-assisted CeO_2 nanowires by Lu et al. [43]. CTAB-assisted CeO_2 nanoparticles have reported by Laosiripojana et al. [44] for testing the chemical sensing performance. CO oxidation response of CTAB-surfactant-mediated CeO_2 nanoparticles have examined by Yuejuan et al. [45]. Chemical sensing properties of cationic surfactant and tripolymer-assisted wet chemically synthesized mesoporous CeO_2 material have attempted by Nermeen Nabih et al. [47].

Syed Ahtzaz ul Hassan Gillani et al. [48] investigated the biological properties of nonionic surfactant-PEG and cationic surfactant-CTAB-assisted CeO_2 nanostructures. They noticed the biological activity response of CeO_2 compounds under the influence of the chosen surfactant. Gusliani Eka Putri at al. [49] and Ramprit Baitha et al. [50], Nesrin Korkmaz et al. [51], Jawahar Sukumaran et al.[52], and Al-Shimmary et al. [53] reported the green- synthesized CeO_2 nanostructures were examined for antibacterial, antioxidant, and anticancer activity. Shaidatul Najihah Matussin et al. [55] is clearly highlighting the CeO_2 nanomaterials as serving as functional properties. It is inferred from the literature reports that biofunctional property changes are highly dependent on the method of synthesis and size and morphology-controlled CeO_2 nanostructures.

In our present research goal, we attempted the facile and cost-effective surface modifier-mediated microwave-precipitation method to develop the CeO_2 nanostructures. The prepared surfactant and complexing agent-mediated CeO_2 samples are comprehensively examined in the structure and morphological behaviour studies. Moreover, better structure, crystallite size and morphological features of CeO_2 material have been taken and performed the photocatalytic activity, followed by obtained results have been discussed in detail.

2. Experimental

Initially, 0.1 M of the cerium trichloride heptahydrate was mixed in the aqueous solvent under continuous magnetic stirring until a clear homogeneous solution formation. Subsequently, 0.05 M of citric acid (CA)-complexing agent was added slowly to the solution under vigorous stirring. Thereafter, 0.3 M caustic soda (NaOH) pellets were introduced gradually into the reaction mixture under maintaining constant stirring. The addition of NaOH led to the formation of a milky white suspension, indicating the precipitation of cerium hydroxide precursor species. The resulting suspension was then subjected to microwave irradiation at 800 W for 15 min. After microwave treatment, the obtained product was collected, followed by washing it several times with aqueous solvent to eliminate the residual impurities and unwanted by-products. The purified product was subsequently dried at 80° C for 2 hrs, followed by calcined at 450 °C for 2 h to obtain pale yellow-coloured ceria (CeO_2) nanopowder with a good crystallinity state.

For comparison, the same synthesis procedure and reaction conditions were employed for the preparation of cationic surfactant-CTAB and anionic surfactant-SDBS mediated-ceria nanoparticles. In these cases, CTAB and SDBS were used individually in place of CA as surfactant agents during the microwave-assisted precipitation process.

2.1 Materials characterizations

To assess the structural properties of surfactants-CTAB and SDBS-mediated CeO_2 as well as complexing agent-CA-mediated CeO_2 material by using a X-Ray Diffractometer-Make & Model: PANALYTICAL/X'PERT3, Perkin Elmer Model: Spectrum TWO, SEM-Carl Ziess Model: EVO 18.

2.2 Photocatalytic experiment

In our earlier report, the photocatalytic experiment procedure is followed for the present work of using CTAB-mediated CeO_2 as a photocatalyst and visible light instead of samarium oxide and UV light [58]. We used MG dye and the HEBER photoreactor annular type, in this photocatalytic experiment. Photodegradation absorbance vs wavelength graphs were recorded from the UV-visible spectrophotometer- Shimadzu-UV-2450.

