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SYNTHESIS AND STRUCTURAL CHARACTERIZATION OF ZNO NANOPARTICLES

In the present work, zinc oxide (ZnO) nanoparticles were synthesized by direct precipitation method in aqueous solution using zinc acetate dihydrate and sodium hydroxide as precursors. The molar ratio of Zn^{2+} to OH^- was 1:2. The obtained precipitated compound was treated at different temperatures. The crystal phase and structural parameters of each prepared ZnO samples such as interplanar spacing, crystallite size, dislocation density, micro strain were determined by X-ray diffraction (XRD) analysis for different crystallographic planes. Other crystallite parameters such as lattice constants, unit cell volume, Zn–O bond length, crystallinity of synthesized ZnO samples also were calculated from the XRD data. The XRD patterns show the successfully synthesized ZnO phase with wurtzite hexagonal structure and average crystallite sizes of 24.6 nm, 25.6 nm and 28.1 nm for the samples that dried at room temperature without heat treatment (S1), dried at 60°C without calcination (S2), dried at 60°C and calcinated at 300°C in air atmosphere for 2 hours (S3) respectively. The calculated structural parameters of the synthesized ZnO nanoparticles are in good agreement with the standard reported data (JCPDS 36-1451). The effect of heat treatment on the structural parameters of synthesized ZnO nanoparticles was examined and it indicates that increasing the thermal decomposition temperature improves the quality of crystalline material. The XRD data show that the synthesized S3 sample was free of impurities and characterized by lattice constants $a = 3.252 \text{ \AA}$, $c = 5.210 \text{ \AA}$, unit cell volume $V = 47.703 \text{ \AA}^3$, bond length Zn–O = 1.9789 Å and crystallinity 93%. The results are of practical value for simple synthesis of highly dispersed forms of ZnO and its possible application in textile finishing.

Keywords: ZnO, nanoparticles, precipitation, structure, XRD.

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СИНТЕЗ І СТРУКТУРНА ХАРАКТЕРИСТИКА НАНОЧАСТИНОК ZNO

У даній роботі наночастинки цинку оксиду (ZnO) синтезовані методом прямого осадження у водному розчині з використанням цинку ацетату дигідрату і натрію гідроксиду як прекурсорів. Молярне співвідношення Zn^{2+} до OH^- складало 1:2. Отриманий осад обробляли при різних температурах. Кристалічну фазу і структурні параметри кожного отриманого зразка, такі як міжплощинна відстань, розмір кристалітів, щільність дислокацій, мікронапруження, визначали за допомогою рентгеноструктурного аналізу (XRD) для різних кристаліграфічних площин. Інші параметри кристаліту, такі як константи решітки, об'єм елементарної комірки, довжина зв'язку Zn–O, кристалічність синтезованих зразків ZnO, також розраховували за даними XRD. Рентгенограми демонструють успішно синтезовану фазу ZnO з гексагональною структурою вюрцити і середніми розмірами кристалітів 24,6 нм, 25,6 нм і 28,1 нм для зразків, висушених при кімнатній температурі без термообробки (S1), висушених при 60°C без прожарювання (S2), висушених при 60°C і оброблених при 300°C в атмосфері повітря протягом 2 год (S3), відповідно. Розраховані структурні параметри синтезованих наночастинок ZnO добре узгоджуються зі стандартними звітними даними (JCPDS 36-1451). Досліджено вплив термічної обробки на структурні параметри синтезованих наночастинок ZnO, який свідчить про те, що підвищення температури термічного розкладу покращує якість кристалічного матеріалу. Дані XRD показують, що синтезований зразок S3 не містить домішок і характеризується параметрами решітки $a = 3.252 \text{ \AA}$, $c = 5.210 \text{ \AA}$, об'ємом елементарної комірки $V = 47.703 \text{ \AA}^3$, довжиною зв'язку Zn–O = 1.9789 Å і кристалічністю 93%. Результати мають практичне значення для простого синтезу високодисперсних форм ZnO і їх можливого застосування в опорядженні текстилю.

Ключові слова: ZnO, наночастинки, осадження, структура, XRD.

Introduction

ZnO nanoparticles are semiconductor materials, which have been widespread used due to their unique physical and chemical properties, such as surface effect, quantum size effect and macroscopic quantum effect [1].

The application of ZnO nanoparticles to textile has attracted extensive attentions due to the advantages of ZnO nanoparticles, such as UV-shielding, antibacterial activity, biocompatible, biodegradable and biosafety for environmental applications.

