Effect of precursor type on Optical, grain sizes and Structural Properties of Zinc Oxide Nanoparticles using Cassia leaf extract as capping and Reducing agent

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Abstract: Because of its special qualities and uses in optoelectronic devices, zinc oxide nanoparticles have drawn the attention of researchers in recent years. At ambient temperature, zinc oxide nanoparticles have a very significant excitation binding energy of 60 meV and band gap energy of 3.37 eV. It is also environmentally benign, non-toxic, and transparent to the visible spectrum. Zinc acetate, zinc chloride, and zinc sulphate precursors were used in the current study to create zinc oxide nanoparticles utilizing a green technique. Cassia leaf extract was used as a precursor and capping agent. Distilled (DW) water was utilized as the solvent, and 1M sodium hydroxide was added in drops to get the pH to 11.6. The produced zinc oxide nanoparticles' morphology and structure were examined by the use of X-ray diffraction (XRD), spectroscopy, and scanning electron microscopy.

Key-words: Precursor, Grain size, Nanoparticles, Cassia Plant, Capping agent, Reducing agent and Zinc Oxide

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1. Introduction

Since it was developed as a startling discovery in the world of things on the nanoscale between 1 and 100 nm, nanotechnology is the most dynamic field of present scientific success in materials science (Gnanabangetha and Suresh, 2020). Due to its potential and the wide range of applications that may be achieved when handling many materials at the nanoscale, nanotechnology has brought about a paradigm shift in life as a result of recent advancements in nanoscale materials (Demissie et al., 2020). Thus, it is safe to say that in the current technological era, nanotechnology has greatly expanded and advanced many technologies (Hajiashrafi et al., 2015).Nanoparticles have unique properties, like a large surface area to volume ratio, when compared to their bulk counterparts, which makes them an excellent choice for operation-oriented applications. The main effect of size on nanoparticles is electron confinement, as noted by Sierra, Harrera, and Ojeda (2018), because the influence of nanoparticle size and surface area became more pronounced or stringent as the sizes reduced.

A variety of chemical techniques, such as thermal evaporation, microwave method, sol-gel processing, homogeneous precipitation, organometallic synthesis, and green method, have been employed to create nanoparticles. With the exception of green, all of these techniques have been shown to have the potential to be harmful to both humans and the environment because they involve the release of hazardous byproducts into the environment, the use of complex, challenging-to-operate equipment, high temperatures and pressures, high costs or financial implications, and, lastly, the loss of medical applications for nanoparticles due to the risk of poisoning from absorbing toxic substances on their surface. (Denmez, 2020). The synthesis of green

nanoparticles has gained recognition as a competitive substitute for conventional techniques. According to Balogun et al. (2020), green synthesis employing plant extract is easy to implement and advantageous for the environment because it does not involve high pressure, heat, energy, or hazardous materials during the synthesis processes (Ossai et al., 2020). The primary characteristics of the conventional techniques for synthesizing nanoparticles (NPs), their spherical shape, and their antibacterial activity, according to Hajiashrafi et al. (2018), depended on temperature and time. Tomato, onion, cabbage, and carrot extracts are used by Degefa et al. (2021) as stabilizing and reducing agents to create ZnO NPs with a single-phase hexagonal form with average particle sizes of 17 nm, 18 nm, 24 nm, and 15 nm using the same precursor, According to Bandeira et al. (2018), the amounts of the biological extract and zinc precursors significantly affect the final properties of the manufactured ZnO nanoparticles. Wafula et al. (2020) produced ZnO-NPs with a crystalline size of 20.91 nm by bio-reduction of Zn2+ using TDLE in an environmentally friendly manner. The antibacterial capabilities of the particles appear promising. Noorjahan et al. (2015) also use neem leaf extract to make zinc oxide nanoparticles. Their SEM research shows, nanoflakes and spindle-shaped nanoparticles as small as 50 nm have been formed. Owing to its many environmentally beneficial qualities, it has been studied as a potent catalyst for a range of organic transformations. Taherian et al., (2018) use Satureja plants to synthesize green nanoparticles of zinc oxide. They chose Satureja because it contains chemical compounds that are responsible for antioxidant and regenerative activities. They obtain crystalline nanoparticles with spherical shapes. XRD confirmed the average particle diameter to be 35.88 nm which proved to be low-cost, simple, low-risk, and environmentally friendly, capable of producing nanoparticles of appropriate size, and should be used instead of environmentally hazardous chemical methods. In another separate study, Vaishnav et al., (2017) produced ZnO nanoparticles from Celosia argentea leaf extract. The average size of the nanoparticles synthesized was 25 nm. The antibacterial activity of the particles against E.coli, Salmonella, and Acetobacter bacteria were comparatively good against Salmonella, but moderate against E. coli and Acetobacter. Zinc oxide nanoparticles were also found to have anti-inflammatory and anti-cancer properties in their study. Kaningini et al., (2020) used Athrixiaphylicoides natural extract as a reducing agent to synthesize ZnO nanoparticles. According to XRD and UV-Vis analysis, synthesized ZnO nanoparticles have a spherical shape with an average crystallite size of 24 nm.
Thus, there was no single clear factor for controlling the size of the nanoparticles, which directly controls their electrical and optical properties. This study will look at the effect of types of precursors on the size, electrical properties, and optical properties of Zinc Oxide nanoparticles synthesized via the Green method using three different precursors (Zinc Acetate, Zinc Chloride, and Zinc Sulphate) using cassia leaves as reducing and capping agent. This study will look at the effect of precursor types on the size, electrical properties, and optical properties of Zinc Oxide nanoparticles synthesized via the Green method using 4g/100 cm^3 of four different precursors (Zinc Nitrate hexahydrate $Zn(NO_3).6H_2O$, Zinc acetate dehydrate $Zn(C_4H_6O_4).2H_2O$, Zinc Chloride $ZnCl_2$ and Zinc Sulphate heptahydrate $ZnSO_4.7H_2O$ using Jatropha leaves as reducing and capping agent.

