Arabian Journal of Chemical and Environmental Research

Vol. 08 Issue 02 (2021) 382–417

Synthesis of CeO_2 Nanoparticles by Various Methods

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Received 29 Sept 2021, Revised 29 Oct 2021, Accepted 02 Nov 2021

Abstract

In this journal, we analyze a suitable CeO_2 nanoparticle synthesis method that can be applied in the material industry. The data collection method that we use is by reviewing secondary sources through journals related to CeO_2 nanoparticles. Then, we compare each journal that has been collected so that it can be concluded that the appropriate method to produce ceria crystals is obtained. Because this method is cheaper, uses relatively low temperatures, has fast reaction times, reproducible particle size distribution, homogeneity, and greater electrochemical performance, and is more environmentally friendly.

Keywords: CeO_2 nanoparticles, synthesis method, ceria.

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1. Introduction

Cerium oxide (CeO_2) or ceria is a pale yellow powder that is widely used in modern technology or industry. CeO_2 can be used as a sharpening agent, catalyst, UV absorber, electrolyte for solid oxide fuel cells (SOFCs), high refractive index materials similar to nescents, insulators, gas sensors, hybrid solar cells, oxygen pumps, and free radical scavengers [1]. However, the performance is influenced by its morphological form. There are many methods of synthesis of cerium oxides such as mechanochemical, hydrothermal, template-precipitation aid, sol-gel, spray drying system, plasma-spray technique, microemulsion procedure, self-assembled system, solvothermal method, and thermal decomposition [2].

In several journals with different methods of synthesizing ceria particles using the same precursor, cerium nitrate ($Ce(NO_3)_3.6H_2O$) [3] or other chemical precursors but using different capping agents or hydrolysis such as cassava starch [4], *Morinda citrifolia L.* citrus extract [5].

In this paper, we discuss the most effective CeO_2 synthesis method with an appropriate and easy-to-find capping agent. This paper consists of three major parts, namely: general information related to ceria and its application. In the second part, we discuss the methods and capping agents used in the synthesis of ceria. And in the third part, we discuss the results of the cheerful synthesis of various methods from the research that our group found and read.

In previous research, we reviewed several journals related to the methods in the synthesis of cerium oxide, namely (1) hydrothermal, (2) solvothermal, (3) sol and gel, (4) mechanochemical processing, (5) spray pyrolysis, and (6) solution combustion (SCS). The purpose of this paper is to provide information related to several synthesis methods of ceria and its capping agent as well as to determine the method of synthesis of CeO_2 nanoparticles which is easiest to perform and with large particle yields on an industrial scale. With the information in this paper, we hope that the industry can choose the most effective method that can be used in the synthesis of ceria.

2. Recent studies on precursors and methods for synthesizing ceria

Several methods and capping agents that can be used in the synthesis of CeO_2 nanoparticles can be seen in Table 1. From previous research, there are several methods in the ceria synthesis, but there are five major methods that are often used, namely hydrothermal, solvothermal, sol-gel method, and precipitation and green synthesis.

Table 1. Synthesis of CeO_2 by various methods. The table was adopted from Yulizar [5].

Precursor	Method	Hydrolyzing and Capping Agent	Particle Size	Morphology
$Ce(NO_3)_3.6H_2O$	Green Synthesis	*Hibiscus sabdariffa* leaf extract	3.9 nm	Ordered reticular planes
$Ce(NO_3)_2$	Green Synthesis	*Datura metel* leaf extract	5-15 nm	Agglomerated
$Ce(NO_3)_3.6H_2O$	Green Synthesis	*Salvia machrosipon boiss*	20-47 nm	Agglomerated particles
$CeCl_3$	Green Synthesis	*Gloriasa superba*	5 nm	Spherical shape
$Ce(NO_3)_3.6H_2O$	Green Synthesis	*Jatropha curcas*	3-5 nm	Homogenous particle

$Ce(NO_3)_3.6H_2O$	Green synthesis	*Prosopis farcta*	30 nm	Agglomerated spherical
$Ce(NO_3)_3.6H_2O$	Precipitation	Ethylene glycol Samarium	5-10 nm	Square
$Ce(NO_3)_3.6H_2O$	Precipitation	Sarium	10-13 nm	Spherical
$Ce(NO_3)_3.6H_2O$	Precipitation	PVP	27 nm	Spherical shape
$Ce(NO_3)_3.6H_2O$	Hydrothermal	Hexamethylenetetramine, NaOH	5-10 nm	Spherical shape
$Ce(NO_3)_3.6H_2O$	Hydrothermal	Trisodium phosphate dodecahydrate	<5 nm	Spherical
$Ce(NO_3)_3.6H_2O$	Solvothermal	Ethylene glycol	unidentified	Plate
$Ce(NO_3)_3.6H_2O$	Hydrothermal	NaOH	18-25 nm	Homogenous particle
$Ce(NO_3)_3$	Ball milling	Tetrabutyl ammonium hydroxide	5-56 nm	Spherical
$Ce(NO_3)_3.6H_2O$	Sol-Gel	Diphenyl ether/oleylamine	1.2-35 nm	Not uniform
$Ce(NO_3)_3.6H_2O$	Sol-Gel	Methanol	8-30 nm	Not uniform spherical
$Ce(NO_3)_3.6H_2O$	Green synthesis	*Morinda citrifolia L. fruit extract*	51.6 nm	Agglomerated spherical shape

3. Results and discussion

In the literature study, several journals from various years were searched with the keywords CeO_2 synthesis which discussed the method of the synthesis of cerium oxide. The previous research studies are contained in Table 2 below.

Table 2. Precursors, methods, procedures, CeO_2 synthesis results.

Precursor	Method	Procedure	Result	Reference
$Ce(NO_3)_3.6H_2O$	Sol-Gel	0.5 M $Ce(NO_3)_3.6H_2O$ was added to 500 g/L cassava starch solution at 25°C. The resulting mixture was stirred with a magnetic stirrer for one hour until the	Particle structure analysis was read at a temperature of 200°C-500°C with a particle size of 8-16 nm.	[4]

		gelatinization was complete. Then the gel was stored in an oven at 100°C overnight to obtain an amorphous solid (xerogel). The xerogel is heated in a temperature range of 200-500°C.		
$Ce(NO_3)_3.6H_2O$	Sol-Gel	40 mL of $Ce(NO_3)_3$ was added to a solution of Morinda citrifolia L orange extract and then stirred with a magnetic stirrer for 6 hours at 80°C until a colloid was formed. Then the colloid was calcined at 500°C for two hours.	CeO_2 NPs were successfully synthesized using Morinda citrifolia L fruit extracts. The size of the particle is 51.6 nm.	[5]
$Ce(NO_3)_3.6H_2O$	Sol-Gel	Cerium nitrate and triethanolamine were mixed with a magnetic stirrer on a hotplate at 90°C until a dark brown solution (sol) was formed. Then it is heated at 270°C for 2 hours.	The SSA of the CeO_2 nanoparticles via sol-gel procedure was calculated as 47.57 m2 /g.	[2]
$Ce(NO_3)_3.6H_2O$	Sol-Gel	$Ce(NO_3)_3.6H_2O$ was dissolved in ethanol and then stirred on a hotplate at 60°C for 15 minutes. Then cooled and added 1 mL of HCl and stirred for 2 hours. After 24 hours, the sol is poured into a petri dish and dried. After 48 hours of	Based on the half-height of the (111) diffraction peak of CeO_2, the average particle size of CeO_2 in samples with Ce/Si mass ratios of 0.1, 0.2, and 0.3 is about 4.1,	[6]