Emerging studies on functionalized cotton fibers modified with ZnO nanoparticles expanded the use of cotton fabrics for various applications, such as conductive [2], magnetic [3], photocatalytic [4], antimicrobial [5, 6], super hydrophobic [7] and fire retardant [8, 9] textiles.

Several in situ or ex situ approaches have been introduced for the incorporation of nanoparticles into textile fibers, in which the nanoparticles are synthesized in the presence of the fibers (in situ) or beforehand (ex situ) and then applied to the fibers [10].

Several ZnO synthesis routes have been described in the literature with different morphologies and excellent characteristics, including chemical precipitation [11], solvothermal [12], sol-gel processing [13], thermal decomposition [14], microwave irradiation [15], chemical bath deposition [16], dip coating [17], spin coating [18], electrical deposition [17], and hydrothermal processing [19]. Among these methods, chemical precipitation provides a practical way for low cost and large-scale production, which does not need expensive raw materials and is expected to realize industrialization.

During the synthesis of nanoparticles, it should be taken into account that the growth and the morphology of nano-ZnO are strongly affected by precursors concentration, pH of synthesis mixture, synthesis temperature, reaction time and heat treatment.

In a number of recent works, which consider the preparation of ZnO nanoforms, various temperature ranges of precursors decomposition are indicated. Typically, low-temperature synthesis methods are more time consuming. In [20] described the synthesis ZnO nanoparticles with an average crystallite size of 16 nm by direct precipitation method at room temperature during 4 hrs followed by drying at 60°C without calcination. Romadhan and Suyatma [21] compared the structure and morphology of ZnO nanoparticles synthesized at three levels of temperatures (60°C, 80°C and 100°C) without or with calcinations (500°C). The results showed that preparation of ZnO nanoparticles by using heating at 80°C followed with calcinations at 500°C produced the smallest size (44 nm). As already stated [22, 23], calcination also influences the structure of the sample formed in terms of morphology. It was shown that the nanopowder agglomerated to form larger particle sizes at the calcination temperature higher than 500°C. In addition, the rate of subsequent crystallization, as well as the properties of the obtained ZnO nanoparticles, depend not only on the conditions of synthesis, but also on the nature of the salt from the solution of which precipitation is carried out. Barabaszová and Hundáková [24] showed that the nanoparticles morphology, crystallite size and particle size as well as pH of the reactions and subsequently ξ -potential values are mainly depending on the type of Zn-precursor (zinc chloride and zinc acetate) used. In the work of Bekele and Berhanu [25] ZnO nanoparticles were synthesized from zinc acetate dehydrate, zinc sulphate heptahydrate and zinc nitrate hexahydrate as precursors with sodium hydroxide and polyvinyl alcohol at the same temperatures of synthesis, drying and calcination at 90°C, 150°C and 400°C, respectively. The XRD results showed that the synthesized nanoparticles have wurtzite hexagonal structure with the average particle size of 44 nm, 42 nm, 30 nm from zinc nitrate hexahydrate and polyvinyl alcohol concentration, zinc acetate and sodium hydroxide, zinc sulphate heptahydrate and sodium hydroxide samples respectively.

In modern conditions, it is economically viable to develop technologies that can reduce the cost of chemical materials and energy resources. Thus, it is of interest to study a simple method for the synthesis of ZnO nanoparticles with good crystallinity at low temperatures.

The purpose of present work is the synthesis and characterization of ZnO nanoparticles that are suitable for textile finishing. A simple precipitation method was used to prepare the ZnO nanoparticles in aqueous solution at low temperature in a short time. The influence of the synthesis conditions, namely, the thermal decomposition temperature, on the structural parameters of nano-ZnO was studied using the X-ray diffraction analysis.

Materials and methods

Zinc acetate ($\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$) and sodium hydroxide (NaOH) were of analytical grade and used in this work as precursors without further purification.

ZnO nanoparticles were synthesized by direct precipitation method in aqueous solution. The molar ratio of Zn^{2+} to OH^- was 1:2. Alkali solution added drop wise into salt solution under vigorous stirring, which resulted in the formation of a white suspension. The reaction was allowed to proceed at 70°C for 2 hours to complete growth of nanoparticles. The resulting suspension was separated by centrifugation. The obtained white precipitate was washed three times in distilled water and two times in ethanol to remove any impurities or possible adsorbed ions. Subsequently, the washed precipitate was divided into three parts, labeled S1, S2 and S3 samples. Then S1 sample was dried at room temperature without heat treatment, S2 sample was dried in an oven in air atmosphere at 60°C without calcination, S3 sample was dried at 60°C followed by calcination at 300°C for 2 hours.