2. Materials and Methods

For the synthesis of nanoparticles from the Cassia leaf, three Zinc precursors, Zinc acetate dehydrate$Zn(C_4H_6O_4).2H_2O$ (219 g), Zinc Chloride $ZnCl_2$ (126 g) and Zinc Sulphate heptahydrate $ZnSO_4.7H_2O$ (291 g) and triple distilled water were used. All important

materials are cleaned by using nitric acid and additionally by deionized water, and then dehydrated by keeping it in a hot air oven before the preparation of nanoparticles. The leaves of the cassia plant were gathered from Shaffa Village in Hawul Borno state, Nigeria. All the synthesis processes were carried out in SHESTCO Chemistry Advanced Research Lab. Sheda Abuja Nigeria

3. Preparation of Extraction from Cassia Plant Leaves

The leaves of the Cassia plant (Figure 1) are collected in the Hawul local Government area of Borno state, Nigeria, and washed by using warm water to eradicate dirt adverts. The leaves dried in the air after three weeks since the season in which the present research was conducted was summer; after drying, the leaves were powdered by using a metal mortar and wood pestle till they ground very well. The extraction of Cassia leaves was done by measuring 20 g of powder of Cassia leaves which was put into 20 milliliters of distilled water at an adjusted temperature of 60°C for 30min, and the pH value of the solution was measured to be 5. The solution was finally filtered and kept in a freezer at 7°C for further work as enumerated in Figure 1

(a). Cassia Leaves

b) Cassia Leaves Powder

(c) Cassia Leaves solution

Figure 1: a) Cassia Plant b) Cassia leaves Powder and C) Cassia Leaves Solution

where figure 1(a) show the cassia plant, 1(b) show the cassia powder which was grounded and ready for extraction and finally 1(c) is the filtrate or extracted solution from the plan ready for green synthesis of ZnO nanoparticles.

4. Synthesis of ZnO Nanoparticles from Cassia Leaf Extraction

The precursor basis for the zinc ion used in this study were Zinc acetate dehydrate, Zinc Chloride and Zinc Sulphate heptahydrate, which was taken from chemical shops from Abuja, Nigeria. The Zinc salts (Zinc acetate dehydrate, Zinc Chloride and Zinc Sulphate heptahydrate) were dissolved in deionized water. For synthesis of ZnO nanoparticles, Conical flask volume of 500 ml, 200 ml of the source of zinc (Zinc Acetate) (50 g/dm^3) was mixed with 20 ml of the leaf extract of the Cassia leaf and PH of the solution adjusted to 11.6 by addition of drops of 1 molar solution of sodium hydroxide then stimulated on a magnetic stirrer heated at 70° C, and the stirring was nonstop for 2 hours to allowed formation of uniform solution. The homogenous solution was allowed to overnight then filtered and dry in hot air oven at the temperature of 100-120°C for 60 min. It was then calcined in furnace for two hours at temperature of 550°C, the color of prepared nanoparticles is yellowish and is crumpled in a metallic mortar and pestle to get a green prepared of ZnO nanoparticles.

