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A NOVEL STUDY ON THE SYNTHESIS, CHARACTERIZATION, AND PHOTOCATALYTIC ACTIVITY OF CeO_2 NANOPARTICLES

CeO_2 NANOTANECİKLERİNİN SENTEZİ, KARAKTERİZASYONU VE FOTOKATALİTİK AKTİVİTESİ

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ABSTRACT

The discharge of untreated wastewater from unplanned industrial activities using dyes can cause serious environmental pollution and affect the aquatic environment. Semiconductor photocatalysis is a favorable technology widely used for degrading organic dyes in wastewater. This study dealt with the preparation of CeO_2 nanoparticles via a simple precipitation technique. Information on the structural and morphological features of the developed CeO_2 nanoparticles were determined using Fourier transform infrared with attenuated total reflectance (FTIR-ATR), Raman spectroscopy, X-ray diffraction (XRD), and scanning electron microscopy (SEM) spectroscopic methods. The presence of the characteristic bands of CeO_2 in the FTIR spectrum provided evidence of successful CeO_2 formation. The calculated crystallite particle size utilizing the Scherrer equation was 10 nm. SEM images revealed that the morphology of CeO_2 consisted of almost spherical particles with slight agglomeration. Brunauer-Emmett-Teller (BET) technique was also used to find out the specific surface area of CeO_2 nanoparticles (11 m^2/g). The efficiency of CeO_2 nanoparticles was also confirmed in terms of their photocatalytic activity against Rhodamine B (Rh B) under UV-A light. The results indicated that CeO_2 nanoparticles could be a promising catalyst candidate for industrial wastewater treatment.

Keywords: CeO_2 nanoparticles, photocatalyst, photocatalysis, Rhodamine B, wastewater treatment.

ÖZET

Boya içeren endüstriyel atık suyunun yeterli bir arıtma işlemi yapılmadan boşaltılması sonucunda önemli çevre ve su kirliliği oluşabilir. Yarı iletken fotokataliz, atık sulardaki organik boyaların parçalanması amacıyla yaygın olarak kullanılan uygun bir teknolojidir. Bu çalışmada, CeO_2 nanotanecikleri basit bir çöktürme yöntemi kullanılarak hazırlanmıştır. Fourier dönüşümlü kızılötesi spektroskopisi-zayıflatılmış toplam yansıma (FTIR-ATR), Raman spektroskopisi, X-ışını difraktometresi (XRD) ve taramalı elektron mikroskobu (SEM) yöntemleri kullanılarak CeO_2 nanotaneciklerinin yapısal ve morfolojik özellikleri belirlenmiştir. FTIR spektrumunda yer alan karakteristik bandların varlığı CeO_2 oluşumunun başarılı bir şekilde gerçekleştiğini göstermiştir. Scherrer eşitliği kullanılarak kristal tanecik boyutu 10 nm olarak hesaplanmıştır. SEM görüntüleri, CeO_2'nin morfolojisinin yüzeyde çok az bir topaklanma olsa da neredeyse küresel taneciklerden oluştuğunu ortaya çıkarmıştır. Brunauer-Emmett-Teller (BET) yöntemi kullanılak CeO_2 nanotaneciklerinin spesifik yüzey alanı (11 m^2/g) belirlenmiştir. CeO_2 nanotaneciklerinin etkinliği, UV ışığı altında Rhodamin B (Rh B) boyasına karşı fotokatalitik aktiviteleri incelenerek saptanmıştır. Elde edilen sonuçlar, CeO_2 nanotaneciklerinin endüstriyel atıksu arıtımında kullanılabilecek, ileriye dönük ümit vaat eden bir katalizör olduğunu göstermiştir.

Anahtar Kelimeler: CeO_2 nanotanecikleri, fotokatalizör, fotokataliz, Rhodamin B, atıksu arıtımı.

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INTRODUCTION

Unauthorized industrial wastewater discharge containing dyes from textiles, printing, paper, and food processing is a currently growing pollution problem that can cause adverse environmental and health problems (Solayman et al., 2023). The textile industry is known to be one of the largest consumers of dyes, resulting in excessive amounts of wastewater containing dye effluents (Amalina et al., 2022; Sharma & Soni, 2021). Rh B is widely used as a water-soluble textile dye colorant with carcinogenic and neurotoxic effects that can cause an extremely toxic value in textile wastewater (Al-Buriahi et al., 2022; Al-Gheethi et al., 2022).

