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Effect of calcination temperature on the performance of hydrothermally grown cerium dioxide (CeO2) nanorods for the removal of Congo red dyes

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ABSTRACT

This study investigates the transformation of $CeO₂$ nanostructures through various calcination temperatures and their subsequent impact on morphological, structural, and photocatalytic properties. X-ray diffraction (XRD) analysis reveals the presence of cerium oxycarbonate in the uncalcined samples, transitioning to a face-centered cubic CeO² phase post-calcination at 500 ◦C. The scanning electron microscopy (SEM) imaging delineates a morphological evolution from distinct, rod-like structures in the uncalcined state to sintered, agglomerated forms as calcination temperatures ascend from 500 °C to 800 °C. The crystallite size, calculated using Scherrer's Equation, displayed a proportional increase with temperature. The photocatalytic degradation of Congo red dye under UV light was analyzed using UV-Vis spectroscopy, with the calcined samples exhibiting varying degrees of adsorption and photocatalytic activity.

The study found that higher calcination temperatures correlate with increased photocatalytic performance, potentially due to enhanced crystallinity. This assertion is supported by pseudo-first-order kinetic modeling, indicating improved photocatalytic efficiency with higher calcination temperatures, underlined by increasing rate constants. These findings underscore the intricate relationship between calcination-induced morphological and structural changes and the photocatalytic prowess of CeO₂ nanostructures.

Keywords: *Rare-earth Metal Oxide; Adsorption; Degradation; Wastewater Treatment; Cerium Oxide*

1. INTRODUCTION

Photocatalytic degradation has emerged as a compelling

method to tackle the issue of dye pollution in wastewa method to tackle the issue of dye pollution in wastewater treatment, due to its potential for complete mineralization of organic pollutants into non-toxic byproducts [\[1,](#page-4-0) [2,](#page-4-1) [3\]](#page-4-2). Several semiconductors, such as ZnO, TiO₂, SnO₂, MnO₂, Fe₂O₃, NiO, WO_3 , Nb_2O_5 , and CeO_2 , have been developed as effective photocatalytic materials [\[4,](#page-4-3) [5,](#page-4-4) [6,](#page-4-5) [7,](#page-4-6) [8\]](#page-4-7). Cerium dioxide $(CeO₂)$, in particular, has garnered increasing attention in the field of environmental engineering for its remarkable redox properties, high oxygen storage capacity, and stability [\[9,](#page-4-8) [10\]](#page-4-9). These attributes render it an indispensable material for applications in catalysis, fuel cells, and as an additive in diesel fuels to reduce emissions [\[11\]](#page-4-10). Recent advancements in nanotechnology have further enhanced the utility of $CeO₂$, especially in nanostructured forms, which exhibit unique properties attributable to their high surface-to-volume ratio and quantum size effects [\[12\]](#page-4-11). These nanostructures are typically synthesized using various methods, with the hydrothermal process being one of the most favored due to its relatively mild conditions, scalability, and the quality of the resultant products [\[13,](#page-4-12) [14\]](#page-4-13). The resultant morphologies, including rod-like structures, are often determined by the synthetic conditions and can be fine-tuned to optimize the material's performance for specific applications.

Our previous study highlighted the potential application of CeO₂ nanorods, fabricated using the hydrothermal process, as photocatalysts in dye degradation [\[15\]](#page-4-14). However, their performance needs enhancement to produce highperformance photocatalytic materials. Literature suggests that increasing the calcination temperature leads to materials with better photocatalytic performance [\[16\]](#page-4-15). The calcination process plays a pivotal role in tailoring the properties of metal oxide nanostructures. It is known to induce morphological changes, enhance crystallinity, and remove organic residues or other volatile contaminants [\[17,](#page-4-16) [18\]](#page-4-17). These alterations are crucial as they directly influence the material's catalytic activity, adsorptive capacities, and overall chemical reactivity. Calcination temperature is a critical parameter; it can dictate the phase stability and surface characteristics of the resulting CeO2. While lower temperatures may be sufficient to induce phase changes from precursors such as cerium oxycarbonate to pure $CeO₂$, higher temperatures may lead to sintering, growth of crystallite size, and potentially the formation of secondary phases. This study aims to understand the relation-

Figure 1. The XRD pattern of (a) calcined (CeO₂-500) vs uncalcined CeO₂ and (b) CeO₂ calcined at different temperature. (c) The $CeO₂$ crystallite size calculated from XRD patterns. (d) BET analysis of the $CeO₂$ -500 samples.

ship between calcination conditions and the characteristics of the resulting CeO2 nanostructures, which is essential for optimizing their performance in environmental applications.