		evaporation, the sol turns into a wet gel. The wet gel was dried at 105°C for 12 hours, then the powder was calcined at 500°C for 4 hours.	4.4, and 4.6 nm, respectively, estimated from the Scherrer equation.	
$Ce_2(CO_3)_3.8H_2O$	Mechanochemical processing	Cerium chloride and sodium carbonate are weighed with the molar ratio of 2:3 and put into a nylon bottle. Smoothed with a planetary ball mill at room temperature. Then the powder is calcined at different temperatures. The removal of the by-product NaCl was carried out by washing powder with hot water and ethanol is done gradually. The washed powder was dried in an oven at 75°C for 3 hours later the analysis was carried out. X-ray diffraction. The powder was recorded on a D/Max-3B. X-ray diffraction instruments; particle size was determined by a light laser scattering (LLS) particle size analyzer from LS601; and morphology and final crystal size of the	CeO_2 nanoparticles with 40–70 nm in size were obtained after calcining the as-milled powder at 900°C and washing with deionized water and alcohol successively and drying at 75°C.	[7]

		product were examined by transmission electron microscopy, using a Hitachi H-600.		
$Ce_2(CO_3)_3.8H_2O$	Mechanochemical processing	Cerium carbonate, sodium hydroxide, water, and agate ball were weighed in a weight ratio (4:1:5:40) and placed in a nylon bottle, crushed at room temperature, and atmospheric air. Then the powder was filtered and washed five times with water, dried at about 110°C, ignited at different temperatures, and then analyzed. Sample powder diffraction pattern recorded on the D/Max-3B X-ray diffraction instrument. Thermal analysis and differential thermogravimetry were carried out using the DT-40 thermoanalyser system. The particle size was determined by laser diffraction particle size analyzer LS601 and the morphology and particle size morph of the final product were examined by	The synthesized nano-sized CeO_2 by mechanochemical processing crystallites whose primary crystal size ranges from 10 to 20 nm.	[8]

		transmission selection microscope using the Hitachi H-600.		
eO_2 γ - Al_2O_3 support	Aerosol (*pyrolysis spray*)	An $AlCl_3.6H_2O$ solution (AR, >99.0%, Sinopharm Chemical Reagent Co., Ltd.) was used as a precursor. The solution containing Ce was added to the solution containing Al in a molar ratio of 1: 6. The spray pyrolysis system consists of a homemade atomizer, a corundum tube in a tube furnace, three cyclones as collectors, a gas compensation tank, and the chimney gas absorber. The precursor droplets are expelled by blowing compressed air through the fully atomized atomizers. After passing through the diffusion dryer, the droplets are partially dry. This is carried into a corundum tube which is placed in a tube furnace, and the pyrolyzate. The resultant is finally collected by the filter sampler. The roasting temperature is controlled and regulated within the	CeO_2 obtained on Al_2O_3 carrier has a narrow particle size distribution, with 77.25% particles distributed between 5.68 and 26.75m, specific surface area spherical hollow or scaly, and cerium oxide is evenly distributed over the Al_2O_3.	[9]

		range from 900 to 1100°C, and residence time is 2.6 to 3.0 seconds.		
$Zn(CH_3COO)_2 \cdot 2H_2O$ and $Ce(NO_3)_2.6H_2O$	Aerosol (*pyrolysis spray*)	The FTO substrate was washed in deionized water (DI), acetone and ethanol and dried in an oven at 78°C. Zinc acetate (0.15 M), cerium nitrate (0.03 M), and thiourea (0.03 M) were dissolved in deionized water and used as a precursor solution. The nanocomposite was prepared at FTO substrate with spray atomization pyrolysis technology. The atomization frequency is 1.8 MHz. Use compressed air as carrier gas with a flow rate of 15 mL min 1. The distance from the nozzle to the substrate is 10 cm and the substrate temperature is set to 150°C. Spray the starting solution on the substrate for 20 minutes.	XRD analysis confirmed the formation of ZnO and CeO_2 with crystal size around 20 nm. Nanocomposite deposited on the substrate as morphologically entangled aggregates.	[10]
Cerium acetate hydrate	Aerosol (*pyrolysis spray*)	Dissolve cerium acetate hydrate (Aldrich 99.9%; 1.5% or 0.15%) in water and mix with ethanol (1:2 W/EtOH). Ultrasonic atomizer conventional	In an oxidizing atmosphere, a dense nanosphere was found, while a porous structure with a lower density	[11]

		resonant frequency is used to spray the precursor solution into a fine mist. 1.65MHz. Dry airflow or 2L/min Ar/O_2 flow transporting mist to high-temperature tube quartz reactor. The length and diameter of the reactor are 1000 mm and 70 mm, respectively. Fog ignited by a 3-zone furnace, which causes the metal precursors to burn and generate cerium oxide particles. The cheerful dust in the river is collected by dust collectors.	is generated by the airflow, which corresponds to the relatively larger particles in the size distribution.	
$Ce(NO_3)_3{\cdot}6H_2O$	Aerosol (*pyrolysis spray*)	In a typical process, 0.01 mol/L $Ce(NO_3)_3{\cdot}6H_2O$ is dissolved in 15 mL of distilled water and 15 mL of ethanol. Immerse the solution in an ice bath with constant stirring, and add 3 mL of acetylacetonate and 4 mL of propylene oxide to the solution. We added concentrated ammonium hydroxide dropwise to the solution until the pH is about 6 and a yellow solution is formed. The solution was then stirred	Increase photocatalytic activity CeO2 under visible light irradiation. The first-order level of the photocatalytic color change constant of CeO_2/FACs is 1.9 times that of pure CeO_2. Moreover, due to its low density, the composite material is easily separated	[12]

		vigorously for 0.5 h, then 10 g of FAC was added and the mixture was stirred for another 2 hours in an ice bath. The mixture was then cooled overnight, evaporated, dried and calcined at 450°C for 1 hour. Then use CeO_2/FAC to represent the prepared sample. Based on the results of EDX and XPS, the loading rate of CeO_2 on the coal cell surface is 14.5% to 14.8%.	from the water after the reaction.	
Ceria oxide (CeO_2)	Solvothermal	Add 7.5 mL of serum nitrate ($Ce(NO_3)_3.6H_2O$, Alpha Aesar, 99.5%) (16.7 mM) in water into 20 mL autoclaved Teflon-coated stainless steel. Transfer 7.5 mL toluene, 0.75 mL oleic acid (OA, Sigma Aldrich, 90%) and 75 L tert-butylamine (TBA, TCI, >98%) respectively. autoclave. The sealed autoclave is then placed in an oven that has been preheated to a temperature of 180°C and stored for 24 hours. After the autoclave has cooled to room	Research has shown that CeO_2 nanocubes, truncated nano octahedrons, and nano octahedrons were synthesized using traditional hydrothermal methods and two-phase solvothermal methods. Research has also shown that protective ligands with different functional groups play various roles in controlling the	[13]

		temperature, cloudy, the organic layer on top of the chocolate was separated using a separating funnel and collected. After adding a sufficient amount of ethanol to the organic suspension, the product is separated by centrifugation. The product is further purified by adding a mixture of hexane and ethanol (1:2, vol.) and ethanol (200 runs), followed by centrifugation for several cycles and drying in a vacuum oven.	growth and shape evolution of nanocrystals.	
Cerium nitrate hexahydrate $Ce(NO_3)_3 \cdot 6H_2O$	Solvothermal	Dissolve an amount of sodium nitrate in 10 mL of methanol. The solution was transferred to a Teflon-coated stainless steel autoclave and placed in an oven at 130-210°C for 12 hours. The resulting material was collected by filtration, then washed 3 times with absolute ethanol and dried at 60°C for 12 hours. The surface and crystallinity of the prepared composite particles were characterized using an X-	The methanol solvothermal method is an effective method for synthesizing cubic nanoparticles of CeO_2. CeO_2 particle size increased with increasing $Ce(NO_3)_3$ concentration from hundreds of nanometers to several microns.	[14]

