

MICROWAVE ASSISTED SYNTHESIS OF CERIA AND SILVER DOPED CERIA NANOPARTICLES FOR THE COLORIMETRIC DETECTION OF HYDROGEN PEROXIDE



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Abstract:

Ceria nanoparticles have found many applications due to its optical properties but in order to maximize its absorption potential, doping can be a good strategy for enhancing its performance. In this research, pure ceria and Ag -doped ceria were prepared by the microwave assisted synthesis using ammonium ceric nitrate [(NH₄)₂Ce(NO₃)₆] as the precursor, and diethylene glycol (DEG) as the capping agent. The assynthesised ceria nanoparticles were characterized by UV/Visible spectrophotometer and scanning electron microscope. The undoped and Ag-doped ceria were then tested as a colorimetric sensor for the determination of hydrogen peroxide using UV spectrophotometer and the results revealed that the Ag doped ceria had a lower detection limit (0.1 ppm) compared with the undoped ceria having a detection limit of about 0.5 ppm. These nanomaterials were also immobilized on paper strips and used as colorimetric detector for hydrogen peroxide and the Ag doped still exhibited lower detection than the undoped ceria. These undoped and Ag-doped have potential applications in food storage and safety.

Keywords:

Ag-doped ceria, Ceria nanoparticles, Colorimetric sensor, Food safety, Hydrogen peroxide, Nanoparticles

Introduction

Nanotechnology has found diverse applications in sensing across various fields due to the unique properties of nanomaterials. Sensors are crucial for healthcare in drug discovery, disease diagnosis (Salehabadi et al., 2023), in environmental monitoring for air, water, soil quality as well as food safety (Tovar-Lopez, 2023) amongst other applications. As such different types of sensors including electrochemical (Simões et al., 2017), chemical sensors (Faridbod et al., 2018), temperature and optical sensors have been developed. (Rai, 2007) Colorimetric sensor is a type of chemical sensor that holds a distinctive level of interest due to its cost effectiveness, ease of use, rapid response, selectivity and sensitivity (Mo et al., 2023). It also provides clear visibility, even with the naked eye, allowing for easy and convenient detection (Srivastava, 2022). For instance, Xiao et al., 2023 employed silver nanoparticles as colorimetric sensor in industrial zinc electrolyte for cobalt detection. Alowakennu et al., 2022 used ceria nanoparticles as a colorimetric probe to detect ascorbic acid.

Ceria nanoparticles refer to nanoparticles of cerium oxide (CeO₂), a rare earth metal oxide which has gained a lot of interest due to its unique properties, such as high surface area (Carola et al., 2016), catalytic activity (Khan et al., 2019), excellent redox properties amongst others. Ceria nanoparticles have been used in various applications, including as catalysts in chemical reactions (Dey and Dhal, 2020) in fuel cells (Jaiswal et al., 2019) as a UV blocker in sunscreen (Ortiz et al., 2019), as an antioxidant in biomedical applications (Kandhasmy and Premkumar, 2023), and as colorimetric sensors for different analytes (Alowakennu et al., 2022, Jiao et al., 2012, Guo and Jing, 2010). Its excellent redox properties make it suitable as a scavenger for reactive oxygen species (Kim et al., 2023). This in turn has made it valuable in the detection of environmental and biological active species like hydrogen peroxide. Hydrogen peroxide is a compound composed of hydrogen and oxygen elements. It is a reactive oxygen species that can cause oxidative stress or the activity of

spoilage causing microorganisms (Andrés et al., 2022). Different methods have been used to detect/monitor hydrogen peroxide such as luminescence (Malyukin et al., 2018), electrochemical (Akitoye et al., 2020, Chen et al., 2012) and fluorescence methods (Li et al., 2022). Ceria nanoparticles have been used as a colorimetric sensor for the detection of glucose in biological samples via enzymatic reaction in the presence of hydrogen peroxide. For instance, Ornatska et al., 2011 explored ceria nanoparticles for colorimetric bioassays, assessing stability, reproducibility, and reusability in the ceria-based glucose assay. The study further detected glucose concentrations in human serum samples, demonstrating the efficiency of the ceria paper assay. Jiao et al., 2012 synthesized ceria nanoparticles using hydrothermal method, which displayed exceptional catalytic activity with the classic peroxide substrate in the presence of H₂O₂ which formed the basis of creating the glucose detection method in diluted human serum samples. Guo et al., 2016 used nanoceria as a highly selective colorimetric method to determine hydrogen peroxide which was later coupled with glucose oxidase to detect glucose in mouse serum samples.