2. EXPERIMENTAL SECTION

2.1 Materials

Cerium nitrate hexahydrate (Ce(NO₃)₃ \cdot 6H₂O), urea (CO(NH₂)₂), and Congo red (CR, C.I. 22120) were purchased from Merck, Darmstadt, Germany. The deionized (DI) water was used as solvent both during the hydrothermal and dye degradation measurements. All materials were used as received without any further purifications.

2.2 Preparation of CeO2 nanostructures and materials characterizations

The CeO₂ nanorods were fabricated similar to our previously reported study [\[15\]](#page-4-14). Typically, 3.0 g of $Ce(NO₃)₃$ and 4.2 g of $CO(NH₂)₂$ were dissolved in 70 mL deionized water using a magnetic stirrer until the salt completely dissolved. The mixed solution was then placed inside a 100-mL Teflon-lined autoclave and sealed tightly for the hydrothermal process. The hydrothermal growth took place at a temperature of

 100° C for 12 hours inside an electric oven. The solid was then washed with DI water and filtered several times using a vacuum filter. The solid powder was dried in an electric oven at 80 ◦C for 4 hours before calcination. To investigate the effect of calcination temperature on the dye removal performance, the calcination temperatures were varied (i.e., 500, 600, 700, and 800 $^{\circ}$ C) and the samples were named CeO₂-500, CeO₂-600, CeO₂-700, and CeO₂-800, respectively. Each sample was calcined using a predetermined holding temperature and held for 2 hours, with a ramp of 5° C/min. A yellowish powder was obtained for each calcination temperature and used for materials characterization and dye removal investigations. To investigate the effect of the calcination temperature on the CeO₂ morphology and crystalline structure, scanning electron microscopy (SEM, JEOL JSM-6510) and Xray diffractometry (XRD, BRUKER D8 ADVANCE ECO) were used respectively.

2.3 Photocatalytic dye degradation measurements

For the photodegradation investigation, we used a photoreactor chamber equipped with four Phillips UVC lamps (10 W, $\lambda \approx 253.7$ nm). Congo red (CR) aqueous solution was

Figure 2. The SEM images of the (a) uncalcined $CeO₂$, and calcined $CeO₂$ (b) $CeO₂-500$, (d) $CeO₂-600$, (e) $CeO₂-700$, and (f) CeO₂-800 samples

prepared with a concentration of 10 ppm as the model dye. Typically, 50 mg of $CeO₂$ powder was put into 100 mL CR solutions (10 ppm) and stirred inside the photoreactor chamber. The reaction was initially taken place under dark conditions for 30 min to achieve stable adsorption conditions. After that, the UV lamp was then turned on, and the CR solution was subjected to UV irradiations. Every 20 min, 3.5 mL suspension was taken and filtered using polyvinylidene fluoride (PVDF) syringe filters to separate the $CeO₂$ powder. The absorption spectra of 3.5 mL of each time variation were then measured by a UV-Vis spectrophotometer (Shimadzu UV-1280). The measurement was done for all samples ($CeO₂$ -500, $CeO₂-600$, $CeO₂-700$, and $CeO₂-800$). The concentration of the CR solutions was determined using the absorbance value at the maximum wavelength ($\lambda = 498$ nm).

3. RESULTS AND DISCUSSION

Figure [1](#page-1-0) shows the XRD pattern of the uncalcined and calcined $CeO₂$ samples. The uncalcined $CeO₂$ samples show characteristics of cerium oxycarbonate $(Ce_2(CO_3)_2O \cdot H_2O)$ in accordance with the PDF number 44-0617 (Figure [1a](#page-1-0)), which is similar to previous literatures [\[19\]](#page-4-18). This phase is a well-known oxycarbonate of cerium that is mostly obtained through precipitation of cerium salt through hydrolysis of urea $[20]$. After calcination (CeO₂-500), a new crystal phase was obtained, which has excellent similarity with the XRD pattern of face-centered cubic (FCC) $CeO₂$ (PDF#43-1002) [\[21,](#page-5-1) [22\]](#page-5-2). Previous studies found that the organic residues obtained during the hydrothermal process of $CeO₂$ start to decompose at a temperature of 320 $\mathrm{^{\circ}C}$ [\[23,](#page-5-3) [24\]](#page-5-4). Based on that information, we believed that a calcination temperature of 500 °C is sufficient to convert cerium oxycarbonate into face-centered cubic CeO₂ [\[25\]](#page-5-5). The peaks were observed at 2θ values of 28.5°; 33.1°; 47.5°; 56.3°; 59.1°; 69.4°; 76.7°; 79.1°; and 88.4◦ , which correspond to the (hkl) planes of (111), (200), (220), (311), (222), (400), (331), (420), and (422), respectively.