3. Results and discussion

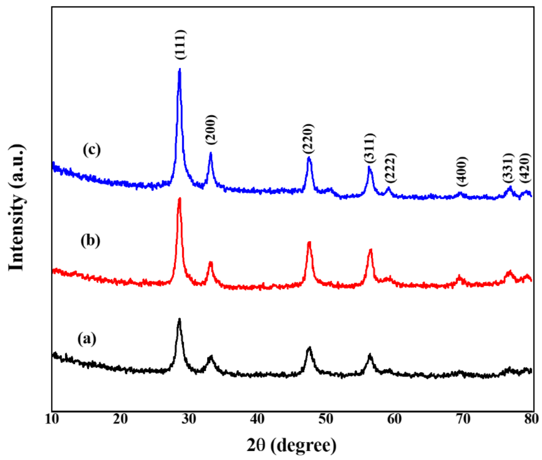

Fig. 1 Powder XRD pattern of (a) CTAB, (b) SDBS, and (c) CA -mediated ceria nanoparticles.

Fig. 1 displays the powder XRD spectra of ceria samples synthesized using CTAB and SDBS as surfactants and CA as surface modifier. All the diffraction patterns can be indexed to the face-centered cubic fluorite structure of CeO_2, which is closely related to the reported standard JCPDS card No.81-0792 of CeO_2 [59]. The prominent diffraction peaks observed at the corresponding crystallographic planes confirm the successful formation of pure phase-ceria nanoparticles. Apart from pure ceria peaks, no other impurity phase were detected, which indicates that the prepared samples are the high-purity phase. A noticeable variation in the diffraction peak intensity and peak broadening was observed among the CeO_2 samples prepared with different surfactants and surface-modified mediating agents. The CA-mediated ceria nanoparticles exhibited sharper diffraction peaks with comparatively higher intensity than the CTAB and SDBS-mediated samples. The XRD pattern of CA-assisted CeO_2 indicates a sharper and narrower width diffraction peaks, which suggests the formation of relatively larger crystallite sizes. This could arise from the effective capping and growth extension ability of CA during nanoparticle formation. In contrast, the CTAB and SDBS-mediated samples showed relatively wider diffraction peaks, indicating the formation of smaller crystallites. The difference in crystallite growth behaviour can be attributed to the distinct molecular interactions of the surfactants with the ceria precursor during the nucleation and growth processes. There was not much effective peak shifting in all samples, further confirming that the fluorite lattice structure of CeO_2 remains unchanged irrespective of the surfactant or surface modifier employed.

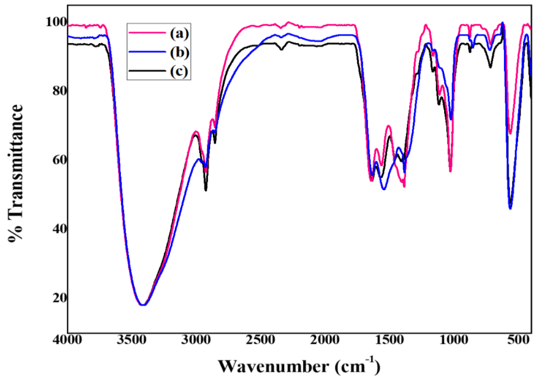

Fig. 2 FT-IR spectra of (a) CTAB, (b) SDBS, and (c) CA-mediated as prepared ceria dried sample.