In order to enhance the performance of ceria as a colorimetric sensor, doping with transition metals can be employed. Doping is the intentional introduction of dopants, notably metal ions or non-metals into the ceria lattice which confers bespoke functionalities and heightened performance vis-à-vis their undoped counterpart. For example, copperdoped ceria influences the size of nanoparticles, reduces the band gap, and increases reducibility (Mužina *et al.*, 2021), Furthermore, Manganese-doped ceria increases the specific surface area and catalytic activity for toluene oxidation (Kurajica *et al.*, 2022).

The method of synthesizing the nanoparticles is very crucial for the intended application (Nyoka *et al.*, 2020). As a result, different techniques have been reported to synthesize ceria and they include precipitation method (Pujar *et al.*, 2019, Beisuz *et al.*, 2016, Guo and Jing, 2010), hydrothermal method (Renu *et al.*, 2012, Mužina *et al.*, 2021), microwave

assisted method (Thakur and Patil, 2014, Tao *et al.*, 2010, Kumar *et al.*, 2010), green synthesis method (Arumugan *et al.*, 2015, Priya *et al.*, 2014), sol-gel method (Ferreira *et al.*, 2016, Yulizar *et al.*, 2021, Periyat *et al.*, 2011). The microwave-assisted method of synthesizing nanoparticles is known to be more efficient than the conventional heating method because of its rapid heating which accelerates and facilitates chemical reactions leading to the formation of nanoparticles with controlled size, shape and composition (Hayes, 2004, Yang *et al.*, 2005).

In this study, ceria and the Ag-doped ceria were synthesized using the microwave-assisted method and then tested for the detection of hydrogen peroxide. As far as we know, there isn't much done using doped ceria as a colorimetric sensor to detect hydrogen peroxide. The fabricated strip discussed here will function as a probe for the colorimetric determination of hydrogen peroxide, offering potential applications in ensuring food safety.

Experimental Section

Ammonium ceric nitrate $[(NH_4)_2Ce(NO_3)_6]$ was obtained from Guangdong Guanghua Sci-Tech Co Ltd, China. A stock solution of hydrogen peroxide was prepared from analytical grade hydrogen peroxide obtained from Sigma-Aldrich. Other reagents such as acetone (C_3H_6O) , diethylene glycol $(C_4H_{10}O_3)$ were analytical grade (AnalaR) reagents obtained from Sigma-Aldrich, used as received with no further purification. Aqueous solutions were prepared using distilled water. UV Vis spectra studies were carried out using a Shimadzu UV-2600-230 V spectrophotometer. The surface morphology of the nanoceria were determined using a Phenom World Scanning Electron Microscope located at Ahmadu Bello University, Department of Chemical Engineering, Abu, Zaria, Nigeria.

Methodology

Synthesis of ceria and silver doped ceria

Ceria nanoparticles was synthesized by using the microwave assisted method adopted by Wang et al., (2002). 1.096 g (0.002 mol) of ammonium cerium nitrate was dissolved in 100 mL of distilled water to get ceria solution followed by the addition of 4 mL of diethylene glycol as a capping agent. The obtained solution was magnetically stirred for 30 minutes. Afterwards, it was then placed in a microwave operating at medium high power for 30 minutes. The paleyellow colloidal solution obtained was allowed to cool to room temperature before the addition of 10 mL acetone which serves as the precipitating agent. The precipitate was centrifuged for 30 minutes at a working rate of 2000 rpm. This procedure was repeated twice while washing with acetone. The residue was dried in the oven at 40 °C and then characterized. The Ag doped ceria was synthesized by adding 5 % wt dopant using silver nitrate to the ceria solution followed by the same procedure as described for ceria.

Interaction of hydrogen peroxide with ceria and Ag doped

A 1000 ppm stock solution of hydrogen peroxide was prepared from 30 % w/v hydrogen peroxide in a 100 mL volumetric flask. From the 1000 ppm stock solution, working standards of 100 ppm, 80 ppm, 40 ppm, 20 ppm, 10 ppm, 1 ppm, 0.5 ppm and 0.1 ppm were also prepared. Then, 1 mL of each concentration was mixed and stirred with 2 mL

of 2000 ppm nanoceria solution. The resulting reddishorange solutions in different gradients were characterized using a UV-Vis spectrophotometer. For the doped ceria, 5 % Ag-doped ceria was used and the same procedure employed for the undoped ceria was followed.

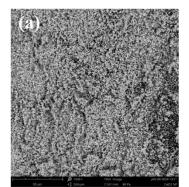
Preparation of the ceria and Ag-doped ceria-based paper strips

To immobilize the undoped ceria and Ag-doped ceria solution unto a paper strip, a round shaped Whitman filter was cut with a scissors into a rectangular paper strip and soaked for 10 minutes in a freshly prepared 2000 ppm solution of undoped and Ag-doped ceria. The soaked paper strip was dried in an oven operating at 40 °C for 10 minutes.