The crystal phase and structural parameters of the synthesized ZnO nanoparticles were determined by powder X-ray diffraction (Bruker D8 ADVANCE, Germany, CuK_α radiation). The peaks of the X-ray diffraction patterns were compared with the standard available data for the confirmation of the structure.

The processing of experimental results was carried out using Origin 2021b and Excel 2019 software.

Results and discussion

X-ray diffraction (XRD) is a simple and effective method for studying crystalline substances. The electrons of crystal atoms are responsible for the scattering of X-rays entering the crystal. The intensity of the diffraction scattering maxima is determined by the density of electrons in the atoms of those crystal planes from which scattering occurs. Analysis the profiles of diffraction peaks makes it possible to determine the distance between the crystal planes, the degree of their filling with atoms, the dimensions of the unit cell, and to get a complete picture of the crystal structure.

The powder XRD patterns of the ZnO samples S1, S2 and S3 synthesized at different decomposition temperatures are shown in Fig. 1.

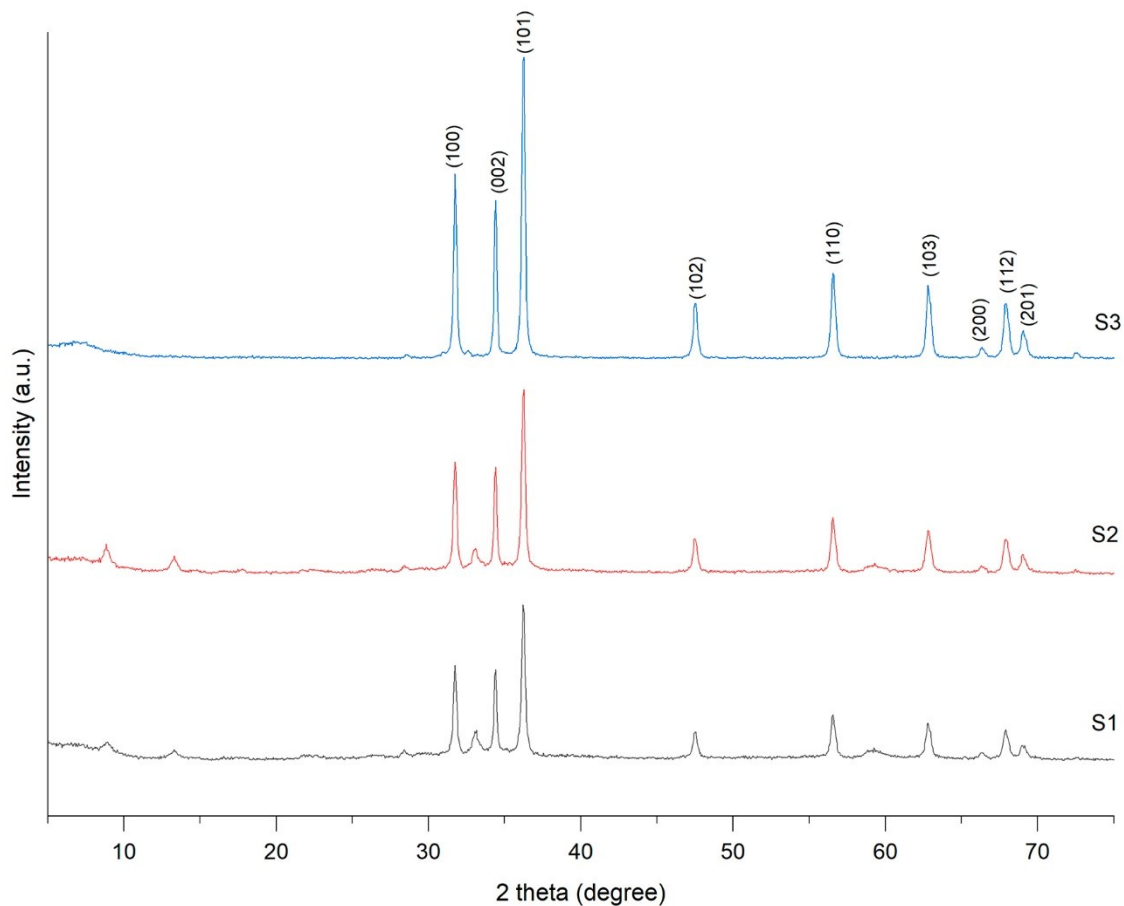


Fig. 1. XRD patterns of synthesized ZnO samples

The appearance of diffraction peaks (Fig. 1) corresponding to the reflection from (100), (002), (101), (102), (110), (103), (200), (112) and (201) crystal planes is consistent with the standard XRD peaks of bulk ZnO reported in JCPDS (Joint Committee on Powder Diffraction Standards) card number: 36-1451 and indicates the formation of ZnO with hexagonal wurtzite structure.

Shifts of the diffraction peaks positions are very small and quite consistent with that of JCPDS data (Table 1). It is obvious that the width and intensity of the diffraction peaks changed with increasing thermal decomposition temperature of precipitate. The peak broadening of the diffraction pattern is mainly due to four factors: microstrains (deformations of the lattice), faulting (extended defects), crystalline domain size and domain size distribution [20]. If we assume that the analyzed fine ZnO samples are free of strains and faulting, the peak broadening is only due to the crystalline domain size. Thus, definite peaks broadening in the XRD patterns indicates that small nanocrystals are present in the synthesized ZnO samples. The reduction in peaks width in XRD patterns with an increase in the