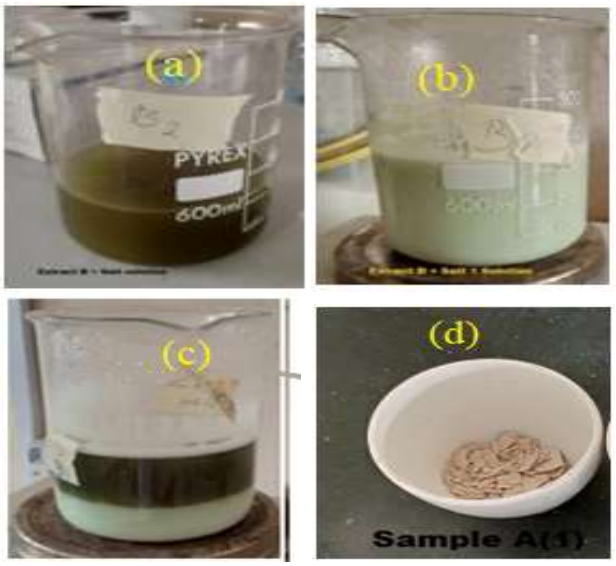


Figure 2: Steps in Synthesis of ZnO Nanoparticles using Cassia Plant Extract

The Nanoparticles obtaied was label as CCC (1), this procedure was repeated for the other precursors (Zinc Chloride and Sulphate heptahydrate) all at concetration of 50 g/dm^3, where CCC (2) is from Zinc Chloride and CCC (3) is from Zinc Sulphate heptahydrate

4.1 Characterization of synthesized ZnO Nanoparticles

(i) *Optical Properties:*

This characterization was carried out in Chemistry Advanced Research Laboratory SHESTCO Using UV-Vis. Spectrophotometer. The Synthesized nanoparticles were ground into fine powder. A very small mass of the powder was suspended in solution of water and ethanol in the ratio of 2:1 in a standard cuvette of length 1 cm. The second cuvette contains only the solution without any suspension then absorption of the nanoparticles was scanned within the range of 190 nm to 1100 nm. The result was displayed on a computer attached to the spectrophotometer. Using Origin Lab Software and CSV file of the raw data, the graphs of wavelength versus absorption of light by the nanoparticles were plotted. The wavelength at which the highest absorption take place within the 300 nm 450 nm range for ZnO nanoparticle was measured by the help or Origin Lab software. Therefore, maximum absorption wavelength λ_{max} and the band gaps of the entire three (3) nanoparticles were calculated using the equation (1).

$$E_g = \frac{1240}{\lambda_{max.}} \ (eV \qquad (1)$$

(ii). Structural Properties

The three Nanoparticles synthesized were sent for XRD analysis at Kaduna Geological survey center Kaduna State. The particles were scanned within the range of 2Ɵ is (0 - 70 degree). From the plot of intensity against the angle of diffraction, the half width at half maximum (HWHM) was calculated, and subsequently the grain sizes of the particles. Even though, the particles are not the same size, an average of the particles sizes was calculated. Also from the result of the XRD spectroscopy, the zinc sites (miler indices) were automatically or directly read from the machine output. The particles sizes were obtained using calculation for the 101 peak using the Debye - Scherrer's formula (Talam et al. 2012)

$$D = \frac{0.9\lambda}{\beta cos\theta} \qquad (2)$$

where, λ is the x-ray wavelength (1.504 nm), θ is the Bragg diffraction angle, and β is the full width at half maximum.

(iii) Morphological Properties

The morphology of ZnO nanoparticles was examined by means of scanning electron microscopy (SEM) SU3500, Hitachi with spectral imaging system Thermo Scientific NSS (EDS) at Umaru Musa Yar'adua University Katsina, the tape of detector (BSE-3D), acceleration voltage (15.0 kV), working distance (11.6 mm), pressure (in the case of variable vacuum conditions, 40 Pa). The results of the SEM reveal the structures of particles while the EDS reveal the atomic and weight percentages of the element present in the nanoparticles.

