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Effect of Polyvinylpyrrolidone on the Structural and Morphological Properties of ZnO Nanoparticles

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In this study, zinc oxide (ZnO) nanoparticles were synthesized using polyvinylpyrrolidone (PVP) as a capping and stabilizing agent. X-ray diffraction (XRD), scanning electron microscopy (SEM), and energy-dispersive spectroscopy (EDS) were used to investigate the structural and morphological properties. The XRD results confirmed the formation of the typical hexagonal wurtzite structure in all samples, with average crystallite sizes of 23, 24, and 27 nm for the as-prepared ZnO, PVP-ZnO-cal, and ZnO-cal, respectively. These results indicate that the PVP-assisted sample effectively limits crystal growth. SEM images revealed that the as-prepared ZnO NPs are irregular and agglomerated, while the calcinated samples display more defined morphologies with average particle sizes of 37, 50, and 65 nm for the as-prepared ZnO, PVP-ZnO-cal, and ZnO-cal, respectively. Both XRD and SEM show the same trend where PVP-ZnO-cal has smaller sizes than ZnO-cal, confirming PVP's role in limiting growth and agglomeration. EDS confirmed the purity of Zn and O elements in all samples. These findings demonstrate the important role of PVP in controlling and modifying the growth of ZnO nanoparticles.

1. Introduction

Metal oxide semiconductors have been studied intensively in the last decades due to their properties and a wide range of possible technological applications [1-3]. Among them, zinc oxide (ZnO) is considered one of the most attractive candidates with interesting properties such as the direct wide bandgap 3.37 eV, large exciton binding energy 60 meV, good transparency in the visible region, and good chemical stability [4-6]. These make ZnO promising for gas sensors, the environment, optoelectronics, and energy conversion devices [7-11].

ZnO nanoparticles synthesized by various techniques and the chemical methods are widely used to prepare ZnO nanoparticles and thin films with different sizes, shapes, and morphologies, including the sol-gel, hydrothermal, co-precipitation, spray pyrolysis, and polyol methods [12-18]. The polyol method allows good control of the particle size, shaping through simple processing steps. However, the uncontrolled nucleation and particle agglomeration lead to non-uniform morphologies. Surfactants and polymeric stabilizers are often used to modify the growth kinetics to overcome these issues [19].

Polyvinylpyrrolidone (PVP) as a capping agent and stabilizer may serve and act as a protective agent in the synthesis of NPs to achieve a controlled and tailored size, shape, and morphological NPs. The carbonyl groups in the PVP can coordinate with the metal ions to control the nucleation rate and prevent excessive growth and agglomeration [20, 21].

In this study, ZnO nanoparticles were synthesized via the polyol method in the presence and the absence of PVP, as-prepared samples were calcinated at 500 °C for 1 hour to improve the crystallinity and remove the organic compounds. The structural and morphological properties were investigated using X-ray diffraction (XRD), scanning electron microscopy (SEM), and energy-dispersive spectroscopy (EDS) to provide insight into the influence of PVP as a capping and stabilizing agent on the particle size, morphological, and compositional purity of the ZnO crystal growth.

2. Results and Discussion

The XRD diffraction patterns of the as-prepared ZnO, and calcinated ZnO synthesis with and without

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PVP is shown in Figure 1. As can be seen from the figure, all the diffractions peaks confirm the formation of ZnO hexagonal wurtzite crystal structure in accordance with JCPDS Card No. 36-1451, without the presence of any impurities, The observed to 2θ values where at 31.7°, 34.4°, 36.2°, 47.5°, 56.6°, 62.8°, 66.4°, 67.8°, 69.1°, 72.5° and 76.9° corresponding to the lattice planes of (100), (002), (101), (102), (110), (103), (200), (112), (201), (004) and (202), respectively. Both ZnO-cal and PVP-ZnO-cal exhibit sharper and more intense diffraction peaks compared to the as-prepared ZnO NPs, as can be seen in Figure 1(b), confirming the improvement in crystallinity after the calcination. Furthermore, a slight shift to higher 2 theta values can be seen for the case of the calcinated samples, attributed to the removal of residual organic species during the calcination process. The grain size was calculated from the three most intense peaks for ZnO using the Debye-Scherrer equation [22]:

$$D_c = {}^{\kappa\lambda}/_{\beta cos\theta} \qquad (1)$$

where λ is the x-ray wavelength (CuKa=1.5406 Å), κ is the crystallite shape factor (0.94), β is the peak full width at half maximum (in radians) at the observed peak angle θ. The structural parameters of the obtained NPs were summarized in Table 1.