decomposition temperature indicates an increase in the crystallites size. The observed sharper diffraction peaks of the S3 sample, due to the increase in intensity, indicate the enhancement ZnO particles crystallinity with increasing decomposition temperature. Further the synthesized S3 sample was free of impurities as no additional peaks associated with any other phase or impurity were observed apart from the characteristic peaks of ZnO.

The crystallite sizes of synthesized ZnO samples were estimated from XRD data using the Scherrer's formula (1) [26]:

$$D = \frac{K\lambda}{\beta \cos\theta}, \quad (1)$$

where D is the crystallite size, K is Scherrer's constant (0.89), λ is the X-ray wavelength (0.15406 nm), θ is the Bragg diffraction angle, and β is the full width at half maximum (FWHM) of the diffraction peak. For the calculation we fitted the diffraction peaks with Gauss function. The crystallite size is assumed to be the size of a coherently diffracting domain and it is not necessarily the same as particle size.

The dislocation density (δ), which represents the number of defects in the sample is defined as the length of dislocation lines per unit volume of the crystal and was calculated from the crystallite size (D) using the relation (2):

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

Micro strain is defined as the root mean square of variations in the lattice parameters across the sample. The micro strain itself depends on the non-uniform lattice distortions, faulting, dislocations, antiphase domain boundaries, and grain surface relaxation. The micro strain value (ϵ) for synthesized ZnO samples was calculated from XRD data using the formula (3):

$$\epsilon = \frac{\beta}{4 \tan \theta} \tag{3}$$

Interplanar spacing (d_{hkl}), which is the perpendicular distance between two successive crystallographic planes formed by the individual cells in a lattice structure, was calculated for different observed (hkl) planes from the Bragg's equation (4):

$$n\lambda = 2d_{hkl} \sin \theta, \tag{4}$$

where n is the diffraction order ($n = 1$).

All the calculated parameters for different crystallographic planes of each synthesized ZnO sample in comparison with standard data (JCPDS 36-1451) as well as the variation of the FWHM of various diffraction peaks are summarized in Table 1.

Table 1

Structural parameters of synthesized ZnO samples from XRD data for corresponding (hkl) planes