5. Result and discussion

5.1: Optical Characterization

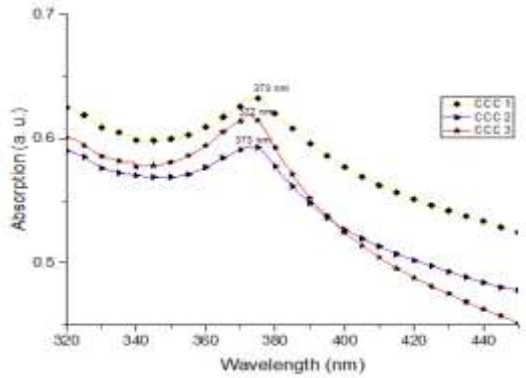


Figure 3. UV-Vis spectra of nanoparticles

Figure. 3 is the spectral lines obtained from the nanoparticles synthesis using 50 g/dm^3 of three different precursor and Cassia leaves as the reducing and capping agent. The result clear shows that, the different precursors have greatly improved the behavior of the absorption spectra of the particles Uv–Visible spectroscopy. For the different precursors there was a defined pattern of absorption with peak absorptions at 374 nm for CCC 1 and 372 nm and for both CCC2 and 373 nm for CCC3 respectively. The energy gap for the nanoparticles were found to be 3.33, 3.33 eV and 3.34 eV for Zinc acetate dehydrate, Zinc Chloride and Zinc Sulphate heptahydrate precursors respectively.

3.1: Structural Characterization

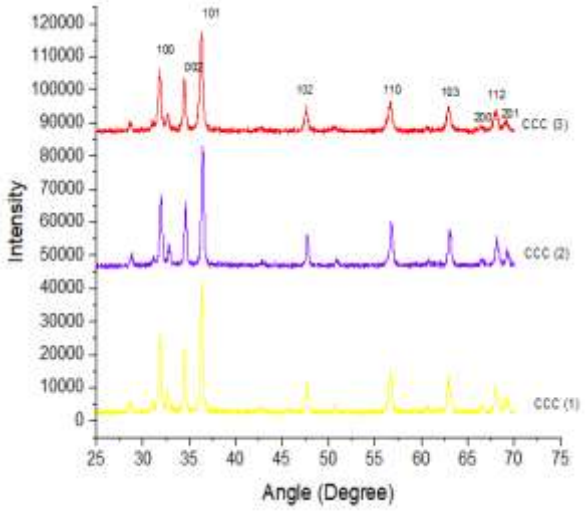


Figure 4: XRD spectra of the nanoparticles

From the above XRD spectra, it can be seen that, they seem to have similar pattern of spectra but their height differs which make the grain sizes different from each other, for sample CCC1 with a concentration of 50g/dm^3 of Zinc Acetate, the particle size was calcurate using Scherer's equation below:

$$D = \frac{k\lambda}{\beta \cos\theta} \quad (3)$$

Where: k = Scherer's constant (usually 0.9), λ = Wavelength of X-Ray source, Cu Kα radiation (1.5406), β = Full width at half-maximum (FWHM) of the diffraction peak in radian

θ = Bragg's diffraction angle and β obtained from the XRD data was converted to radian unit using the equation shown below:-

$$\beta = \frac{\text{FWHM in } 2\theta \text{ x } \pi}{180^O} \quad (4)$$

was found to be 24.22.87 nm similarly, CCC2 and CCC 3 with the same concentrations as Zinc Acetate using Zinc Chloride and Zinc Sulphate were found to have19.27 nm and 18.31 nm respectively. From the data obtain above it is clear that the sizes of the nanoparticles changes with types of precousor. This is not far away from the work of Barzinjy and Azeez (2020) where they biologically synthesized ZnO NPs from Eucalyptus globulus leaf extract for water pollution removal, solar cell fabrications and medical/cosmetic applications with spherical nanoparticles range from 27 to 35 nm grain sizes.