Fig. 2 presents the FT-IR spectra of as-prepared ceria dried samples in the presence of different structure-directing agents, such as CTAB, SDBS, and CA. In all the samples, the broad absorption bands centered at ~3416 and 1631cm^{-1} are ascribed to the stretching vibration of surface hydroxyl (–OH) groups and adsorbed water molecules on the surface of the ceria nanoparticles [9, 10, 40]. The weak bands located at approximately 1542, 1341, and 722 cm^{-1} are associated with the asymmetric stretching, symmetric stretching, and bending vibrations of surface carbonate (CO_3^{2-}) species**,** which are commonly formed by the adsorption of atmospheric CO_2 on the basic surface of ceria nanoparticles [60]. The SDBS- and CA-assisted samples exhibit weak bands at 2919 and 2850 cm^{-1}, which are assigned to the C-H stretching vibrations [43]. These bands indicate the presence of residual organic species originating from the surfactant or surface modifier adsorbed on the nanoparticle surface. A prominent band at ~1097 cm^{-1} is observed for the CTAB-assisted sample, whereas, the SDBS and CA-assisted samples exhibit a comparatively weaker band around 1062 cm^{-1} [61, 62]. Finally, the intense strong bands located at 547, 517, and 559 cm^{-1} for the CTAB, SDBS, and CA-assisted samples**,** respectively, are assigned to the Ce–O lattice stretching vibrations [9]. The presence of these characteristic bands confirms the successful formation of ceria.

Fig. 3 (a-d) depicts the SEM photographs of nanostructured ceria particles synthesized using the microwave-precipitation process in the presence of different structure-directing agents, such as CTAB, SDBS and CA. In this present work, cerium trichloride heptahydrate and caustic soda served as the cerium precursor and precipitating agent, respectively. As shown in Fig. 3a, the CTAB-assisted ceria sample consists of well-dispersed spherical-like particles. This morphology can be ascribed to the adsorption of positively charged CTAB molecules onto the growing ceria nuclei [9], which lowers the surface energy of specific crystal facets and promotes dispersed spherical particle growth. In contrast, the SDBS-assisted sample (Fig. 3b) exhibits aggregated spherical-like nanoparticles with some places are showing elongated spherical particles in the SEM.

The negatively charged sulfonate groups of SDBS can strongly adsorb onto the positively charged ceria nuclei during nucleation [9], providing electrostatic repulsion between the developing particles, resulting in aggregated spherical with a smaller particle size when compared to the CA-mediated CeO_2 sample. This suppresses particle agglomeration and limits crystal growth, resulting in uniformly dispersed nanoparticles with reduced particle size. The CA-assisted sample (Fig. 3c) displays small rod-like nanoparticles largely aggregated rods with nanostructure formation. Citric acid acts as a chelating and capping agent by coordinating with Ce^{3+} ions through its carboxylate groups, thereby controlling the release of cerium species during nucleation and subsequent crystal growth.