The average crystallite size values from the three main peaks are 23.18, 27.15, and 24.51 nm for as-prepared ZnO, ZnO–cal, and PVP–ZnO–cal, respectively. The smaller crystallite size for the as-prepared ZnO is understandable and confirming less crystallinity compared to the calcinated samples, for the D of PVP–ZnO–cal is less than the ZnO–cal, indicating that PVP effectively limits the ZnO growth, which is also supported by the increase in the FWHM. This is confirming that PVP act as capping and stabilizing agent that suppress the grain growth without altering the lattice spacing (d) [23]. It is worth mentioning the slight increase in the intensity of certain peaks observed in the (002) and (102) planes compared to ZnO–cal of PVP–ZnO–cal compared to ZnO–cal, and this suggests that PVP may affect the preferred growth orientation of ZnO [24].

Figure 2 shows the SEM images of the as-prepared ZnO, ZnO-cal, and PVP-ZnO-cal samples. The as-prepared ZnO exhibits irregular and highly agglomerated nanoparticles with an average particle size of about 37 nm. In the case of PVP-ZnO-cal, it is clearly evident that the nanoparticles become more defined after calcination, which is consistent with the XRD results, with an average particle size of approximately 50 nm. Meanwhile, the ZnO-cal sample, prepared without PVP, also exhibits well-defined spherical shapes and uniform distribution, with an average particle size of about 65 nm. The average particle sizes were determined using ImageJ software from the SEM images. The EDS spectra confirm the presence of only Zn and O elements, with no other impurities in any of the samples. For the as-prepared sample, the presence of C is understandable due to organic residue, which is removed after calcination, as seen in the calcined samples. The quantitative EDS results (Atomic %) are: As-prepared ZnO: C 51%, O 35%, Zn 12%; PVP-ZnO-cal: O 47%, Zn 51%; ZnO-cal: O 44%, Zn 54%. Both calcinated samples have low carbon content; however, the PVP-ZnO-cal exhibits a Zn:O atomic ratio closer to the ideal stoichiometric value of 1:1, indicating better composition control, more refined nanoparticles, and reduced agglomeration.