No.	(hkl) planes	2 θ (degree)		Interplanar spacing, d_{hkl} (Å)		FWHM (degree)	Crystallite size, D_{hkl} (nm)	Dislocation density, $\delta \times 10^{-3}$ (nm $^{-2}$)	Micro strain, $\epsilon \times 10^{-3}$
		Standard (JCPDS 36-1451)	Synthesized	Standard (JCPDS 36-1451)	Synthesized				
S1									
1	100	31.770	31.740	2.8143	2.8169	0.28998	28.165	1.261	4.451
2	002	34.422	34.389	2.6033	2.6057	0.24153	34.048	0.863	3.406
3	101	36.253	36.226	2.4759	2.4777	0.30124	27.439	1.328	4.018
4	102	47.539	47.506	1.9111	1.9124	0.33595	25.549	1.532	3.331
5	110	56.603	56.562	1.6247	1.6258	0.37665	23.685	1.783	3.055
6	103	62.864	62.821	1.4771	1.4780	0.39651	23.215	1.856	2.833
7	200	66.378	66.354	1.4071	1.4076	0.43248	21.703	2.123	2.886
8	112	67.961	67.914	1.3782	1.3790	0.46316	20.450	2.391	3.001
9	201	69.100	69.045	1.3582	1.3592	0.56812	16.784	3.550	3.604
S2									
1	100	31.770	31.742	2.8143	2.8168	0.27552	29.643	1.138	4.228
2	002	34.422	34.392	2.6033	2.6056	0.22833	36.016	0.771	3.219
3	101	36.253	36.230	2.4759	2.4774	0.29195	28.312	1.248	3.894
4	102	47.539	47.510	1.9111	1.9122	0.32735	26.220	1.455	3.245
5	110	56.603	56.565	1.6247	1.6257	0.36274	24.593	1.653	2.942
6	103	62.864	62.823	1.4771	1.4780	0.42453	21.683	2.127	3.033
7	200	66.378	66.354	1.4071	1.4076	0.44090	21.288	2.207	2.942
8	112	67.961	67.923	1.3782	1.3789	0.43231	21.910	2.083	2.801
9	201	69.100	69.056	1.3582	1.3590	0.46151	20.662	2.342	2.927
S3									
1	100	31.770	31.751	2.8143	2.8160	0.26181	31.196	1.028	4.017
2	002	34.422	34.399	2.6033	2.6050	0.22699	36.230	0.762	3.200
3	101	36.253	36.232	2.4759	2.4773	0.27377	30.192	1.097	3.651
4	102	47.539	47.524	1.9111	1.9117	0.31088	27.611	1.312	3.081
5	110	56.603	56.575	1.6247	1.6255	0.34447	25.899	1.491	2.793
6	103	62.864	62.843	1.4771	1.4776	0.37364	24.639	1.647	2.669
7	200	66.378	66.354	1.4071	1.4076	0.30998	30.280	1.091	2.069
8	112	67.961	67.932	1.3782	1.3787	0.39906	23.736	1.775	2.585
9	201	69.100	69.070	1.3582	1.3588	0.40643	23.464	1.816	2.577

It was established that the calculated sizes of crystallites of all synthesized ZnO samples are in the nanosize range (Table 1). Depending on the decomposition temperature of precipitate, the average crystallite size changes in the series $S1 < S2 < S3$ and amounts to 24.560, 25.592 and 28.139 nm, respectively.

An analysis of the obtained values of the interplanar distance (d_{hkl}) for various observed (hkl) planes (Table 1) shows that for all studied ZnO samples, the calculated and standard values of d_{hkl} differ insignificantly. The slight difference may be due to the presence of deformation in the crystals.

It was found that the dislocation density of synthesized ZnO samples is very low (Table 1). The change in the values of this index for different crystallographic planes is associated with the anisotropy of crystal growth. The

lowest values of the dislocation density, as well as micro strain of the crystal lattice, are observed for sample S3, which indicates the formation of a good quality crystalline material.

To evaluate the quality of the obtained nanocrystalline materials, it is also necessary to determine the crystal lattice parameters of the ZnO samples.

The wurtzite structure of ZnO (Fig. 2) can be considered to be composed of two interpenetrating hexagonal close packed (HCP) sublattices of Zn^{2+} cation and O^{2-} anion displaced relative to each other along direction c by length u .