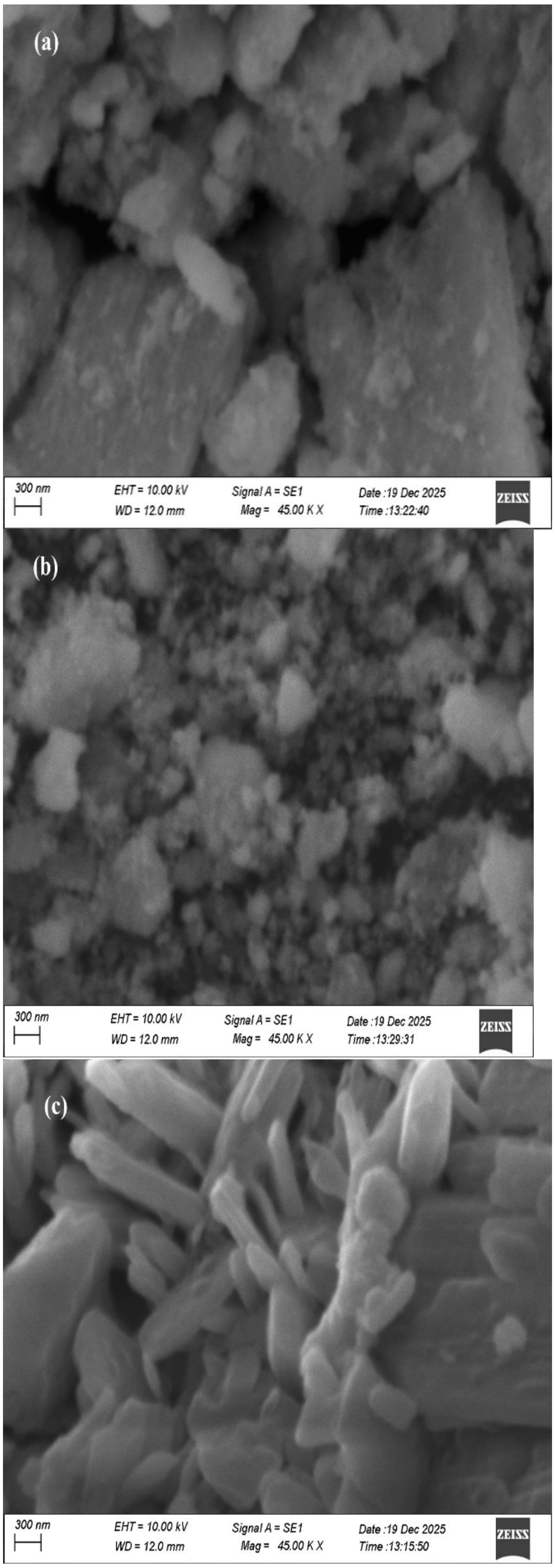

Fig. 3 SEM images of (a) CTAB (b) SDBS and (c) CA mediated ceria nanoparticles.

During the microwave-precipitation process, the selective adsorption of citrate ions on specific crystallographic planes inhibits growth along certain directions while allowing preferential growth among others, leads to the formation of rod-like nanostructures.

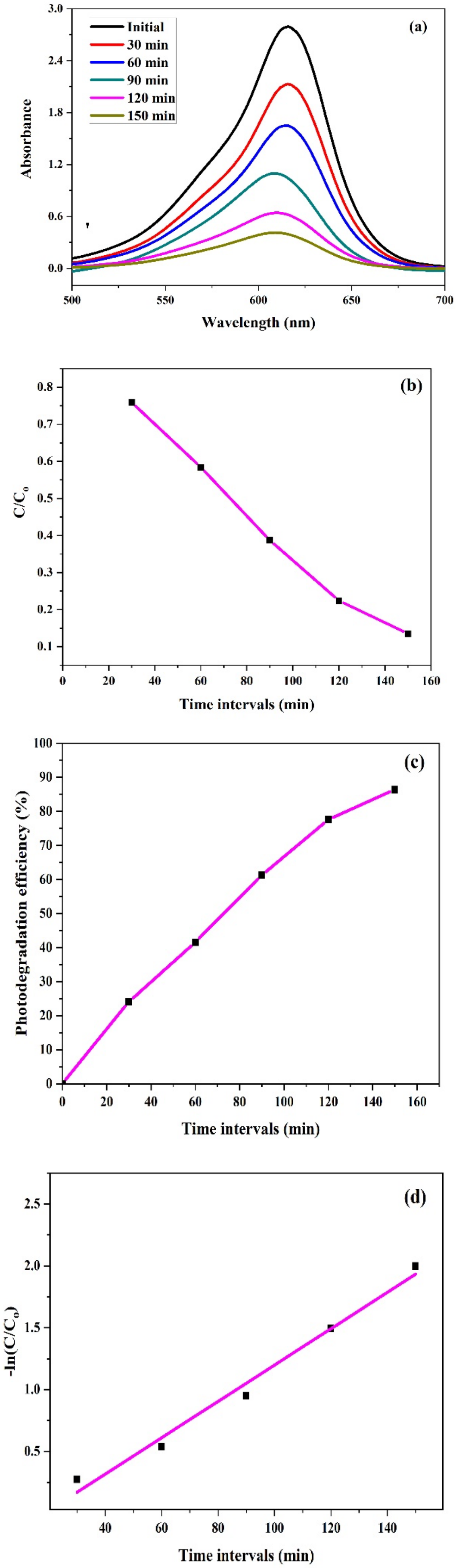

Fig. 4 (a) UV-visible spectra , (b) C/C_o vs time intervals, (c) photodegradation activity vs time intervals and (d) $-\ln(C/C_o)$ vs time intervals of MG dye photodegradation activity using CTAB-mediated ceria nanophotocatalyst under visible light.