The significance of developing efficient and modern water treatment methods has become an urgent task to prevent the negative impact of the widespread distribution of Rh B contaminated, incompletely treated wastewater into water bodies and thus to ensure the safe disposal of dye effluents in aquatic environments (Al-Buriahi et al., 2022; Linghu et al., 2023; Ma et al., 2021; Solayman et al., 2023). Despite global efforts to address this challenging issue, traditional approaches are still inadequate and have many disadvantages. Physical methods are insufficient for the complete removal of organics from wastewater. Biological treatment techniques are expensive and require long preparation cycles. On the other hand, advanced oxidation processes (AOPs) are applied as a highly effective chemical method that can degrade organics entirely with a rapid oxidation rate. The most commonly used AOPs are photocatalytic oxidation, Fenton reactions, and electrochemical oxidation reactions that do not cause secondary pollution (Ma et al., 2021; Solayman et al., 2023). Among the AOPs, photocatalysis has proven to be fast, low operating cost, chemical free, environmentally friendly, highly stable, and has high removal efficiency for recalcitrant contaminants from water. In photocatalysis, reactive oxygen species (ROS) are produced during oxidation processes when a semiconductor is captured by light (Durodola et al., 2023; Solayman et al., 2023).

Ceria, also known as CeO_2, plays a key role in a diversity of applications, particularly in optical devices, solar cells, fuel oxidation catalysis, photocatalysis, and the biological field, as this n-type semiconductor has a unique crystal structure and electronic features (Ma et al.,2023; Pansambal et al., 2022). Today, CeO_2 nanoparticles are considered as one of the most promising catalyst candidates, showing encouraging results in wastewater treatment technologies as well as photocatalysis (Kurian, 2020; Kusmierek, 2020). The exceptional characteristics and superior properties of CeO_2 are being non-toxic, inexpensive, having high chemical stability, and an efficient oxygen storage capability that makes this semiconductor as a pivotal importance photocatalyst (Tran et al., 2022). The reaction Equations (1-7) corresponding to the photocatalytic degradation mechanism of organic pollutants using CeO_2 are given as follows (Ma et al., 2019):

$$CeO_2 \xrightarrow{h\nu} CeO_2(e^- + h^+) \quad (1)$$
$$e^- + O_2 \rightarrow O_2^{\cdot -} \quad (2)$$
$$O_2^{\cdot -} + H^+ \rightarrow \cdot OOH \quad (3)$$
$$2 \cdot OOH \rightarrow O_2 + H_2O_2 \quad (4)$$
$$e^- + H_2O_2 \rightarrow OH^- + \cdot OH \quad (5)$$
$$h^+ + OH^- \rightarrow \cdot OH \quad (6)$$
$$\text{Organic pollutants} + \text{radicals} \rightarrow H_2O + CO_2 + \text{products} \quad (7)$$

The photocatalytic degradation mechanism of CeO_2 begins with the irradiation of light on the semiconductor surface ($>E_g$), resulting in electron excitation followed by a photogeneration process to produce electron/hole (e^-/h^+) pairs. These charge carriers produced in the photocatalyst are forwarded to redox reactions to form ROS containing superoxide and hydroxyl radicals as represented in the equations. Highly oxidizing hydroxyl radicals attack organic pollutants forming products as well as converting them into harmless compounds such as CO_2 and H_2O via a complete mineralization (Ma et al., 2019).

The present study reported the preparation of CeO_2 nanoparticles using a simple precipitation route that could provide broad possible aspects in wastewater treatment. FTIR-ATR, Raman spectroscopy, XRD analysis were utilized to confirm the structural properties of CeO_2 nanoparticles and the morphology of CeO_2 was investigated by SEM analysis. BET method and BJH pore size distribution technique were applied to identify the surface properties of CeO_2. Furthermore, the photocatalytic application of CeO_2 nanoparticles in the photodegradation of Rh B dye under UV light irradiation was investigated.

MATERIALS AND METHODS

Materials

Ammonium cerium (IV) nitrate ($Ce(NH_4)_2(NO_3)_6$), Sigma-Aldrich, ACS reagent, ≥98.5%) and sodium hydroxide (NaOH, Sigma-Aldrich, ACS reagent, ≥97.0%, pellets) were used without further purification. Distilled water was used for the preparation of all solutions used in experiments.