Results and Discussion

Scanning electron microscopy (SEM)

Nanoparticles morphology is examined through the utilization of a Scanning Electron Microscope (SEM). **Figure 1** exhibits the SEM representation of the ceria nanoparticles at different magnifications. The visual inspection reveals a majority of particles having a nearly spherical shape, alongside an observed tendency for particle agglomeration.



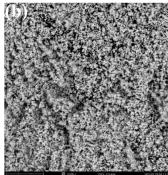


Figure 1: SEM images of ceria nanoparticles at different magnifications (a) at x1000 (b) at x2000

UV-Visible Spectroscopy

Ceria is known to absorb UV light strongly at both +3 and +4 oxidation states. UV-Vis data of ceria nanoparticles provides quantitative information about the type of oxidation state as well as the rate of reaction. Ce³⁺ is known to show absorbance between 200 to 250 nm ranges while Ce⁴⁺ absorbs UV light in the range of 300 nm to 400 nm (Sakthiyel *et al.*, 2013).

By diluting the ceria nanoparticle solution multiple times, the concentration was appropriately adjusted, minimizing scattering and self-absorption effects, and enabling accurate and reliable UV-Visible measurements of the absorption characteristics specific to the +3 and +4 oxidation states of cerium. The UV-Vis spectra of the synthesized ceria nanoparticles is given in **Figure 2** and it shows a maximum absorption at approximately 300 nm. The spectra above proves that the microwave assisted method of synthesis applied in the study produced a +4 oxidation state of the CeO₂ nanoparticles.

The UV-Vis studies of the solution of the undoped ceria with various concentrations of hydrogen peroxide is shown in **Figure 3** and it revealed that there is decrease in the

absorbance as the concentrations of the hydrogen peroxide decreases and a corresponding shift in the wavelength of absorption from 300 to 260 nm indicating the reduction of Ce ⁴⁺ to Ce ³⁺. The detection limit was found to be 0.5 ppm of the hydrogen peroxide solution. This colour gradient was also observed visually in sample bottles as indicated in **Figure 4.**

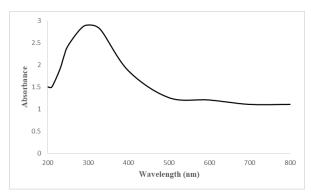


Figure 2: UV-Vis spectrum of ceria nanoparticles

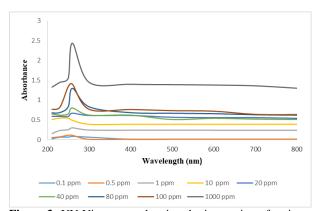


Figure 3: UV-Vis spectra showing the interaction of ceria and hydrogen peroxide at different concentrations.

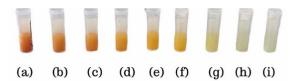


Figure 4: Colour gradients of ceria and different concentrations of hydrogen peroxide ranging from 1000 ppm to 0.1 ppm: (a) 1000 ppm (b) 100 ppm (c) 80 ppm (d) 40 ppm (e) 20 ppm (f) 10 ppm (g) 1 ppm (h) 0.5 ppm (i) 0.1 ppm.

The UV-Vis spectrum of the Ag doped ceria is shown in **Figure 5** shows it absorbs at 230 nm. The addition of silver to ceria can lead to a distortion in the lattice parameter indicating a change in the structure. In the UV spectrum of the silver doped ceria, there is peak at 230 nm compared to that of the undoped ceria which absorbs at 300 nm indicative of a blue shift which can be attributed to size effect and electron transition (Rajesh *et al.*, 2020).

The UV-Vis spectrophotometer was also used to analyze the change in absorbance versus concentration of the hydrogen

peroxide of the Ag-doped ceria as shown in Figure 6 and it was found that as the concentration of hydrogen peroxide decreases, the absorbance also decreases and a lower detection limit of about 0.1 ppm was obtained. The change in colour as the concentration of hydrogen peroxide decreased was also observed visually in sample bottles as shown in **Figure 7.** It has been found that Ag doped gives better optical absorbance and this has been explained by the fact that in silver doped ceria, the electrons released in the oxygen vacancy formation are transferred mainly to silver atoms, causing a stronger reduction (oxygen loss) and leading to a higher absorbance (Righi et al., 2022, Rajesh et al., 2020). Therefore, the enhanced detection capabilities offered by silver-doped ceria nanoparticles is likely attributed to the synergistic effects of silver doping on the catalytic activity of ceria nanoparticles.

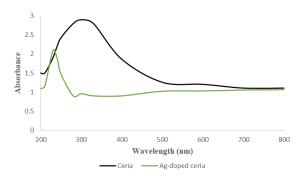


Figure 5: UV-Vis spectrum of ceria and Ag-doped ceria.