Table 1: Zincite and Coresponding 2θ for NPS CCC 1, CCC 2 and CCC3

CCC 1	2θ	31.812	34.51	36.313	47.603	56.593	62.916	66.349	68.025	69.07
	Zincite	100	002	101	102	110	103	200	112	201
CCC 2	2θ	31.928	34.604	36.421	47.684	56.745	62.97	66.454	68.06	69.15
	Zincite	100	002	101	102	110	103	200	112	201
CCC 3	2θ	31.799	34.449	36.291	47.6	56.525	62.83	67.93	69.02	
	Zincite	100	002	101	101	110	103	112	201	

According to Sahai and Goswami (2013) a hexagonal system like ZnO, lattice constants $a = b \neq c$ andcrystallographic axes$\alpha = \beta = 90°, \gamma = 120°$. In this case, the lattice constants

a *and* b were calculated for those XRD peaks for which l = 0 and lattice constant c were also calculated only for those XRD peaks for which h = k = 0. Based on these conditions, the equations below were used to calculate of a *and* b usinig data in table 1

The lattice constant a is given by Zak et al (2011) as

$$a = \frac{\lambda}{\sqrt{3}sin\theta} \quad (5)$$

The lattice constant a is given by by Zak et al (2011) and Taha et al. (2015) as

$$c = \frac{\lambda}{sin\theta} \quad (6)$$

The constant μ is displacement of each atom with respect to next atom along the axis 'c' was given by:

$$\mu = \frac{a^2}{3c^2} + 0.25 \quad (7)$$

The Zn–O bond length L is given by Sahai &Goswami as:

$$L = \left[\left(\frac{a^2}{3}\right) + \left(\frac{1}{2} - \mu\right)^2 c^2\right]^{1/2} \quad (8)$$

The dislocation density (δ), which defines the length of dislocation line per unit volume of the crystals were calculated by the equation below as in Sahai &Goswami (2011) and Yadav & Chauhan (2019) as

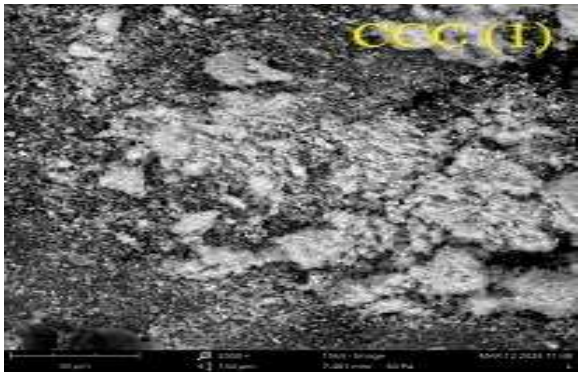
$$\delta = \frac{1}{D^2} \quad (9)$$

Table 2: Summary of a, c, c/a, μ and L for the 24 NPs CCC 1, CCC 2 and CCC 3

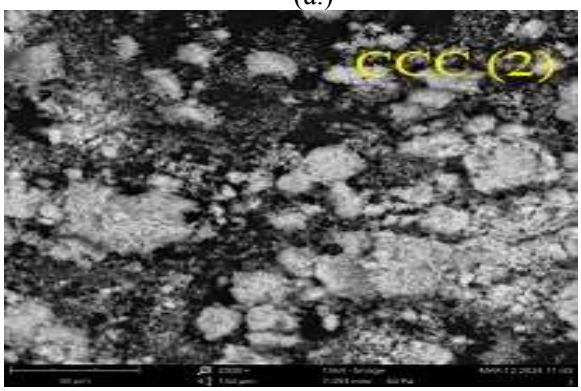
a	c	c/a	μ	L	D	δ
3.24504	5.1942	1.60066	0.3801	1.94897	24.22	0.0017
3.23442	5.18023	1.60159	0.37995	1.93695	19.27	0.00269
3.24623	5.20297	1.60278	0.37976	1.95203	18.31	0.00298

Table 2 present the summary of the parameters list above from the nanoparticles CCC 1 CCC 2 and CCC 3 where the parameters above confirm that particles are hexagonal wurzite