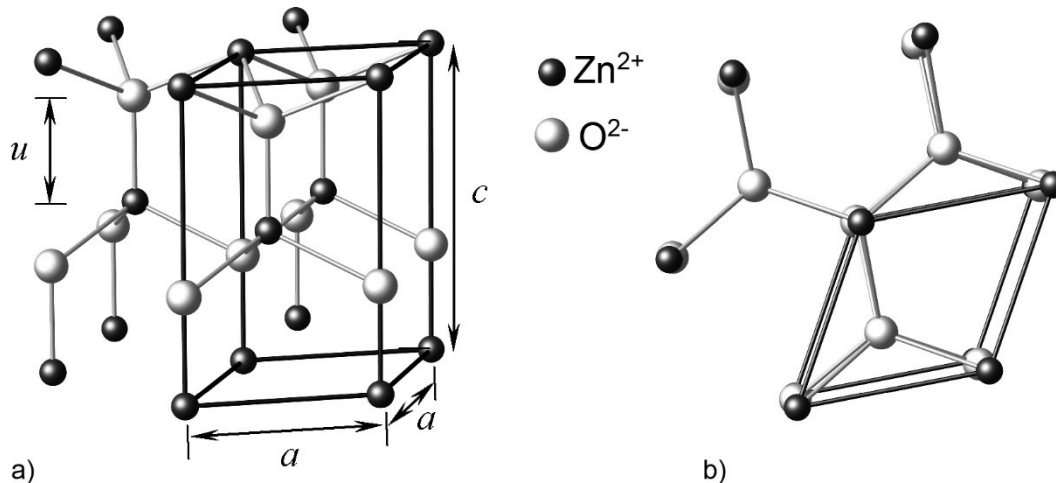


Fig. 2. Hexagonal structure of wurtzite ZnO:

a) – projection perpendicular to direction c ; b) – projection along direction c .

As seen in Fig. 2, in the crystal lattice of ZnO, layers of zinc ions alternate along the direction c with layers of oxygen ions. Each Zn^{2+} cation is surrounded by four O^{2-} anions located at the vertices of a tetrahedron (similarly, each anionic site is surrounded by a tetrahedral environment of four cationic sites).

The lattice parameters in the ZnO hexagonal structure were estimated using the equation (5), which relates the interplanar spacing (d_{hkl}) to the lattice constants a , c and the Miller indices h , k , l :

$$\frac{1}{d_{hkl}^2} = \frac{4}{3} \left(\frac{h^2 + k^2 + hk}{a^2} \right) + \frac{l^2}{c^2}. \quad (5)$$

The volume of the wurtzite unit cell (V) is expressed as (6):

$$V = \frac{\sqrt{3}}{2} (a^2 c). \quad (6)$$

The Zn–O bond length (L) is calculated by (7):

$$L = \sqrt{\frac{a^2}{3} + \left(\frac{1}{2} - u \right)^2 c^2}; u = \left[\frac{1}{3} \left(\frac{a}{c} \right)^2 + 0.25 \right], \quad (7)$$

where u is the positional parameter in the wurtzite structure and is a measure of the amount by which each atom is displaced with respect to the next along the c -axis.

The crystallinity of the synthesized ZnO samples was determined according to XRD data from the ratio of the crystalline regions area to the total area of amorphous and crystalline regions.

The calculated lattice parameters of synthesized ZnO samples in comparison with standard unit cell data (JCPDS 36-1451), average crystallite sizes and crystallinity are summarized in Table 2.

Table 2

General crystallite parameters of synthesized ZnO samples calculated from the XRD data

Sample	Average crystallite size, D (nm)	Lattice constant			Unit cell volume (\AA^3)	Bond length Zn–O (\AA)	Crystallinity (%)	
		a (\AA)	c (\AA)	c/a (\AA)				
Standard (JCPDS 36-1451)	–	3.250	5.207	1.6020	47.630	–	–	
Synthesized	S1	24.560	3.253	5.211	1.6022	47.749	1.9796	85.85
	S2	25.592	3.253	5.211	1.6022	47.743	1.9795	84.24
	S3	28.139	3.252	5.210	1.6023	47.703	1.9789	93.02

The calculated lattice parameters of the synthesized ZnO samples (Table 2) are in good agreement with the standard reported data (JCPDS 36-1451). The observed deviations in the values of the lattice constants can be caused by a slight change in the peaks position due to the defect. The lattice constants $a = 3.252 \text{ \AA}$, $c = 5.210 \text{ \AA}$ and the unit cell volume $V = 47.703 \text{ \AA}^3$ closest to the standard correspond to sample S3. The calculated Zn–O bond length in the unit cell of sample S3 is 1.9789 \AA . The crystallinity of the synthesized ZnO powder increases from 85.85 and 84.24% for samples S1 and S2 to 93.02% for sample S3 after calcination.

The results of the present study show that the formation of a pure ZnO phase in a wurtzite hexagonal structure with high quality of crystalline material and average crystallite size of 28 nm can be obtained from zinc acetate and sodium hydroxide as precursors at a synthesis temperature of 70°C , a drying temperature of 60°C , and a calcination temperature of 300°C .