		ray diffractometer. Determine the particle morphology, inter-particle structure, and surface characteristics of a particle.		
Cerium oxide (CeO_2)	Solvothermal	First, 0.434 g $Ce(NO_3)_3 \cdot 6H_2O$ and 0.11 g PVP were dissolved in 1, 2, and 5 mL of deionized water, respectively, and mixed with DEG to obtain a total solution of 12 mL. In addition, the mixed solution is stirred until clear, then transferred to a layered autoclave Te has a capacity of 20 mL and is heated at 180°C for 6 hours. Finally, the resulting solution was centrifuged and washed with water and ethanol, dried naturally, and then calcined at 600°C for 2 hours. Synthesize the product with 1, 2, and 5 mL of water, respectively, and label them S1, S2, and S3.	Water, DEG high viscosity, and PVP surfactant were found to have a synergistic effect on the blistering and final form of CeO_2 PNS. A series of characterization results show that PNS. The synthesized CeO_2 showed good crystallinity, large surface area, wide, and high thermal stability. Furthermore, the synthesized PNS CeO_2 had a high adsorption capacity.	[15]
Cerium oxide (CeO_2)	Solvothermal	1.0 g of HA and 0.1 g of sodium polyacrylate were dissolved in 20 mL of ethylene glycol by ultrasonic treatment to form a mixed solution. Then with	The size of the CeO_2 nanosheet is about 100-500 nm. The adsorption behavior of the congo red (CR) composite was	[16]

		ultrasonic treatment for 20 minutes, 30 mL of $Ce(NO)_3$. solution 0.1 mol/L was added to the mixed solution, the mixture was transferred to a 100 mL PTFE-coated autoclave, and heated at 180ºC for 12 hours. After the autoclave was cooled to room temperature, the precipitate was washed several times with distilled water and dried at 100°C. The resulting product was recorded as $HAsCeO_2$. Prepare an NP sample of pure CeO_2 without HA using the same procedure.	in accordance with the pseudo-order model and the Langmuir adsorption model, and the maximum adsorption capacity for CR reached 2 60 mg g^{-1}.	
Cerium(III) nitrate hexahydrate	Solvothermal	Dissolve 8 g of $Ce(NO_3)_3 \cdot 6H_2O$ in a mixture of 200 mL of deionized water (DI) and 100 mL of anhydrous alcohol to form a serious precursor solution. Next, add 2 mL of hydrogen peroxide (HO, 60%) to the serum precursor solution. To increase the purity of the powdered product, 20 mL of ammonium hydroxide (NH_3H_2O) was added to the	The nano CeO_2 aggregates showed the best and highest crystallinity, with a specific surface area of 110.92 m^2g^1. The results showed that the nano CeO_2 aggregates showed high efficiency in removing arsenic from low concentration As solutions.	[17]

		solution, at a rate of 1 ml.min1 through a constant flow pump. The above reaction was carried out in a water bath at 30°C, with a constant magnet and stirring speed 400r min1. After being stirred and allowed to stand for 6 hours, the precipitate was centrifuged and washed several times with deionized water and anhydrous alcohol. Weigh all precipitates, add 37.5 mL of absolute alcohol, transfer to a 50-mL autoclave coated with polytetrafluoroethylene, and heat at 180 °C for a period of time. After the solvothermal process, nano CeO2 can be obtained by filtration and vacuum freeze drying at 55°C for 6 hours.		
Ceria oxide (CeO_2)	Solvothermal	Under typical synthesis conditions, 0.01 mol octadecylamine (2.695 g) was dissolved in a solution containing 40 mL of deionized water and 40 mL of absolute ethanol. After	Pure phase nanorods of CeO_2 (about 4050 nm in diameter and 0.32 m in length) were synthesized by solvothermal	[18]

		octadecylamine was completely soluble, 0.01 moles of cerium(III) chloride hydrate (3.725g) were added with magnetic stirring. After that, 5 mL of anhydrous ethylenediamine was poured into the solution to control the pH value (pH 910) and adjust the viscosity of the resulting suspension. The mixture was stirred for 3 hours and then transferred to a 100 mL autoclave coated with polytetrafluoroethylene. The autoclave was then tightly closed and stored at 160°C for 72 hours. After that, the autoclave was naturally cooled to room temperature. The supernatant was discarded, and the remaining product was washed several times with acetone, hot ethanol solution, and deionized water, and then centrifuged. The product is dried in a vacuum oven at 80°C for 6 hours and then calcined at 400°C in the air.	synthesis. The ultraviolet-visible absorption spectrum and photoluminescence spectrum of CeO_2 nano bars exhibited increased redshift and abnormal light emission, respectively.	

Ceria oxide (CeO_2)	Solvothermal	CeO_2:Er/Yb nanorods were synthesized by the solvothermal method. All chemicals in this work are analytically pure and can be used directly without further purification. In the typical process of synthesis of CeO_2:Er/Yb nanorods, 1 mL (NH_4) $2C_2O_4.5H_2O$ (0.3 M), mL M $(NO_3)_3$ (M = Ce, Yb, Er, 0.1 M, Ce: Yb: Er = 78:20:2), 1.5 mL of deionized water and 2.8838 g of sodium dodecyl sulfate (SDS) were dissolved in 60 mL of cyclohexane. The above solution was stirred at room temperature for several minutes and then transferred to a 100mL stainless steel autoclave coated with Teflon, placed in the oven, and heated at 160°C for 6 hours. After cooling to room temperature, the precipitate was collected by centrifugation, washed 3 times with deionized water and absolute ethanol, and dried in an oven at 60°C. Finally, the powder was	The structure and morphology of the CeO_2:Er/Yb nanorods were characterized by XRD, SEM, and HRTEM. Nanorod emits two kinds of visible light: green (2H11/2, 4S3/2 > 4I15/2) and red (4F9/2 > 4I15/2), and the room temperature is stimulated by a near-infrared 980 nm continuous wave laser.	[19]

		calcined at 700°C at a heating rate of 3°C/min for 3 hours.		
Ceria oxide (CeO_2)	Solvothermal	Pour 1.17 g TEA into 8.68 g $Ce(NO_3)_3$ $6H_2O$, mix, then add ethanol to a final volume of 30 ml. CeO_2, 8.14 g $Ce(NO_3)_3$ $6H_2O$, and 2.63 g HMT co-doped with CN were also used, and methanol was added to a final volume of 30 ml. For comparison purposes, three undoped CeO_2 samples were synthesized without TEA or HMT. Then, 30 mL of each solution was carefully poured into a 50 mL Teflon-coated stainless steel autoclave and sealed. Samples were autoclaved at 120/140°C for 24 hours. The precipitate formed with NS was washed with distilled water and ethanol then dried under vacuum at 60°C for 12 hours. Finally, all samples were calcined in air at 400 °C for 1 hour.	The diameter of the doped and undoped CeO_2 single crystal is less than 10 nm. Nitrogen and carbon-nitrogen have been certified to join the CeO_2 network. Results showed that the relationship between AO7 degradation in the presence of nanoCeO_2 was as follows: CN co-doped CeO_2>N-doped CeO_2>>undoped CeO_2, which were 98.8%, 97.6%, and 48.2%, respectively.	[20]
Cerium nitrat ($Ce(NO_3)_2.6H_2O$)	Hydrothermal	0.217g Cerium nitrate hexahydrate [$Ce(NO_3)_2$ $6H_2O$] and 0.280g NaOH were added to 5mL and	XRD study revealed an average grain size of 12.8 nm, SEM study 23 nm,	[21]