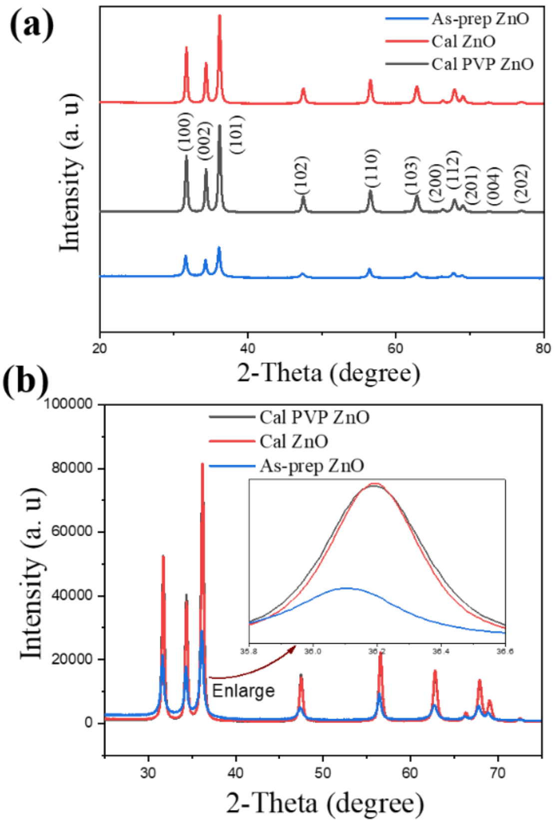


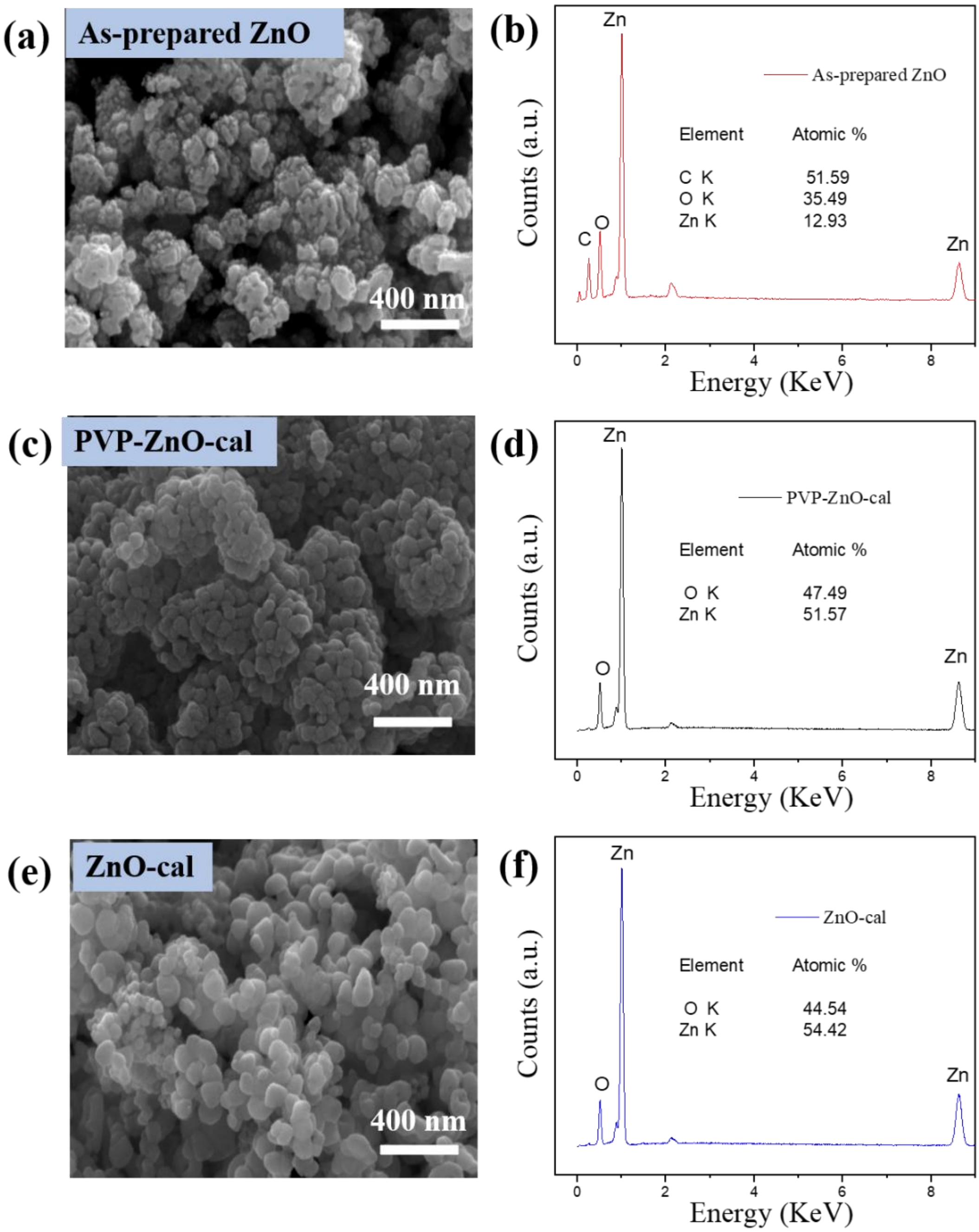
Figure 1. The XRD diffraction patterns of the as-prepared ZnO, and calcinated ZnO synthesis with and without PVP.