Conclusions

The ZnO nanoparticles were synthesized successfully by direct precipitation method under different conditions. The ZnO phase formation was confirmed by XRD analysis. The XRD results show that the ZnO nanoparticles were formed in a wurtzite hexagonal structure with average crystallite size of 24.6–28.1 nm. The calculated structural parameters of the synthesized ZnO nanoparticles are in good agreement with the standard reported data (JCPDS 36-1451). The study of influence of the precipitate treatment temperature on the structure of crystalline material shows that calcination at 300°C (S3 sample) makes it possible to obtain ZnO nanoparticles free from impurities with a crystallinity of 93%. The results confirm the high quality of the synthesized ZnO nanoparticles, which make them suitable for possible application in textile finishing.

References

1. Look D.C. Recent Advances in ZnO Materials and Devices / D.C. Look // Materials Science and Engineering B-Solid State Materials for Advanced Technology. – 2001. – 80(1-3). – P. 383-387. – DOI: 10.1016/S0921-5107(00)00604-8.
2. Li L. Surface micro-dissolution process for embedding carbon nanotubes on cotton fabric as a conductive textile / L. Li, T. Fan, R. Hu, et al. // Cellulose. – 2017. – 24(2). – P. 1-8. – DOI: 10.1007/s10570-016-1160-2.
3. Fan T. Fabrication of magnetic cotton fabrics using surface micro-dissolving technology in ZnCl_2 aqueous solution / T. Fan, Z. Zhao, J. Zhou, et al. // Cellulose. – 2018. – 25(2). – DOI:10.1007/s10570-017-1623-0.
4. Thennarasu G. Enhanced visible photocatalytic activity of cotton ball like nano structured Cu doped ZnO for the degradation of organic pollutant / G. Thennarasu, A. Sivasamy // Ecotoxicology and Environmental Safety. – 2016. – 134(Pt 2). – P. 412-420. – DOI: 10.1016/j.ecoenv.2015.10.030.
5. Salat M. Durable antimicrobial cotton textiles coated sonochemically with ZnO nanoparticles embedded in an in-situ enzymatically generated bioadhesive / M. Salat, P. Petkova, J. Hoyo, et al. // Carbohydrate Polymers. – 2018. – 189. – P. 198-203. – DOI: 10.1016/j.carbpol.2018.02.033.
6. Abdelhady M.M. Preparation and Characterization of Chitosan/Zinc Oxide Nanoparticles for Imparting Antimicrobial and UV Protection to Cotton Fabric / M.M. Abdelhady // International Journal of Carbohydrate Chemistry. – 2012. – DOI: 10.1155/2012/840591.
7. Xu B. Fabrication of a Superhydrophobic ZnO Nanorod Array Film on Cotton Fabrics Via a Wet Chemical Route and Hydrophobic Modification / B. Xu, Z. Cai // Applied Surface Science. – 2008. – 254(18). – P. 5899-5904. – DOI: 10.1016/j.apsusc.2008.03.160.
8. Lam Y.L. Flame-retardant finishing in cotton fabrics using zinc oxide co-catalyst / Y.L. Lam, C.W. Kan, C.W.M. Yuen // Journal of Applied Polymer Science. – 2011. – 121, 1. – P. 612-621. – DOI: 10.1002/app.33738.
9. Abdel-Hady M.M. Flame retardancy and UV protection of cotton based fabrics using nano ZnO and polycarboxylic acids / M.M. Abdel-Hady, A. Farouk, S. Sharaf // Carbohydrate Polymers. – 2013. – 92, 1. – P. 400-406. – DOI: 10.1016/j.carbpol.2012.08.085.
10. Sapana J. Modification of Textiles via Nanomaterials and Their Applications / J. Sapana, V. Anurakshree, A. Rizwan // Frontiers of Textile Materials. – 2020. – P. 135-152. – DOI: 10.1002/9781119620396.ch6.
11. Anandan M. Tuning the crystalline size of template free hexagonal ZnO nanoparticles via precipitation synthesis towards enhanced photocatalytic performance / M. Anandan, S. Dinesh, N. Krishnakumar, K. Balamurugan // J. Mater. Sci. Mater. Electron. – 2017. – 28. – P. 2574–2585. – DOI: 10.1007/s10854-016-5833-2.
12. Zak A.K. Synthesis and characterization of a narrow size distribution of zinc oxide nanoparticles / A.K. Zak, R. Razali, W.A. Majid, M. Darroudi // Int. J. Nanomed. – 2011. – 6. – P. 1399–1403. – DOI: 10.2147/IJN.S19693.
13. Tabib A. Structural and optical properties of Na doped ZnO nanocrystals: Application to solar photocatalysis / A. Tabib, W. Bouslama, B. Sieber, et al. // Appl. Surf. Sci. – 2017. – 396. – P. 1528–1538. – DOI: 10.1016/j.apsusc.2016.11.204.
14. Liang Y. Organozinc Precursor-Derived Crystalline ZnO Nanoparticles: Synthesis, Characterization and Their Spectroscopic Properties / Y. Liang, S. Wicker, X. Wang, E. Erichsen, F. Fu // Nanomaterials. – 2018. – 8, 22. – DOI: 10.3390/nano8010022.
15. Ali T.T. Effect of pretreatment temperature on the photocatalytic activity of microwave irradiated