Preparation of CeO_2 Nanoparticles

CeO_2 nanoparticles were synthesized via a modified precipitation method (Seeharaj et al., 2019). In the usual synthesis procedure, 100 mL of 0.1 M $Ce(NH_4)_2(NO_3)_6$ solution was placed in a flat bottomed flask. 0.5 M NaOH solution was used to adjust the pH=12 and then added dropwise to $Ce(NH_4)_2(NO_3)_6$ solution under vigorous stirring. The entire solution continued stirring well for 2 h. The solution was then filtered, washed with distilled water, and dried in an oven at 80°C for 24 h. The pale-yellow CeO_2 nanoparticles were calcinated for 2 h using a porcelain crucible at 500°C.

Characterization Techniques

FTIR-ATR measurements (Perkin Elmer Spectrum Two) were acquired at a resolution of 4 cm^{-1} in the range of 2000-400 cm^{-1}. Dispersive Raman spectroscopy (Thermo Scientific DXR Raman Microscope) was performed using an Ar^+ laser excitation at λ=532 nm with an applied power of 10 mW. SEM analysis (FEI-Philips XL30 Scanning Electron Microscope) was carried out with an operational accelerating voltage of 10 kV. XRD diffractograms (Rigaku-D/MAX-Ultima diffractometer) were obtained using Cu Kα radiation (λ=1.54 Å) with operational parameters of 40 kV and 40 mA. BET technique and BJH pore size distribution model were used on a Quantachrome Quadrosorb SI instrument. The analytical procedure applied for nitrogen isotherm tests was at 77 K.

Photocatalytic Activity Assessment

Photocatalytic activity experiments were performed in a Pyrex vessel illuminated using a black light fluorescent lamp (125 W) placed at the top of the reactor. The volume of the solution in photocatalytic activity test was 50 mL containing a concentration of Rh B solution (10 mg/L) with a dose of CeO_2 nanoparticles (0.25 g/L). The absorption changes of Rh B dye at maximum absorbance wavelength (λ_{max}=553 nm) were monitored using a UV-vis spectrophotometer. Additional detailed information on the photocatalytic system was reported in our previous work (Turkten, 2022).

RESULTS AND DISCUSSION

Characterization of CeO_2 nanoparticles

The surface functional groups and chemical bonding of CeO_2 nanoparticles were proved by FTIR-ATR spectroscopy and the results were presented in Figure 1. The intense band observed at 1341 cm^{-1} was attributed to the stretching mode of vibration ν(Ce–O–Ce) while the band at 467 cm^{-1} corresponded to the stretching ν(-Ce–O) vibration bond in CeO_2. Moreover, a small band at 587 cm^{-1} could be assigned to the stretching ν(O-Ce-O) vibrational signal. The small bands located at 1042 cm^{-1} and 834 cm^{-1} belong to the stretching vibration of $\nu(NO_3^-)$ indicating the existence of residual nitrate moiety (Ramadan & El-Masry, 2021; Turkten, 2022; Vivek & Babu, 2016; Xie et al., 2021).

The Raman spectrum of CeO_2 nanoparticles revealed a major band at 458 cm^{-1} and a small band at 585 cm^{-1} corresponding to F_{2g} mode and LO mode, respectively (Figure 2). The F_{2g} mode was related to the fluorite structure, addressing the symmetrical stretching of oxygen around each cerium, while the non-degenerate LO mode belonged to the O^{2-} vacancies resulting from nonstoichiometric oxygen vacancies in CeO_2 (Babitha et al., 2014; Malleshappa et al., 2016).

The XRD diffractogram of CeO_2 nanoparticles was revealed in Figure 3. The specific diffraction peaks are indexed on the standard card JCPDS NO. 43–1002. The diffraction angles, 2θ = 28.54°, 33.26°, 47.58°, 56.52°, 59.32°, 69.72°, 76.64°, and 79.08° corresponded to (111), (200), (220), (311), (222), (400), (331), and (420) planes of pure CeO_2. The XRD diffractogram of CeO_2 nanoparticles agreed with the ideal fluorite lattice position related to the (111) plane located at 2θ = 28.54° (Cerrato et al., 2022; Chang et al., 2022). The XRD analysis was also well-matched with the fluorite-type structure of CeO_2, as confirmed by Raman data.