		35mL of distilled water, respectively. This solution was mixed and stirred at room temperature for 30 minutes. the solution obtained was heated at a constant temperature of 180°C for 12 hours and 24 hours in an autoclave. The final product was collected from the autoclave and washed with distilled water and ethanol. The product was dried at 80°C for 6 hours.	UV-visible spectroscopic study smaller size CeO2 nanoparticles have bandgap energy of 3.52eV.	
$Ce(NO_3)_3$. $6H_2O$	Hydrothermal	1 mmol $Ce(NO_3)_3.6H_2O$ was dissolved in 30 mL of PVP/ethanol. Then the solution was mixed with 3 mmol of NaOH. The mixture was stirred for 10 minutes and transferred to the reactor. It is then heated in an oven at 200°C for 24 - 48 hours, and then cooled to room temperature. The final product is collected after being centrifuged and washed several times with deionized water to remove residual reactants.	The dominant form of this CeO_2 particle is a regular truncated octahedron and an irregular truncated octahedron.	[3]
Cerium amonia nitrate	Hydrothermal	CeO_2 nanoparticles were synthesized through a	CeO_2 nanoparticles in the size range of	[22]

		hydrothermal process. Cerium ammonia nitrate is dissolved in water, and a solution of ammonia hydroxide is added to precipitate the cerium salt. Then heated at a temperature of 300°C for 2-6 hours. The resulting nanoparticles were poured and washed several times with water.	3-10 nm particle shape are dominated by truncated octahedral.	
Ceriumchloride $CeCl_3 \cdot 7H_2O$ and hexamethylenet-ramine (HMTA; $C_6H_{12}N_4$) 0.1M	Hydrothermal	An aqueous solution of 0.1 M Ceriumchloride ($CeCl_3.7H_2O$) and 0.1M hexamethylenetetramine (HMTA; $C_6H_{12}N_4$), prepared in 80 mL of deionized water (DI) was mixed thoroughly on a magnetic stirrer with constant stirring for 45 minutes. After that, the resulting solution was put into a Teflon glass then packed in a stainless steel autoclave and heated at 160°C for 6 hours. After the reaction was complete, the autoclave was allowed to cool at room temperature and the precipitate obtained was poured off, washed	Synthesis of CeO_2 nanomaterials of average size from 10 - 20 nm seen in FESEM images, Average particle diameter was found to be 15 ± 2 nm from TEM images.	[23]

		with ethanol and deionized water, and dried overnight in an oven at 80°C.		
$CeCl_2$ and urea	Hydrothermal	CeO_2, $CeCl_2$, and urea (molar ratio 1:1) were dissolved in distilled water and the pH was raised above 10 by the addition of NaOH. The solution was then heated at 150°C for 15 hours in a Teflon-coated autoclave. The product obtained was washed, dried, and further calcined at 250ºC for 5 hours. CeO_2 was also synthesized by the same procedure without urea.	CeO_2 cubic morphology crystal with a mean diameter of 12 ± 10 nm.	[24]
Zinc chloride and cerium nitrate	Hydrothermal	50 mL of 0.25 M zinc chloride solution is mixed with 50 mL equimolar of Cerium nitrate solution with continuous stirring. The pH of the reaction solution was adjusted to 8 by careful dropwise addition. 1:1 aqueous NH_3 solution. All solutions were made in distilled water. then put in the microwave for 30 minutes at 200°C. After the reaction is complete, the solution is	Utilization of CeO_2-ZnO nanoparticles for the fabrication of highly sensitive and reproducible nitroaniline chemical sensors.	[25]

		cooled to room temperature followed by filtration and washing with distilled water to remove impurities.		
CuO/CeO_2 catalyst	Solution combustion method (SCS)	Fuel is dissolved in a minimum volume of distilled water. Next, cerium (III)-98.0% and copper(II)-99.0% nitrate are added to this solution, then the solution is stirred until all components are dissolved. After that, the resulting solution is put into a stainless steel beaker, which is heated on a hot plate. maintained until complete evaporation of water from the solution. then burned to form a powder. The combustion products were calcined at 500°C for 2 hours to remove residual carbon and obtain a catalyst.	Used fuel greatly affects the characterization and low-temperature reduction behavior of the CuO/CeO_2 catalyst, and the CuO/CeO_2-urea catalyst shows high stability for CO oxidation for five cycles and water resistance.	[26]
Ceric ammonium nitrate as the oxidizing agent and ethylenediaminetetraacetic acid (EDTA) as fuel	Solution combustion method (SCS)	$(NH_4)_2$ $Ce(NO_3)_6$ and disodium salt EDTA were used as the oxidizing agent and fuel respectively. The stoichiometric ratio of the amount (1:1) of oxidizing agent (2g) and fuel (0.8g) was taken in a container,	XRD: the product obtained is cerianite, a cubic phase of CeO_2 with a crystal size of 35 nm. TEM: indicates spherical particles;	[27]

		added 10 mL of distilled water, and the mixture was stirred for 10 minutes. The homogenized solution is heated and dehydrated on a hot plate. after being dehydrated and forming a gel, then put into a heated furnace, wait for 3-4 minutes and form nanocrystalline CeO_2.	nanoparticle size found 42 nm.	
Crystalline mesoporous CeO_2 catalyst and colloidal SiO_2 as the template	Colloidal solution combustion method (CSCS)	$Ce(NO_3)_3 \cdot 6H_2O$ and 0.4 g of CH_2NH_2COOH dissolved in 5 mL of water. The solution was transferred to a beaker (volume 100 mL) and heated at 150°C on a hot plate. After a few minutes, combustion occurs with a rapid increase in temperature due to the exothermic reaction between $Ce(NO_3)_3$ and CH_2NH_2COOH.	CeO_2 large-scale production indicated by 0.5 kg of mesoporous Ce(III) catalyst produced in 5 hours.	[28]
Pure CeO_2 sample and two samples of CeO_2 doped with 3-6% Fe	Solution combustion method (SCS)	1 g of $((NH_4)_2[Ce(NO_3)_6])$, and 0.023g or 0.047g of iron nitrate $(Fe(NO_3)_3)$ to obtain Ce0.98 Fe0.03 O2 and Ce0.94 Fe0.06 O2 of each composition. 0.5277 g or 0.5395 g of glycine (fuel) are added as determined by	Fe is mostly present in the skin, both in the secondary phase ($CeFeO_3$) and as a dopant, eventually lowering the bandgap of the material, increasing	[29]

		the oxidizing/fuel ratio. The mixture is heated at a high power of 800 W and a frequency of 2450 MHz, for a maximum of 3 minutes. Results in an exothermic reaction with a continuous flame for 24 seconds.	the reducibility of the sample.	
Cerium nitrate hexahydrate (oxidizing agent) and glycine (fuel)	Solution combustion method (SCS)	Cerium nitrate hexahydrate ($Ce(NO_3)_3 \cdot 6H_2O$) and glycine (CH_2NH_2COOH) were weighed in the required molar ratios and mixed in the minimum volume of deionized water to obtain a basic aqueous solution. This solution was after thermal dehydration (at 80°C) on a hot plate for 1 hour to remove excess solvent and put into a preheated muffle furnace at 400°C. The obtained powder was calcined at 500°C for 2 hours to remove the remaining undigested glycine and nitrate, finally, pure Ce crystals were obtained.	Obtained good quality and pure ceria crystal pure. The powder sample is pressed evenly in a hydraulic machine into pellets with a thickness of 0.5–2 mm and a diameter of 12 mm	[30]
Cerium nitrat hexahydrate (Ce ($NO_3)_3.6H_2O$)	Solution combustion method (SCS)	The cerium nitrate hexahydrate ($Ce(NO_3)_3 \cdot 6H_2O$) salt with	XRD: The synthesized CeO_2 nanoparticles had a	[31]

		the appropriate amount of urea is dissolved in distilled water in the ratio of fuel to the oxidizing agent. When the temperature reaches 1000ºC, the water starts to boil and evaporates from the solution which increases the viscosity of the solution significantly. Then the powder was calcined for two hours at a temperature of 6000ºC. The calcined sample produced a yellowish-white CeO_2 nanoparticle powder.	pure cubic fluorite structure with crystal sizes before and after calcination of 10.48 nm and 13.33 nm, respectively.	