The influence of various calcination temperatures on the

crystalline phase of $CeO₂$ is depicted in Figure [1b](#page-1-0). Characteristic peaks of face-centered cubic (FCC) CeO₂ were observed for all samples, without the presence of additional or secondary cerium phases, confirming the successful fabrication of CeO2 nanostructures. Furthermore, the peak intensity and sharpness corresponding to cubic $CeO₂$ increased with the calcination temperature. Figure [1c](#page-1-0) presents the crystallite sizes of the CeO₂ samples at different calcination temperatures, calculated from the XRD data using Scherrer's Equation [\[26,](#page-5-6) [27\]](#page-5-7). The crystallite size was calculated to be 7.2 nm, 8.6 nm, 13.4 nm, and 19.4 nm for the CeO₂-500, CeO₂-600, CeO₂-700, and CeO2-800, respectively. The crystallite size increases with increasing calcination temperature. These findings are consistent with previously reported studies [\[28,](#page-5-8) [29\]](#page-5-9). We also performed the nitrogen (N_2) adsorption-desorption isotherm using surface area analyzer (Quantachrome QuadraWin ©2000- 16). The quantity adsorbed during adsorption/desorption process with various relative pressure were shown in Figure [1d](#page-1-0). It shows type IV hysteresis loop revealing the existence of mesopore similar to previous literatures [\[30,](#page-5-10) [31,](#page-5-11) [32\]](#page-5-12) . Moreover, the BET (Brunauer, Emmett and Teller) surface area (SBET) of the CeO2-500 shows a remarkable value of 105.04 m^2 /g, implying that the material has high surface area.

Figure [2](#page-2-0) showcases the SEM images of (a,b) uncalcined CeO₂, and calcined CeO₂ at (c) CeO₂-500, (d) CeO₂-600, (e) $CeO₂$ -700, and (f) $CeO₂$ -800. The morphological effects of calcination temperature on $CeO₂$ nanostructures were also investigated using the SEM images featured in Figure [2.](#page-2-0) Images of the CeO₂ sample without calcination treatment (Figure [2a](#page-2-0) and $2b$) show that the CeO₂ has formed into elongated, rodlike structures with a relatively uniform size and orientation, indicative of the typical morphology of $CeO₂$ fabricated via the hydrothermal process [\[15,](#page-4-14) [33\]](#page-5-13). A similar morphology was also observed for the CeO₂ sample after calcinations. For example, the $CeO₂$ -500 (Figure [2c](#page-2-0)) also shows a relatively uniform rod-like structure. However, as the calcination temperature were increasing for CeO₂-600 to CeO₂-800 samples in which shown in Figure [2d](#page-2-0) to [2f](#page-2-0) a more distinctive different

Figure 3. The UV-Vis spectra of the Congo red (CR) dyes with increasing reaction times are shown for (a) $CeO₂$ -500, (b) CeO2-600, (c) CeO2-700, and CeO2-800 samples. Panel (e) presents photograph images of the discoloration of CR solutions after increasing contact time with CeO₂-500 and CeO₂-800. Panel (f) illustrates the changes in CR peak intensity at $λ$ of 498 nm. The (g) C/C_0 and (h) $\ln(C_0/C)$ vs. reaction time are also depicted.

were observed. Upon calcination, the $CeO₂$ samples exhibit a distinct morphological evolution from well-defined, rod-like structures to increasingly sintered and agglomerated forms. Initially, the uncalcined $CeO₂$ displays discrete and uniform rods, indicative of a lower temperature synthesis with minimal particle fusion. As the calcination temperature rises, these rods gradually lose their distinctness; they broaden, fuse, and exhibit smoother edges - a transformation signifying increased diffusion and coalescence of particles [\[34\]](#page-5-14). At the highest temperatures observed, the rods become almost indistinguishable, with significant agglomeration leading to a bulkier and denser morphology.