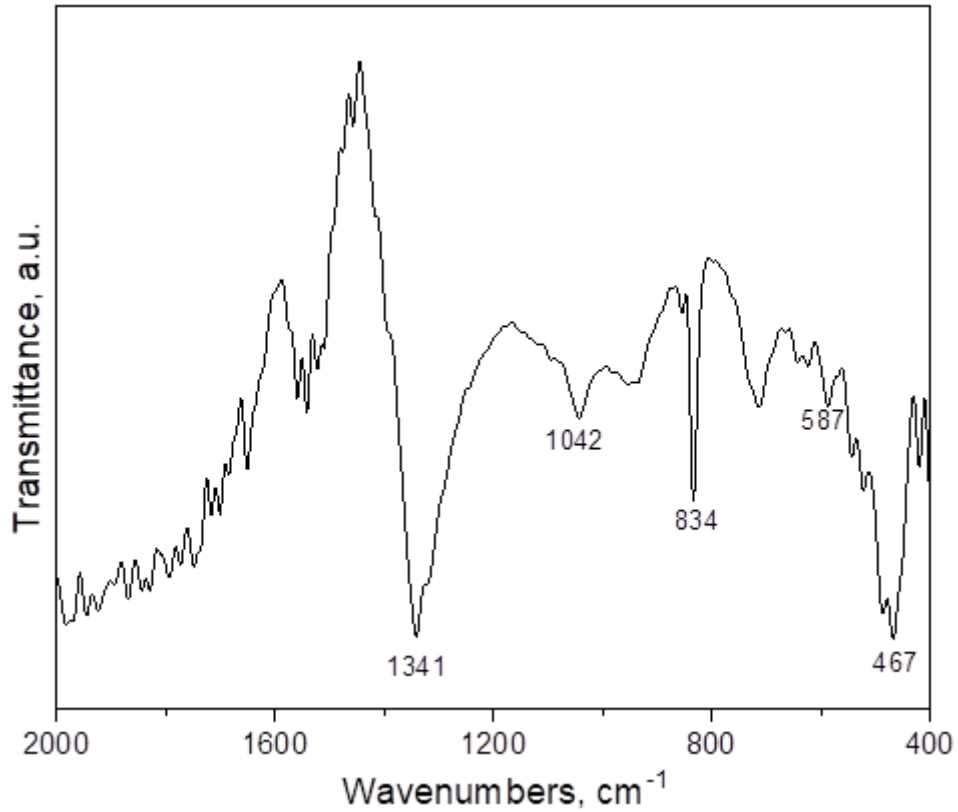

Figure 1. FTIR-ATR Spectrum of CeO_2 Nanoparticles.