3.1. Sol-gel method

The sol-gel method is the process of forming inorganic compounds through chemical reactions in solution at low temperatures. In this process, there is also a phase change from colloidal suspension (sol) to form a continuous liquid phase (gel). After the gel is formed, ripening will occur which can occur in a few days. Finally, the gel will be processed at high temperatures to decompose organic compounds and remove volatile reagents to produce nanoparticles.

From previous research, Ferreira [4] using the sol-gel method. With material Cerium nitrate ($Ce(NO_3)_2.6H_2O$; 0.5 M) and cassava starch extract. The prepared CeO_2 nanoparticles were analyzed by powder x-ray diffraction, thermogravimetric and differential thermal analysis, and high resolution scanning electron microscopy. Fig 1 shows an analysis chart with x-ray powder diffraction (XRD) in the range of 20-80°(2θ) at a temperature of 200°C.

In addition, the morphology and particle size of CeO_2 nanoparticles were evaluated by TEM. TEM images with different magnifications show that CeO_2 NPs have a relatively uniform shape, as shown in Fig 2 (A-B). The TEM image shows a spherical morphology with a face-centered cubital phase of CeO_2 nanoparticles with an average particle size of 51.6 nm.

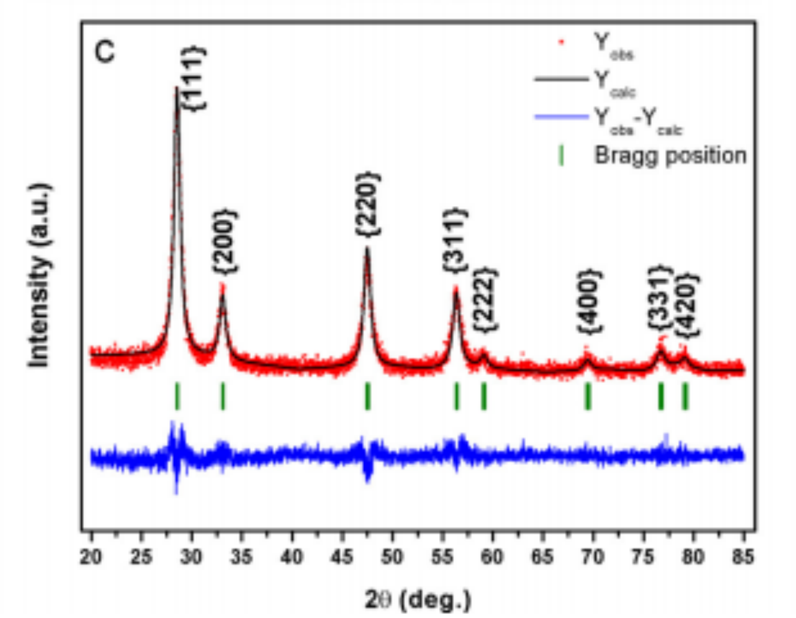

Fig 1. XRD patterns of CeO_2 nanoparticles calcined at different temperatures. The figure was adopted from Ferreira [4].

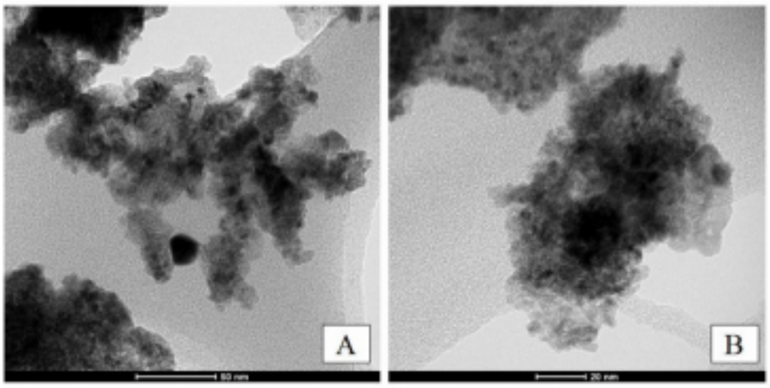

Fig 2. (A-B) TEM images of CeO_2 nanoparticles at distinct magnification. The figure was adopted from Yulizar [5].

3.2. Aerosol (*pyrolysis spray*)

Spray pyrolysis is a process in which a nanoporous atomizer is used to spray or inject a solution containing a precursor onto a hot substrate in an oven to obtain a nanostructure, causing the precursor to decompose to form the desired final material. Nanostructure parameters (particle size, shape, thickness) was obtained by controlling the spray energy (gas inlet of the atomizer, gas flow pressure), the droplet size of the precursor, the spray duration, the distance between the spray gun and the substrate, and the oven and heating furnace to control. We used a manual or automatic control system for substrate temperature. If the parameters can be kept constant, spray pyrolysis is very useful for the preparation of fine nano-films and large-scale clusters.

Fig 3 shows the x-ray diffraction pattern of the sample at 750°C. This pattern is consistent with the characteristic peak of CeO_2, which has a CaF_2 structure and contains no impurity phase. The broad characteristics of the XRD peak indicate that it has nanometer crystals of cerium oxide. The yellow color of the prepared sample also excludes any remaining carbon components in the particles. It is reported that ceria from the aqueous precursor solution exhibits a higher degree of crystallinity than the polymer precursor solution.

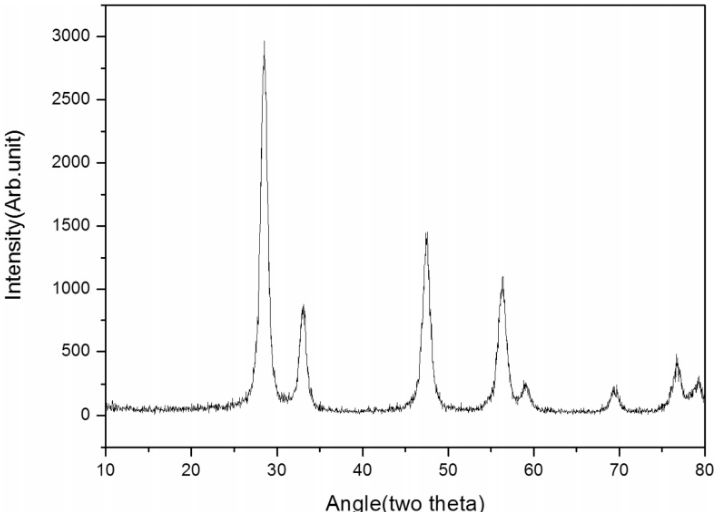

Fig 3. X-ray diffraction patterns of the sample. Figure was adopted from Kim [11].

Fig 4 shows that crystalline cerium oxide nanospheres were synthesized by ultrasonic spray pyrolysis of the cerium acetate precursor without any metal ion doping. Cerium oxide prepared at 750°C in an Ar (40%) / O_2 (60%) atmosphere has a spherical and solid morphology. The particle size range is from 100 nm to 1000 nm, and the average particle size is about 300 nm. However, the abrasive (precursor 1.5%) prepared in airflow of 750°C shows some porous agglomerated structure for larger particles, consisting of small nanoparticles smaller than 10 nm. The abrasive prepared from a lower solution concentration (0.15%) showed a similar particle size and a more uniform particle size distribution. As shown in Fig 3, when the precursor concentration is changed from 1.5% to 0.15%, the average particle size does not show a significant change (~130 nm) because the sintering between the necked primary particles is determined during the post-drying process stage.

The high-resolution TEM image (Fig 5) clearly shows that each particle is not agglomerated and the shape is close to the ideal nanosphere. As shown in the HRTEM image, the sample prepared in the Ar/O2 flow shows a solid spherical shape. On the other hand, the ceria prepared in the airflow is a porous agglomerate of nanoparticles (<10 nm) image from FESEM. The cerium oxide spheres prepared in the air showed a more porous structure, in which the primary crystal grains were loosely bound together.