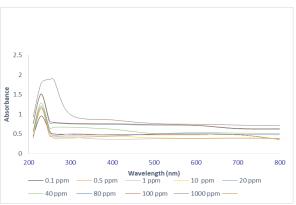


Figure 6: UV-Vis spectra on of Ag-doped ceria and hydrogen peroxide at different concentrations.

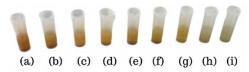


Figure 7: Colour gradients of silver doped ceria and different concentrations of hydrogen peroxide ranging from 1000 ppm to 0.1 ppm: (a) 1000 ppm (b) 100 ppm (c) 80 ppm (d) 40 ppm (e) 20 ppm (f) 10 ppm (g) 1 ppm (h) 0.5 ppm (i) 0.1 ppm.

Immobilization of ceria and Ag-doped ceria nanoparticles on paper and colorimetric detection of hydrogen peroxide concentrations

With no prior treatment carried out on the paper to be used, the nanoceria or Ag-doped was introduced unto the paper by soaking a rectangular filter paper in 2000 ppm nanoceria solution for 10 minutes before oven drying. The effectiveness of the coated paper was tested by adding three drops of the different concentrations of hydrogen peroxide solutions on the strip which spread evenly. As observed in **Figures 8 and 9**, increasing the concentration of the hydrogen peroxide resulted in an increase in the intensity of colour on the paper strip, this observation is indicative of the sensitivity of both the undoped ceria and Ag-doped ceria to the change in the concentration of the hydrogen peroxide. While the undoped ceria was only able to detect up to 1 ppm, the Ag-doped ceria on the other hand could detect as low as 0.5 ppm using the paper strip.

It was observed that the spectral detection has lower detection limit than the paper-based assay in both undoped and Ag- doped ceria. For instance, the Ag-doped ceria spectra assay could detect up to 0.1 ppm while the paper-based assay could not detect lower than 0.5 ppm. An explanation for the difference observed in the values of the detection limit of the paper strips compared to the UV-Vis analysis is due to the fact that the spectra analysis makes use of a specialized sophisticated instrumentation technique that provides quantitative data based on absorption spectrum whereas the paper strip provides semi-quantitative result based on visible colour changes (Morbioli *et al.*, 2017).

This very low spectral detection limit (0.1 ppm) is to our delight but when considering situations whereby spectra analysis is not accessible, a simple, inexpensive fabricated paper assay will be useful in such real time analysis as it has a low detection limit as well (0.5 ppm). A similar observation was reported by Ji *et al.*, when they tested both spectra and paper assay as colorimetric sensors for nitrogencontaining organic bases with spectra analysis having lower detection limit.

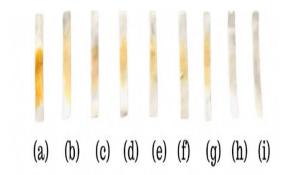


Figure 8: Nanoceria coated paper strip with different concentrations of hydrogen peroxide: (a) 1000 ppm (b) 100 ppm (c) 80 ppm (d) 40 ppm (e) 20 ppm (f) 10 ppm (g) 1 ppm (h) 0.5 ppm (i) 0.1 ppm.

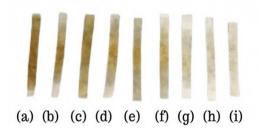


Figure 9: Ag-doped ceria coated paper strip with different concentrations of hydrogen peroxide: (a) 1000 ppm (b) 100 ppm (c) 80 ppm (d) 40 ppm (e) 20 ppm (f) 10 ppm (g) 1 ppm (h) 0.5 ppm (i) 0.1 ppm.

Conclusion

This study successfully synthesized ceria and Ag-doped ceria using microwave-assisted method. The UV-Vis spectra of ceria and doped ceria showed the wavelength and absorbance values, with the ceria spectrum showing the prepared ceria NPs was in the +4 state at 300 nm and upon interaction with hydrogen peroxide, there was a change to +3 state indicated with a shift in wavelength. The Ag-doped ceria spectrum showed the wavelength of absorption at 230 nm revealing the effect of doping on the optical property of the ceria. The ceria and Ag-doped ceria NPs tested for the colorimetric determination of hydrogen peroxide using the UV-Visible spectrophotometer revealed that the Ag-doped ceria had the lowest detection limit of about 0.1 ppm compared with the undoped ceria having value of about 0.5 ppm. The paper-based colorimetric probes, especially silvernanoceria, demonstrated effectiveness quantitatively and qualitatively analyzing hydrogen peroxide up till a detection limit of about 0.5 ppm. The robust and cost-effective nature of these sensors make them suitable for various applications including extending it to food safety for detection of hydrogen peroxide indicating food spoilage and freshness in staple food like cassava and offering flexibility in detecting both higher and lower concentrations of hydrogen peroxide.

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