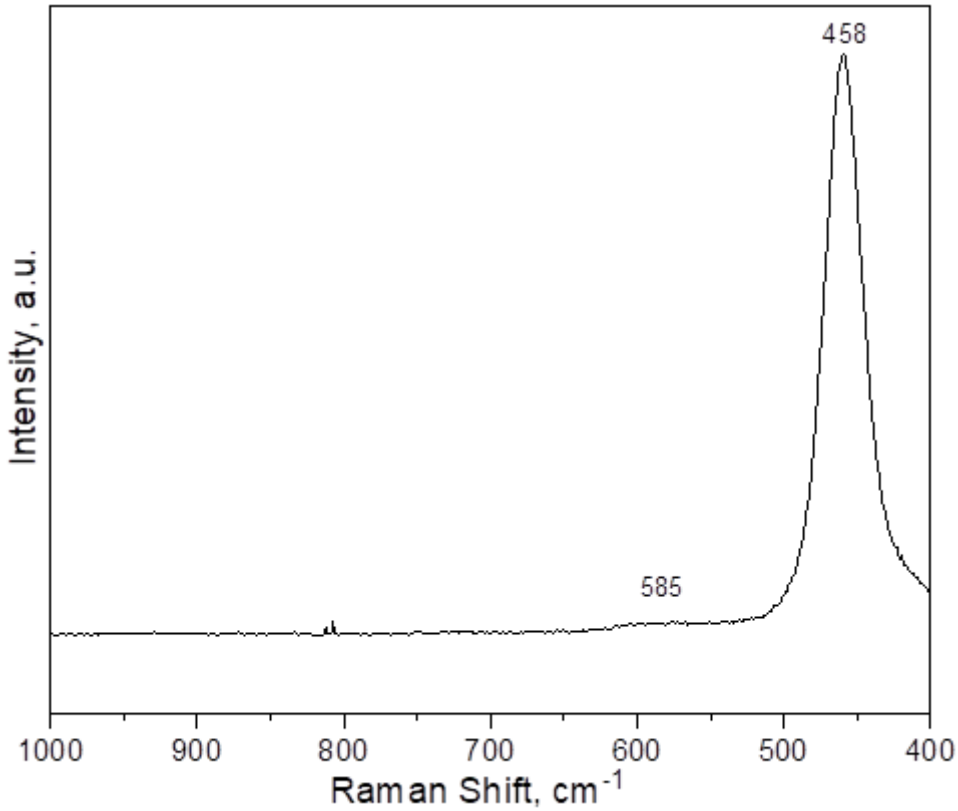

Figure 2. Raman Spectrum of CeO_2 Nanoparticles.

The crystallite size (D, nm) of CeO_2 nanoparticles was estimated from the (111) plane of CeO_2 by according to the Scherrer formula (Equation (8)) (Scherrer, 1918).

$$D = K\,\lambda / (\beta \cos\theta) \quad (8)$$

In Equation (8), K = 0.9, λ=1.5418 Å, θ is the Bragg angle, and β is the full width at half maximum intensity. The calculated crystallite size of CeO_2 particles was 10 nm, therefore CeO_2 particles were designated as CeO_2 nanoparticles throughout the manuscript.

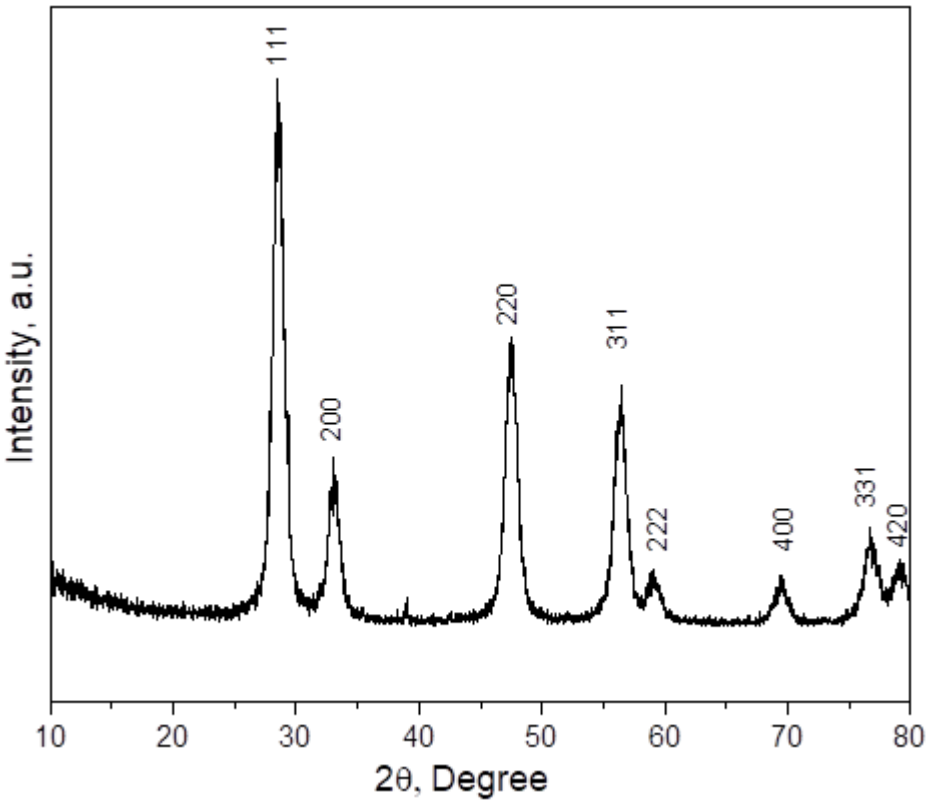

Figure 3. XRD Spectrum of CeO_2 Nanoparticles.