This is very consistent with the fact that in FESEM (Fig 4), it is more frequently found that the density is lower, small, and agglomerated nanoclusters (Fig 4). Particles with a particle size of approximately 1 μm.

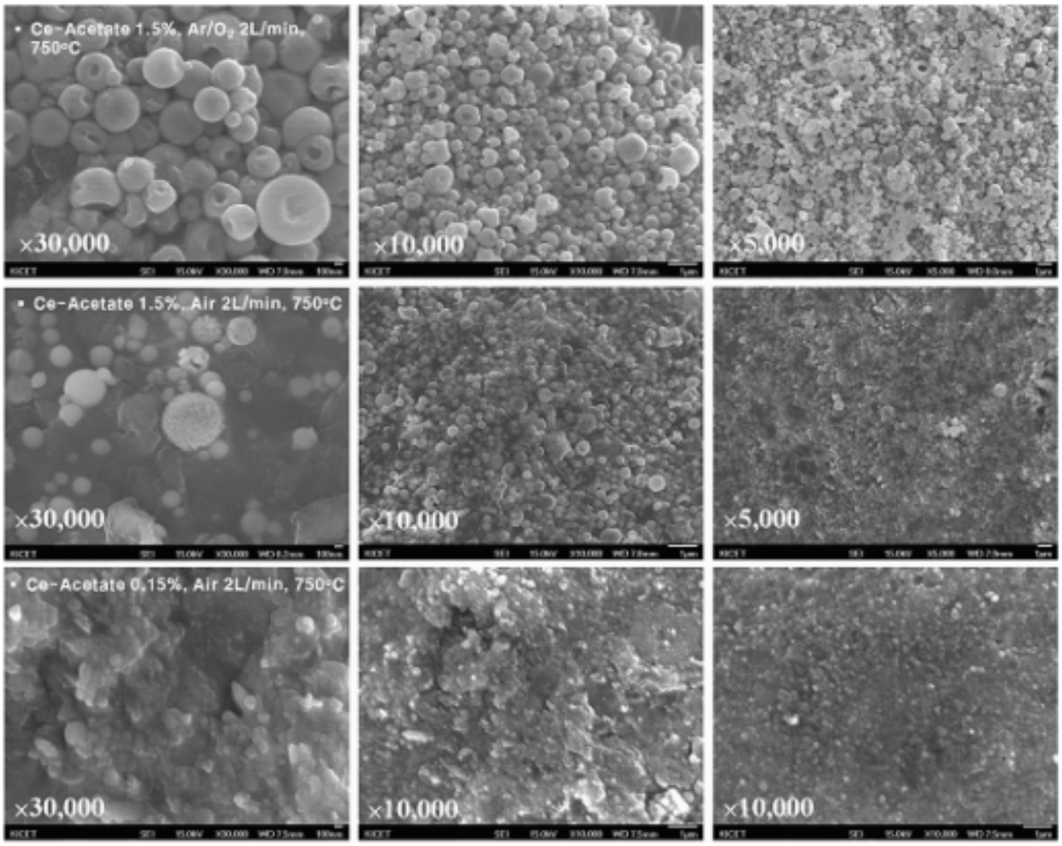

Fig 4. FE-SEM image of the sample. The figure was adopted from Kim [11].

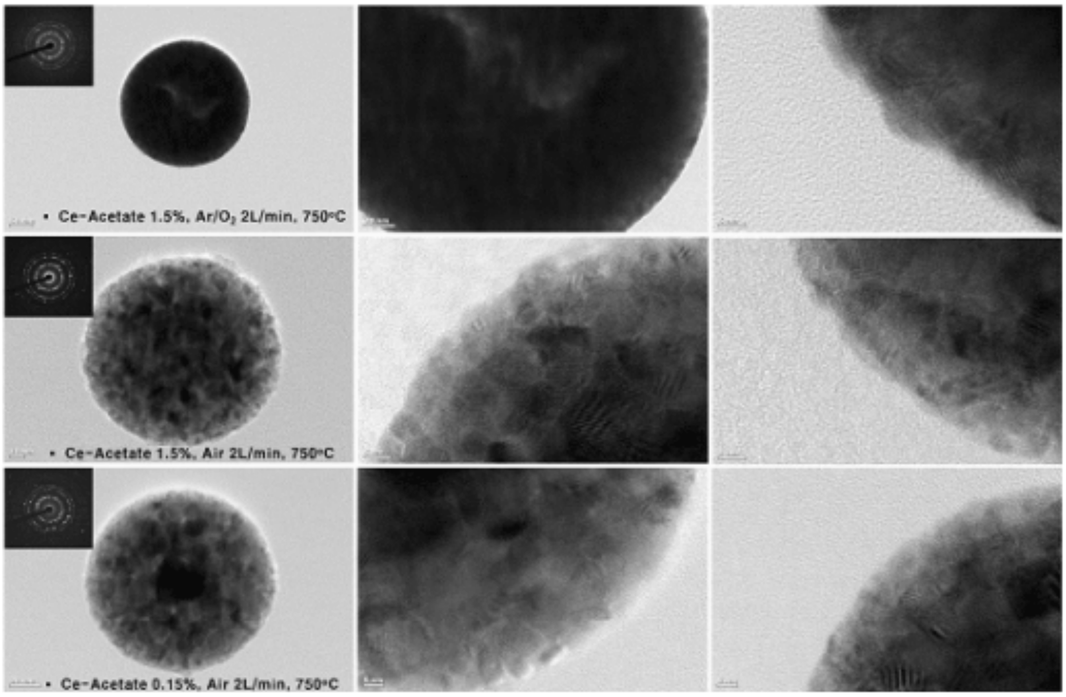

Fig 5. TEM image of the sample. The figure was adopted from Kim [11].

3.3 Solvothermal method

A solvothermal process is a process inducing a decomposition or a chemical reaction(s) between precursor(s) in a closed reaction vessel with the presence of a solvent at a temperature higher than the boiling temperature of the solvent. The pressure can be imposed (the pressure value is higher than 1 bar (105 Pa) or autogenous (the pressure value depends on the filling of the reaction vessel) at the establishment point of the experiment through the compression of the reaction medium). The solvothermal system can be heterogeneous or homogeneous and in subcritical or supercritical conditions depending on the experimental conditions (pressure and temperature).

During the solvothermal process, morphology and particle size of the CeO_2 nanoparticles could be controlled, while the other processes have risks such as some inevitable severe agglomeration caused by high-temperature sintering, but it could be avoided during the solvothermal process. Holding these two merits, the nano-CeO_2 prepared by the solvothermal method could have a large surface and well crystallinity.

The XRD patterns of nanoCeO_2 obtained with different solvation heat treatment times are shown in Fig 6. It can be seen that as the reaction time increases, the characteristic peaks of cubic fluorite CeO_2 acquire more shape and density. In addition to the planes representing (111), (220), and (311) crystals except for the three parts of the moderate slope amplitude, the diffraction peak of the precursor without any time. The reaction sample exhibits a wide concave-convex curve. After 2 hours of solvothermal reaction, the strength of (111), (220), and (311) crystalline planes improved significantly, and (200) crystalline planes appeared in the XRD pattern. After extending the reaction time to 6 hours, the diffraction peaks no longer change significantly. And when the reaction time is increased to 10 h, all these peaks show greater intensity, and the crystal face peak (222) appears. In addition, the peaks (400) and (331) also appeared in the XRD pattern. With the solvothermal reaction time of 18h, all diffraction peaks became more patterned and higher, especially the crystal planes (400) and (the intensity of the diffraction peak of 331) at the high angles of 69.40 and 76.68 at 2h increased significantly, indicating that the crystallinity of nanometer CeO_2 increased. In addition, no additional phase was detected in any of the samples obtained, indicating that high-purity nanoCeO_2 can be prepared by this process. After 20 hours of solvothermal reaction, these diffraction peaks did not show any more obvious changes. Therefore, it can be concluded that in 18 hours CeO_2 can be obtained with good crystallinity. In addition, Fig 6 also shows the XRD pattern of precursor calcined in air for 2 hours. The crystallinity of the solvothermal sample is better than that of the calcined sample. In addition, the specific surface area of the calcined is only 60.03 m^2/g. This means that the active nano interface of the solvothermal sample is better than that of the calcined.