Figures [3a](#page-3-0) to [3d](#page-3-0) present the UV-Vis spectroscopy spectra of Congo red (CR) solutions with varying contact times for all calcined $CeO₂$ samples. The CR solutions exhibit maximum absorption at a wavelength of 498 nm, with peak intensities decreasing as contact time increases, indicating a reduction in CR concentration. This trend is consistent across all samples, demonstrating a clear discoloration of the dye upon reaction or contact, which is visually confirmed (Figure [3e](#page-3-0)). Initially red, the CR solutions progressively lighten upon extended contact with the calcined CeO2. This also agrees with the UV-Vis spectroscopy investigation previously.

Figure [3f](#page-3-0) elucidates the concentration changes of the CR solutions over increasing contact times, assessed by the intensity at the peak wavelength of 498 nm. In the absence of light, the CR concentration diminishes to varying extents across the samples, with decreases of over 44%, 46%, 37%, and 32% after 30 minutes of dark contact for CeO₂-500, CeO₂-600, CeO₂-700, and CeO2-800, respectively. This behavior is attributed to the adsorptive capabilities of $CeO₂$, as documented in previous literature [\[35,](#page-5-15) [36\]](#page-5-16). Previous studies state that with rising calcination temperatures, which typically reduce the surface

area of the materials, the contribution of adsorption decreases [\[37,](#page-5-17) [38\]](#page-5-18).

Under UV irradiation, the CR concentration further declines for all samples, showcasing their photocatalytic activity. The CR degradation rate (C/C_0) , defined as the ratio of the initial CR concentration (C_0) to the concentration after a certain contact time (*C*), was calculated to evaluate the photocatalytic behavior of $CeO₂$ at different calcination temperatures (Figure [3f](#page-3-0)). The results reveal an accelerated CR concentration reduction for CeO₂-800 compared to the other samples. Specifically, after 120 minutes of UV irradiation, the CR concentration decreased by approximately 39%, 50%, 47%, and 54% for CeO₂-500, CeO₂-600, CeO₂-700, and CeO₂-800, respectively. This increase in photocatalytic performance with higher calcination temperatures is likely due to the enhanced crystallinity of the materials, as indicated by the XRD results [\[39,](#page-5-19) [40\]](#page-5-20).

Photocatalytic pseudo-first-order kinetic modeling was employed to further understand the CR degradation by the calcined $CeO₂$, based on Equation (1):

$$
\ln \frac{C_0}{C} = k_t \tag{1}
$$

where C_0 , (C) , (k) , and (t) are the CR initial concentration (ppm), CR solution concentration at given time (ppm), the pseudo-first-order constant (*min*−1), and the contact time (min), respectively. The kinetic modeling, displayed in Figure [3g](#page-3-0), exhibits a linear correlation with high fitting parameters (*R* ² values) of 0.96832, 0.98445, 0.97148, and 0.98586 for $CeO₂$ -500, $CeO₂$ -600, $CeO₂$ -700, and $CeO₂$ -800, respectively. Furthermore, the rate constant k shows an increase with the calcination temperature of the $CeO₂$, implying an enhanced photocatalytic performance.

4. CONCLUSION

In this study, CeO₂ nanostructures were successfully fabricated through a hydrothermal process followed by subsequent calcination at varying temperatures. SEM imaging revealed that all samples maintained a rod-like structure, with crystallite sizes increasing alongside the calcination temperatures. This pattern sharpening and narrowing with elevated temperatures indicate enhanced crystallization and an increase in the size of the $CeO₂$ particles. The photocatalytic capabilities of the calcined $CeO₂$ were assessed through the degradation of Congo red (CR) dye, demonstrating promising potential for wastewater treatment applications. The reduction in CR concentration can be attributed to a combination of adsorption and photocatalytic degradation mechanisms. However, it was observed that higher calcination temperatures somewhat diminish adsorption performance, likely due to alterations in surface charges and a decrease in material surface area. Conversely, the same increase in calcination temperatures correlated with improved photocatalytic activity, which can be ascribed to the higher crystallinity of the materials. These findings highlight the dual role of calcination temperature in modulating both the physical characteristics and functional properties of $CeO₂$ nanostructures. Thus, the optimal calcination temperature for $CeO₂$ nanostructures must balance these effects to maximize their efficacy in environmental applications, particularly in the domain of pollutant degradation in water treatment scenarios.

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AUTHOR CONTRIBUTIONS

AR: Conceptualization, Methodology, Investigation, Writing - Original Draft, Writing - Review & Editing, Visualization, Supervision, Funding acquisition. **RN:** Investigation. **RA:** Resources. **NIK:** Resources, Formal analysis. **KT:** Resources. **TT:** Conceptualization, Methodology, Writing - Original Draft, Writing - Review & Editing, Visualization, Funding acquisition. All authors approved the final manuscript.

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