The surface morphological properties of CeO_2 nanoparticles were examined by SEM analysis (Figure 4). SEM images of CeO_2 nanoparticles revealed almost spherical particles with a slight agglomeration (Figure 4 (a)). The particle size was about 15 nm in diameter. The influence of pores on the CeO_2 surface with a few aggregates was clearly seen in Figure 4 (b).

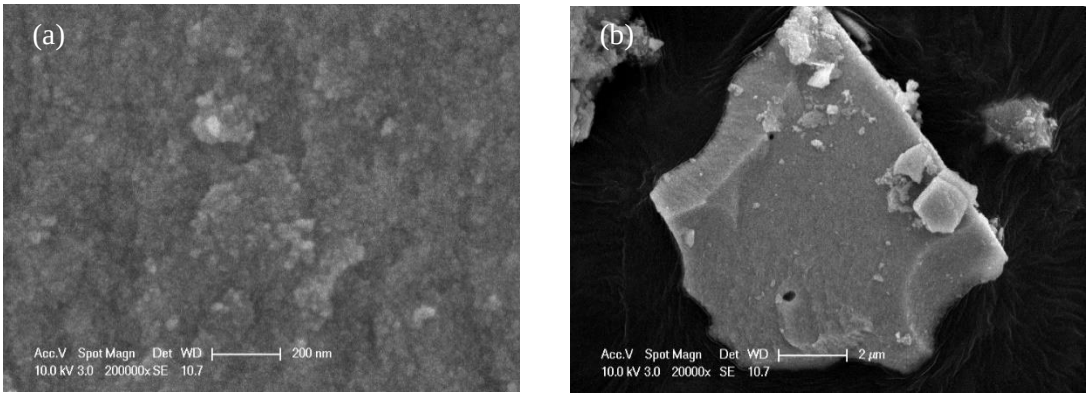

Figure 4. SEM Images of CeO_2 Nanoparticles (a) 200000x, (b)20000x.

The calculated BET specific surface area of CeO_2 nanoparticles was 11 m^2/g. This value was in agreement with the experimental BET surface areas reported in the literature (De Faria & Trasatti, 1994). The pore size diameter obtained

from BJH method was 2.51 nm, which was in agreement with the pore size distribution between 2 nm and 50 nm, indicating a mesoporous character (Sing, 1985).

Photocatalytic activity

The photocatalytic degradation ability of Rh B using CeO_2 nanoparticles over an irradiation time of 60 min to 300 min was presented in Figure 5. Moreover, the degree of Rh B decolorization by using CeO_2 nanoparticles was also estimated by the following (Equation (9)):

$$\text{Decolorization, \%} = ((A_{553,o} - A_{553})/ A_{553,o})) \times 100 \quad (9)$$

where,

$A_{553,o}$:initial absorbance of Rh B dye,
A_{553} :absorbance of Rh B dye irradiated time at t.