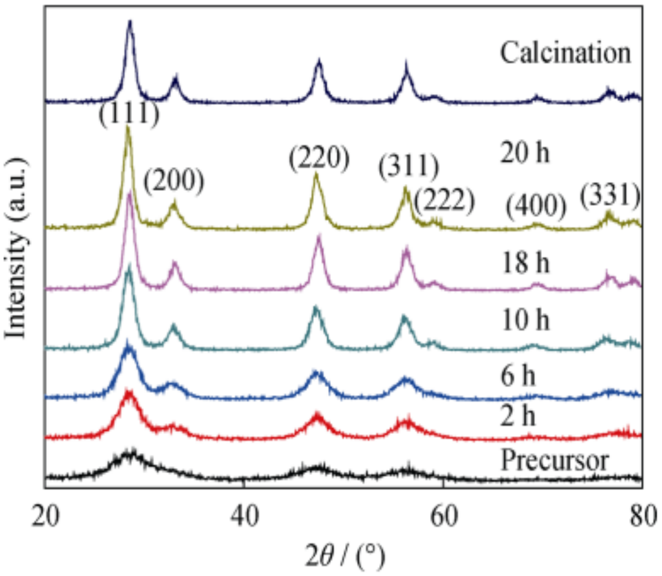

Fig 6. XRD synthesis result of sample. Figure was adopted from Pang [17].

Fig 7 shows the SEM image of the CeO_2 sample obtained by the 18-hour solvothermal process. The nanoparticles show granular facets, and the relatively regular small particles adhere to each other to form a weakly agglomerated spherical structure. The aggregates formed by these small particles have a microporous structure.

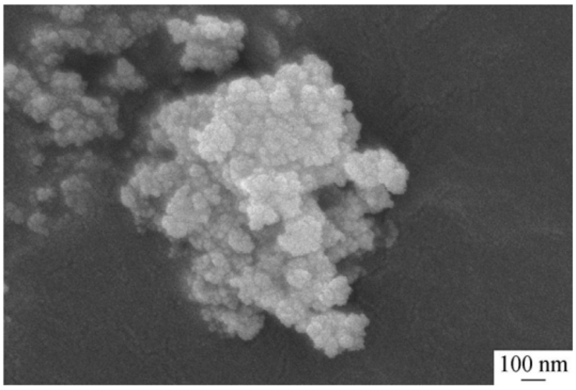

Fig 7. SEM image of the sample. The figure was adopted from Pang [17].

3.4 Hydrothermal method

Hydrothermal engineering is becoming one of the most important tools for advanced material processing, mainly because of its advantages in processing nanostructured materials for various technological applications such as electronics, optoelectronics, catalysis, ceramics, magnetic data storage, biomedicine, biophotonics, etc. The hydrothermal technique not only helps in the processing of monodispersed and highly homogeneous nanoparticles but also acts as one of the most interesting techniques for processing nano-hybrid materials and nanocomposites [32]. In this paper cerium oxide

(CeO_2) nanoparticles (NP) have been hydrothermally synthesized in the presence of urea and studied the effect of particle size on chemical sensing and photocatalytic properties [24].

Jayakumar [21] used the hydrothermal method. Ingredients, cerium nitrate ($Ce(NO_3)_3.6H_2O$; 432.2 g/mol; 99.9% purity) and sodium hydroxide (NaOH; 40 g/mol; 99.9% purity). The prepared CeO_2 nanoparticles were analyzed by x-ray diffraction Powder, UV-visible spectroscopy, Fourier transform infrared spectroscopy, scanning electron microscope and high resolution scanning electron microscope, etc.

XRD analysis of the two samples of CeO_2 nanoparticles had a face-centered cubic (FCC) structure as shown in Fig 8. The peaks were indexed using JCPDS card no: 34-0394. Both samples of CeO_2 nanoparticles have a face-centered cubic structure with lattice parameters a = b = c = 5.411. The diffraction peaks were found at 28.56°, 33.08°, 47.47°, 56.36°, 59.08°, 69.40°, 76.70°, 79.07°, and 88.41° and showed the formation of nano-sized CeO_2. The absence of impurities indicates that pure CeO_2 was synthesized by the hydrothermal method.

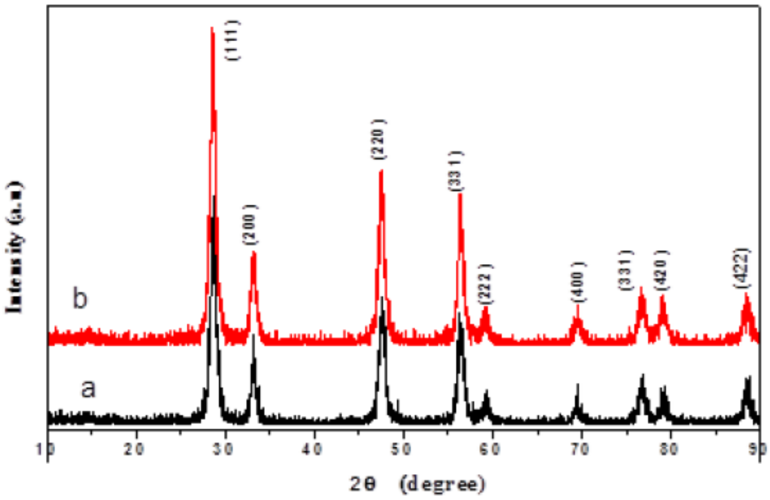

Fig 8. XRD patterns of CeO_2 nanoparticles prepared at (a) 12 hours and (b) 24 hours. The figure was adopted from Jayakumar [21].

Thus the results of the XRD analysis revealed that the CeO_2 nanoparticles had a face-centered cubic structure and had an average grain size of 12.8nm for 12 hours and 9.4nm for 24 hours.

UV-vis analysis the optical properties of the synthesized CeO_2 were studied by UV-Visible spectrophotometer and the results are shown in the wavelength Fig 9. The UV cutoff of the CeO_2 nanoparticles prepared at 12 h was 335 nm while the UV cutoff wavelength of the CeO_2 nanoparticles prepared at 24 h was observed at 352 nm. Moreover, the UV-visible spectrum did not show any other peaks associated with impurities and defects confirming that the nanoparticles were synthesized purely

CeO_2. The bandgap energy was 3.70 eV for CeO_2 nanoparticles prepared at 12 h whereas the band gap energy was found to be 3.52 eV for CeO_2 nanoparticles prepared at 24 h. The UV-visible spectra revealed that smaller size CeO_2 nanoparticles had better optical properties.

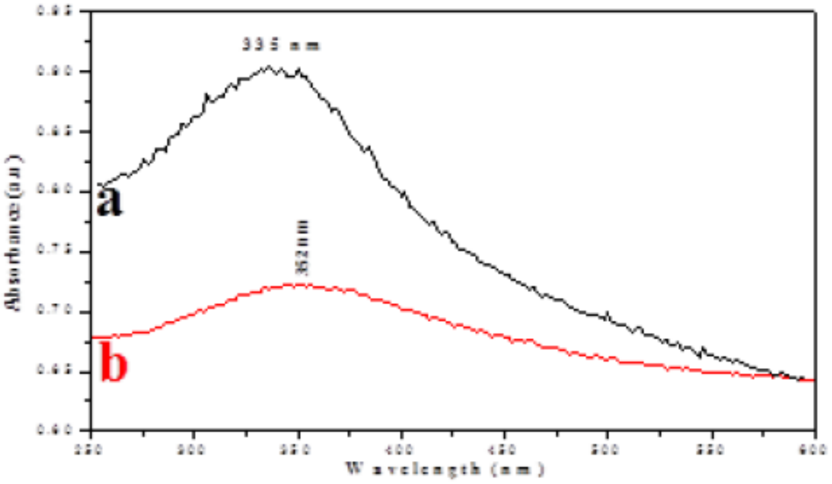

Fig 9. UV-vis spectra of CeO2 nanoparticles prepared at (a) 12 hours and (b) 24 hours. The figure was adopted from Jayakumar [21].