Photodegradation activity was carried out for the mixed MG dye-CTAB-mediated ceria nanophotocatalyst solution were collected up to time irradiation intervals of 150 min under passage of UV light. In each 30 min time interval, the above-mentioned dye solution were taken for the UV-visible spectra, and their results are shown in Fig.4a. A prominent absorbance peaks was traced at the wavelength of 615 nm in the obtained UV-visible spectral images. These peaks are well supported with the reported UV-visible spectra of the MG dye. In addition, the obtained peaks absorption was gradually decreased with an increasing illumination of visible light in the recorded UV-visible spectra of the MG dye-CTAB-mediated ceria nanophotocatalyst solution. The photodegradation efficiency values are computed to be 24.1%, 41.6%, 61.3%, 77.6% and 86.46%, respectively, using the photodegradation efficiency formula ($\eta = 1 - C/C_o$) [64] for the selected irradiation time intervals of 30 min, 60 min, 90 min, 120 min, and 150 min, respectively. In the above formula, C_o and C are the before and after irradiation of MG dye solution. The recorded C_o, C and computed C/C_o, and η values are listed in Table.1. The proposed work of photodegradation activity was completed within 150 min.

Table 1: Recorded C_o, C and computed C/C_o, and η values of MG dye photodegradation activity using CTAB-mediated CeO_2 sample.

Irradiation time	**C_o**	**C**	**C/C_o**	**$\eta = 1 - C/C_o$ (%)**
0	2.79	2.79	0	0
30	2.79	2.12	0.759	24.1
60	2.79	1.63	0.584	41.6
90	2.79	1.08	0.387	61.3
120	2.79	0.625	0.224	77.6
150	2.79	0.378	0.1354	86.46

C/C_o vs time interval graphs (Fig.4b) showing C/C_o is maximum at short irradiation time intervals, while, C/C_o is minimum at prolonged irradiation time intervals. The photo degradation efficiency gradually reduced when time intervals increased from 0 to 150 min (Fig.4c). -ln Pseudo-first-order kinetics model is very useful to find the reaction rate constant with the support of $\ln C/C_o$ vs time interval graph (Fig.4d). Using the $-\ln C/C_o$ vs time interval graph, the rate constant value was found to be 0.01469 min^{-1} for the MG dye/CTAB-mediated CeO_2 photocatalyst. Here, due to electrostatic force, MG dye easily adsorbs on the ceria in the aqueous solution under illumination of visible light during the photocatalytic experiment, consequently initiating the photodegradation activity gradually due to MG dye breakdown, followed by the generated hydroxyl and superoxide radicals. During the generation of these radicals, electrons are transferred from valence band to occupy the conduction band; meanwhile, holes are generated in the valence band, followed by the recombination of electron-hole pairs. The gradual decrement of MG dye indicates that the slow decomposition of pollutant convert to carbon di oxide and carbon monoxide, which leads to a free from toxic in the dye-catalyst solution.

4. Conclusion

We report the cationic (CTAB), anionic (SDBS), and complexing agent (CA)-mediated microwave-precipitation synthesized CeO_2 nanostructures. High purity with a perfect face-centered cubic fluorite phase structure of all synthesized surfactants and complexing agent-mediated CeO_2 nanostructures were traced in the powder XRD pattern. With the support of FT-IR spectra, related functional groups of surface modifier and metal-oxygen bonds (Ce-O) of ceria before calcined sample were carried out. SEM images displayed the morphological variations arise from the different interactions of ionic surfactants and complexing agent with the ceria nuclei during the microwave-precipitation synthesis. Using MG dye, synthesized CTAB-mediated CeO_2 and visible light, the photodegradation efficiency value was computed as 86.46%.

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