3. Conclusion

Zinc oxide (ZnO) nanoparticles were synthesized using polyvinylpyrrolidone (PVP) as a capping and stabilizing agent. The results show that the PVP effectively restricts crystal growth and prevents excessive agglomeration. XRD and SEM confirmed the role of the PVP as a capping agent without altering the ZnO crystal structure. EDS further supports this and reveals a high purity of

Table 1. The structural parameters of the obtained NPs.

Sample	2θ (°)	(hkl)	d-spacing (Å)	FWHM (°)	D (nm)
As-prepared ZnO	31.5852	(100)	2.83036	0.3667	22.51
	34.2648	(002)	2.6149	0.3267	25.45
	36.069	(101)	2.48813	0.3869	21.59
ZnO–cal	31.6742	(100)	2.82261	0.2878	28.69
	34.3269	(002)	2.61031	0.31	26.82
	36.1588	(101)	2.48216	0.3222	25.94
PVP–ZnO–cal	31.6723	(100)	2.82277	0.3392	24.34
	34.3274	(002)	2.61028	0.317	26.23
	36.1584	(101)	2.48218	0.364	22.96



Element	Atomic %
C K	51.59
O K	35.49
Zn K	12.93

Element	Atomic %
O K	47.49
Zn K	51.57

Element	Atomic %
O K	44.54
Zn K	54.42

Figure 2. SEM images and EDS spectra of the as-prepared ZnO, ZnO-cal, and PVP-ZnO-cal sample.

the synthesized samples. These results demonstrate that the PVP-assisted synthesis route provides a simple and cost-effective approach to control the size and morphology of ZnO nanoparticles.

Method

Zinc acetate dihydrate (Zn(OAc)2. 2H2O) was used as the precursor, ethylene glycol EG was used as the solvent and reducing agent. On the other hand, Polyvinylpyrrolidone (PVP, M 40000) served as a capping and stabilizing agent to prevent oxidation and agglomeration. However, deionized water was employed to control the hydration ratio and for the washing process. All the reagents were obtained from Sigma-Aldrich and used as received without further purification.

ZnO synthesis with and without PVP

To prepare 0.1 M ZnO, zinc acetate dihydrate ($Zn(CH3COO)_2\cdot 2H_2O$) was dissolved in 20 ml of ethylene glycol. Then, 0.2 M polyvinylpyrrolidone (PVP) was added to act as a capping and stabilizing agent to prevent oxidation and agglomeration of nanoparticles (this applies for the PVP-ZnO). A 2:1 molar ratio of PVP to zinc precursor (0.2 M PVP to 0.1 M Zn) was used to ensure sufficient polymer coverage for nanoparticles. Next, a fixed ratio of H_2O/Zn of 6, using deionized water, was added to control hydration. Under Ar gas, the solution was magnetically stirred and heated to 185 °C for 30 minutes, then cooled to room temperature. The resulting product was washed with deionized water and centrifuged at 9000 rpm for 6 minutes per cycle. The as-prepared ZnO powder was dried at 80 °C and then directly tested for nanoparticle properties. For the PVP-prepared ZnO, the powder was calcined at 500 °C for 1 hour to remove organic residues and obtain well-defined crystalline ZnO nanoparticles. For comparison, ZnO nanoparticles were synthesized using the same procedure but without PVP, and the final product was similarly washed, centrifuged, and calcined at 500 °C for 1 hour.

Characterization

The prepared NPs were characterized by X-ray diffraction (XRD) using a PANalytical EMPYREAN diffractometer with Cu Kα radiation (λ= 1.54059 Å). The particle size and morphology of the samples were examined using a FEI Quanta 650 scanning electron microscope (SEM), and energy-dispersive X-ray spectroscopy (EDS) was used to determine the chemical composition of the NPs; all data were collected at an accelerating voltage of 20 kV.

Authors' Contributions

AEN: Experiment, Methodology, Writing-Drafting and Editing Article.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Declaration of Ethical Standards

The author(s) of this article declare that the materials and methods used in this study do not require ethical committee permission and/or legal-special permission.

Conflict of Interest

There is no conflict of interest in this study.

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