SEM analysis of the morphological results for the synthesized CeO_2 nanomaterial, as lysed by FESEM, are depicted in Fig 10 (a) and (b). Particles with an average size from 10 to 20 nm are visible in FESEM images at low and high magnification modes. For a deeper insight, the grown nanoparticles were analyzed via TEM and high-resolution TEM (HRTEM).

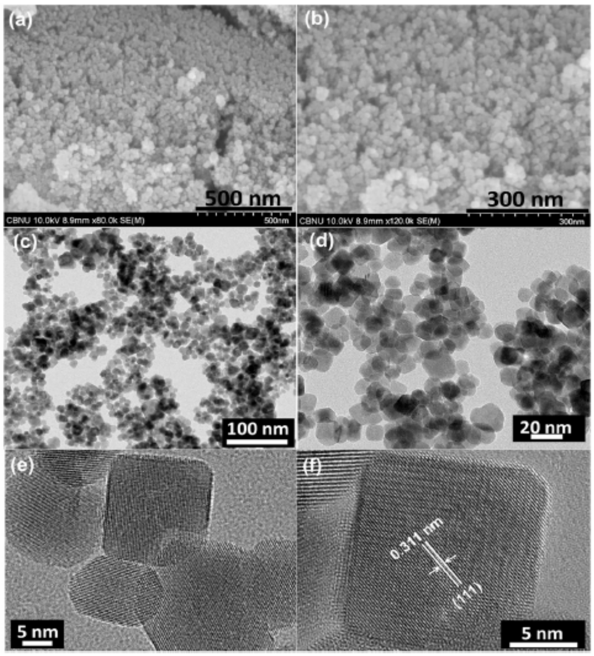

Fig 10. (a, b) FESEM and (c–f) TEM images of synthesized CeO_2 nanoparticles. The figure was adopted from Umar [23].

TEM images are shown in Fig 10 (c) and (d). while high-resolution TEM images of CeO_2 nanoparticles are shown in Figs 10 (e) and (f). The nanoparticle agglomeration was also observed presented similar to the FESEM analysis. The mean particle diameter was found to be 15 ± 2 nm from the TEM images. Nanoparticles with various capable morphologies such as blunt-edged squares, cubes and pentagons, spheres, and elongated hexagons can be seen from the HRTEM images (Figs 10 (e) and (f)). HRTEM further reveals a well-defined 2D latitude plane having a distance of d 0.311 nm.

3.5. Solution combustion method (SCS)

A series of CuO/CeO_2 catalysts were successfully synthesized via the solution combustion method (SCS) using different fuels and tested for CO oxidation. The catalyst is characterized by energy dispersion x-ray analysis (EDXA), x-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), scanning electron microenvironment (SEM), N_2 adsorption-desorption isotherm, and H_2 temperature-programmed reduction (H2-TPR).

XRD analysis, Fig 11 shows the XRD pattern of the synthesis of Ceria nanoparticles measured from the solution burning process. All diffraction peaks in the XRD pattern were well indexed to the (111) (200) (220) (311) (222) (400) and (331) cubic flux planes of the CeO_2 rite structure. The lattice parameter determined from the reflection (111) of the pure CeO_2 NP sample was a = 5,412, which consisted of bulk CeO_2 (a = 5 411). From the XRD pattern, it was observed that no other characteristics were found due to impurities indicating that the precursor had been systematically oxidized to pure CeO_2 after being calcined at 500°C in air. The reflection observed in the XRD pattern is a very sharp peak which confirms the formation of fine particles and small size properties of the obtained material.

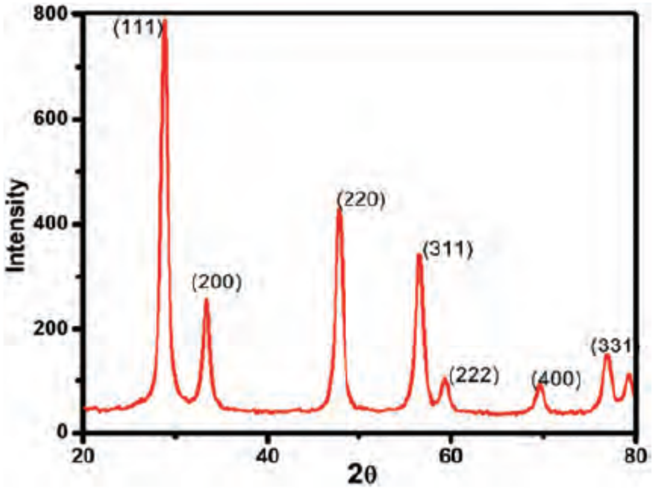

Fig 11. XRD spectra of the CeO_2 NPs. The figure was adopted from Harish [30].

SEM analysis, Fig 12 shows SEM micrographs with a resolution of 2 m for all catalysts. Based on SEM data, it was shown that the type of fuel used showed a strong effect on the morphology of the combustion products: spherical structure building structures formed for CuO/CeO_2 urea, glycine, urotropin catalyst; Sponge-like morphology was observed using ascorbic acid, citric acid, and glucose as fuel. A more uniform morphology was obtained by using urea. All types are formed by agglomerated isotropic nanoparticles in micron-sized structures. The amount of gas produced during the reactions of each of the 6 syntheses was different as observed by us during the experiment, which could have resulted in the formation of different porous, foamy products.

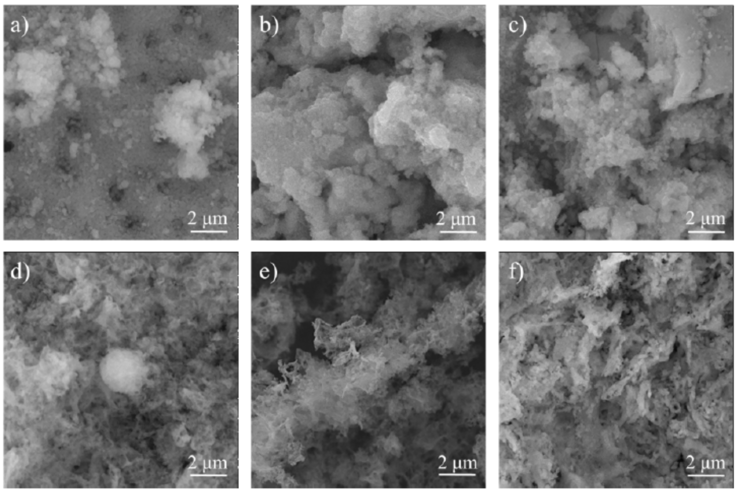

Fig 12. SEM micrographs of CeO_2/CuO nanocomposites with the following fuels used: a) urea; b) glycine; c) urotropine; d) ascorbic acid; e) citric acid; f) glucose. The figure was adopted from Cam [26]

Conclusion

CeO_2 synthesis can be done by several methods such as sol-gel, hydrothermal, mechanochemical processing, solution combustion method, aerosol (pyrolysis spray), and solvothermal methods. For these methods, the most efficient way to synthesize CeO_2 is the hydrothermal method. Because this method is cheaper, uses relatively low temperatures, has fast reaction times, reproducible particle size distribution, homogeneity, and greater electrochemical performance, and is more environmentally friendly.

Acknowledgments

We would like to thank Bangdos, Universitas Pendidikan Indonesia.

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(2021) ; www.mocedes.org/ajcer