- porous nanocrystalline ZnO / T.T. Ali, K. Narasimharao, I.P. Parkin, et al. // *New J. Chem.* – 2015. – 39. – P. 321–332. – DOI: 10.1039/C4NJ01465K.
16. Fiedot M. The Relationship between the Mechanism of Zinc Oxide Crystallization and Its Antimicrobial Properties for the Surface Modification of Surgical Meshes / M. Fiedot, I. Maliszewska, O. Rac-Rumijowska, et al. // *Materials.* – 2017. – 10. – P. 353. – DOI: 10.3390/ma10040353.
17. Sathya M. Growth of pure and doped ZnO thin films for solar cell applications / M., Sathya A. Claude, P. Govindasamy, et al. // *Pelagia Res. Libr. Adv. Appl. Sci. Res.* – 2012. – 3(5). – P. 2591–2598.
18. Balogun S.W. Impact of Post-Deposition Heat Treatment on the Morphology and Optical Properties of Zinc Oxide (ZnO) Thin Film Prepared by Spin-Coating Technique / S.W. Balogun, Y.K. Sanusi, A.O. Aina // *J. Photon. Mater. Technol.* – 2017. – 3(3). – P. 20–28. – DOI: 10.11648/j.jmpt.20170303.11.
19. Sun Y. Synthesis and characterization of twinned flower-like ZnO structures grown by hydrothermal methods / Y. Sun, H. Guo, W. Zhang, et al. // *Ceram. Int.* – 2016. – 42. – P. 9648–9652. – DOI: 10.1016/j.ceramint.2016.03.051.
20. Talam S. Synthesis, Characterization, and Spectroscopic Properties of ZnO Nanoparticles / S. Talam, S.R. Karumuri, N. Gunnam // *ISRN Nanotechnology.* – 2012. – 6. – DOI:10.5402/2012/372505.
21. Romadhan M.F. Synthesis of ZnO Nanoparticles by Precipitation Method with Their Antibacterial Effect / M.F. Romadhan, N.E. Suyatma, F.M. Taqi // *Indones. J. Chem.* – 2016. – 16 (2). – P. 117-123. – DOI: 10.22146/ijc.21153.
22. Chen X. Preparation of ZnO photocatalyst for the efficient and rapid photocatalytic degradation of azo dyes / X. Chen, Z. Wu, D. Liu, Z. Gao // *Nanoscale Res. Lett.* – 2017. – 12. – P. 1-10. – DOI: 10.1186/s11671-0711904-4.
23. Seetawan U. Effect of Calcinations Temperature on Crystallography and Nanoparticles in ZnO Disk / U. Seetawan, S. Jugsujinda, T. Seetawan, et al. // *Materials Sciences and Applications.* – 2011. – 2. – P. 1302-1306. – DOI: 10.4236/msa.2011.29176.
24. Barabaszová K.Č Three methods for antibacterial ZnO nanoparticles preparation / K.Č Barabaszová, M. Hundáková, M. Mackovčáková, E. Pazdziora // *Materials Today: Proceedings.* – 2018. – 5. – P. S11-S19.
25. Bekele B. Synthesis, Characterization and Application of Zinc Oxide Nanoparticles for Textile Materials against Ultra Violet Radiation / B. Bekele, G. Berhanu, D. Shiferaw // *International Journal of Innovative Science and Research Technology.* – 2021. – 6, 1. – P. 1156-1165.
26. Cullity B.D. Elements of X-ray Diffraction / B.D. Cullity, S.R. Stock. – 3rd ed. New Jersey: Prentice Hall, 2001. – 664 p.

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