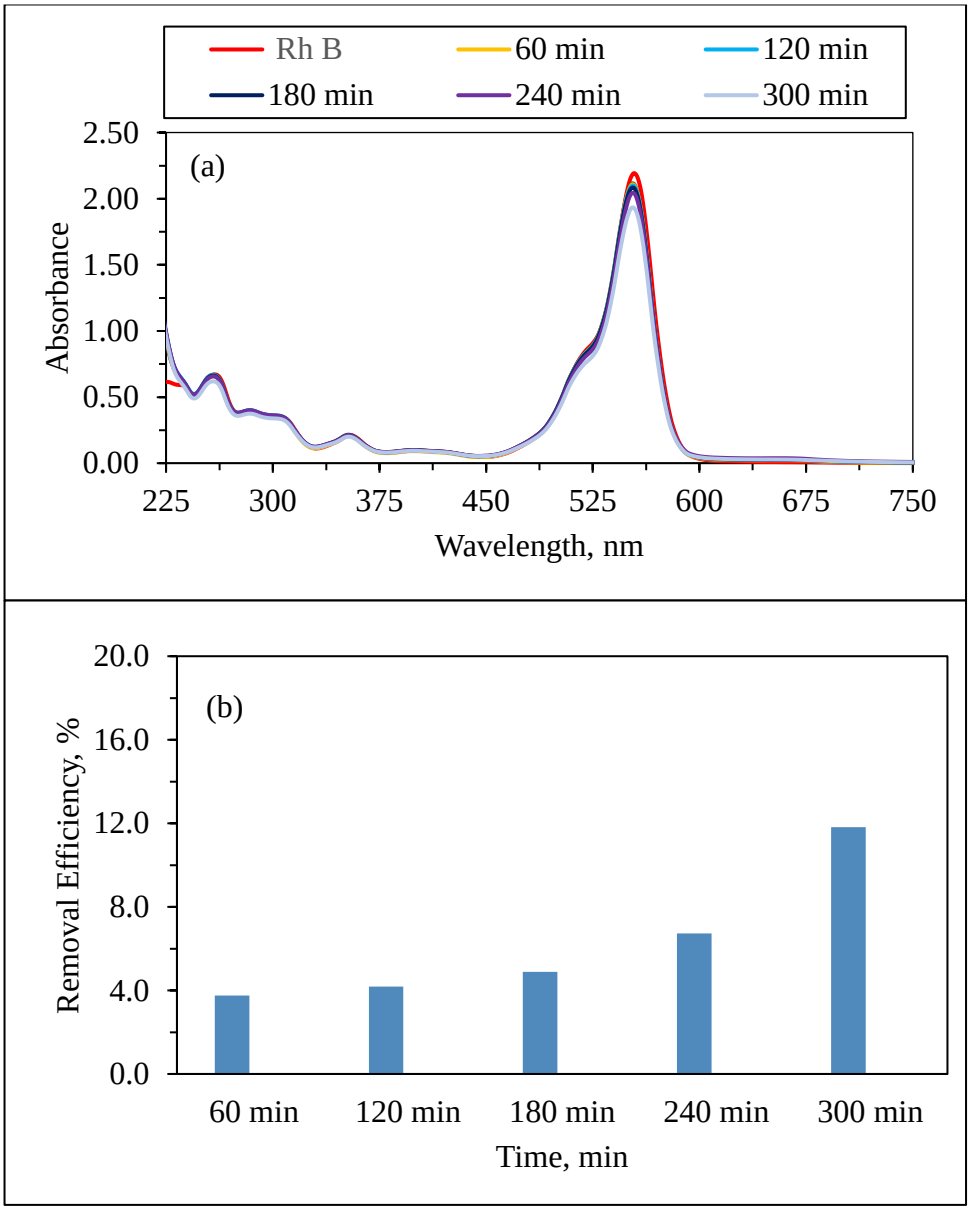

Figure 5. (a) UV-vis Photocatalytic Degradation Profiles of CeO_2 nanoparticles, (b) Photocatalytic Decolorization Degree of Rh B upon the Presence of CeO_2 Nanoparticles.

The observed reduction in the maximum wavelength absorption peak (λ_{max}=553 nm) with time could indicate the destruction of the conjugated xanthene structure of Rh B and the photocatalytic degradation of dye (Hu et al., 2015). The Rh B removal decolorization degree of the CeO_2 nanoparticles was maintained at 12% after 300 minutes of irradiation. This slow degradation could be explained by the surface charges of both CeO_2 nanoparticles and Rh B. It was reported that the isoelectric potential of CeO_2 was pH=6-8-7.3 (Issarapanacheewin et al., 2016; Nadjia, et al.,2018; Wu et al., 2021). The pH of the solution used in this photocatalytic study was at around pH=6.0. Since the surface of CeO_2 was positively charged at this pH value, Rh B cationic dye molecules were preferably repelled from the CeO_2 surface. This repulsion could result in a low decolorization degree of Rh B.

CONCLUSION

In this study, CeO_2 nanoparticles were synthesized in a cost-effective and facile way by precipitation method. Structural and morphological properties of CeO_2 nanoparticles were confirmed by FTIR-ATR, Raman, XRD, and SEM analysis. XRD and Raman results indicated a fluorite structure of CeO_2. The functional groups of CeO_2 were verified from FTIR-ATR results. SEM images revealed that CeO_2 nanoparticles exhibited almost spherical particles with slight agglomeration. Rh B was chosen as the cationic model dye, and the photocatalytic degradation of Rh B upon the use of CeO_2 nanoparticles was also investigated. Henceforth, future photocatalytic studies on anionic dye removal are proposed for prospects in research, with an overall benefit for a further application of CeO_2 nanoparticles used in the field of catalysis. The prepared CeO_2 nanoparticles could be a promising candidate material that could be utilized for various applications, especially in photocatalysis, solar cell, and optoelectronics.

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