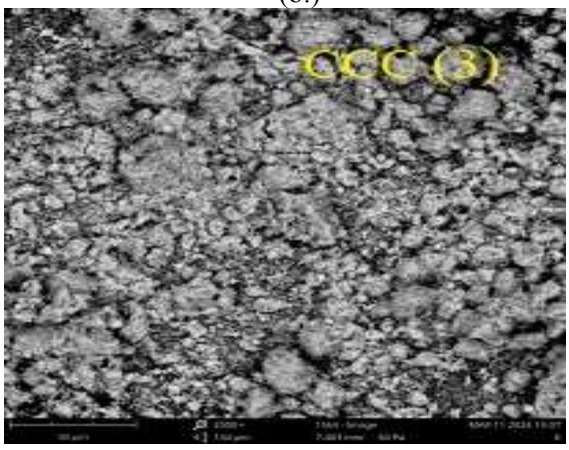
NPs	Elements →	Zn %	Al %	Si %	Mg %	S %	P %	Cl %
CCC (1	Atomic Conc.	88.56	3.21	2.05	1.49	0.85	0.28	2.42
	Weight Conc.	94.46	1.41	0.94	0.58	0.44	0.18	1.4
CCC (3)	Atomic Conc.	81.97	4.83	3.43	4.03	1.86	2.36	0.88
	Weight Conc.	91.25	2.22	1.64	1.67	1.01	1.25	0.53
CCC (4	Atomic Conc.	92.87	2.69	1.01	0.86	1.03	0.65	0.88
	Weight Conc.	96.67	1.15	0.45	0.33	0.52	0.32	0.38



(a.)



(b.)



(c.)

Figure 4: SEM images of the three nanoparticles.

Raha and Ahmaruzzaman (2022), from the finding of their research work, zinc oxide nanoparticles were synthesis from local materials from the environment which has many applications in the environment, among which includes transparent electronics, ultraviolet (UV) light emitters, piezoelectric devices, chemical sensors, opto-electronics, solar cells and spin electronics. Nomura et al., 2003 and Nakada et al., 2004, agree with the finding of this work due to nontoxic, nature of ZnO, they can be extensive use as an excellent photocatalyst for the degradation of a great many emerging organic pollutants. Due to the antibacterial and good antifungal activity, ZnO, the particles synthesis in this research work can be used in production of various raw materials used in medicine such as disinfectant agents and for dermatological applications. On the other hand, ZnO NPs because they absorb UVA and UVB radiations and can be used in sun protective creams just as reported some time ago by El-Diasty et al., 2013.

In the military sector where Water-repellent and self-cleaning fibers come handy in the military world where laundering is difficult and inconvenient with regard to both time and scope Zinc Oxide when incorporated into the fabric can preventing undesirable stains (Zhang and Yang 2009). ZnO NPs ca be use as vehicles for gene delivery, importing doxorubicin (DOX) into cancer cells and efficient gene targeting to the recipient tissues such as tumor cells (Raghupathi et al. 2011; Yuan et al., 2010; Zhang and Liu, 2010; Taylor and Webster, 2011; Asharani et al., 2008) last but not the least, ZnO NPs can increase the growth and yield of food crops they induced considerable stimulation of seed germination, seedling vigor, and stem and root growth food crop like maize as observed in this research and peanuts as reported in Prasad et al., 2012

6. Conclusion

The synthesis of Zinc Oxide nanoparticles was done using green method to investigate the effect of the precursor types on the sizes of the particles. So in the present paper the production ZnO nanoparticles were carried out by green synthesis. Cassia leaves extract was used as capping agent while the precursors used were Zinc Acetate salt ($Zn(NO_3)_2.2H_2O$) (219 g molar mass), Zinc Chloride $ZnCl_2$ (126 g) and Zinc Sulphate heptahydrate $ZnSO_4 .7H_2O$ (291 g) and triple distilled water were used with constant concentration of 50g/dm^3. The biological syntheses of zinc nanoparticles using leaf extract of Cassia provide an environmental friendly, simple and efficient route for synthesis of nanoparticles. The use of plant extract avoids usage of harmful and toxic reducing and stabilizing agents. The characterization of ZnO nanoparticles were carried out using different techniques like XRD, SEM and UV-Vis etc. From the results above it is clear that, size of the nanoparticles are affected by then types of the precursor because with Zinc Acetate the average size of the nanoparticle is 24.22 nm, with Zinc Chloride the particles sizes was 17.27 .87nm and finally with Zinc Sulphate heptahydrate, the particles sizes was 18.31 nm respectively.

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The authors equally contributed in the present research, at all stages from the formulation of the problem to the final findings and solution.

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Conflict of Interest

The authors have no conflicts of interest to declare that are relevant to the content of this article.