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Synthesis, Characterization and Optical Morphology of ZnO Nanoparticles

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Abstract

The ZnO molecule plays an important role in the industry due to it special features, anti-corrosion anti-bacterial properties, as well as due to its low electrical conductivity and heat resistance. In these experimental researches, the sol-gel method was chosen, which enables control of nucleation, aging and growth of particles in the solution. ZnO synthesis was prepared utilizing chemical method with Zinc acetate dyhidrate and NaOH with the appropriate methanol solvent and heating (60˚C). The methods used in identification and characterization are *FTIR*, *UV*/ *VIS*, *OPTICAL MICROSCOPY*, *SEM* and *XRD*. The FTIR spectra of synthesized ZnO with corresponding ones show characteristic bands at the corresponding wavelengths, which confirm the presence of ZnO nanoparticles. SEM characterization of ZnO shows the morphology of needle-shaped nanoparticles. XRD spectar in this research by chemical method indicates the particle size of 17.76 nm.

Keywords

Synthesis, ZnO, Nanoparticles, Sol-Gel Method, FTIR, SEM, XRD

1. Introduction

Zink oxide (ZnO) is the compound of unique physical and chemical features. It appears as white or light yellow powder, almost insoluble in water and alcohol but soluble in diluted mineral acids such as HCl. This chemical compound is important in the ceramic industry due to its hardness, strength and piezoelectric constant [1] [2] [3]. ZnO shows important antibacterial properties [4] which inhibit the growth of a wide range of bacteria [4], quite cheap, of low toxicity and high efficiency from UV rays, excellent for the production of suntan creams. As zinc oxide can behave as a basic and acidic oxide [5], it crystallizes in a hexagonal crystal system at room temperature and pressure. In the research that was done, the main conclusion is that only the formulation containing zinc oxide provides complete protection of the wood samples after washing [6]. Zinc-based compounds have been shown to be effective in various studies and effective in terms of termite mortality and rot resistance against fungus. Bak and Nemeth [7] [8] reported the efficacy of different nanoparticles against fungi. Nanoparticles are small particles ranging in size from 1 to 100 nm that may contain carbon, metal, metal oxides or organic substances [9]. They show enormous morphological diversity such as spheres, cylinders, disks, plates, hollow spheres, tubes, etc. The synthesis of metal nanoparticles is an important area of research that has attracted enormous attention from scientists. ZnO nanoparticles can be synthesized by various methods, such as hydrothermal, sol-gel, precipitation, sonochemical and microemulsion methods [10]. The method used in this study for the synthesis of ZnO nanoparticles is the sol-gel method. The sol-gel method has raised a great interest among researchers since it offers controlled consolidation, shape modulation, nanostructure shaping [11] and low processing temperature [12]. In this experimental study, the influence of methanol solvent on the synthesis of ZnO nanoparticles from $Zn(CH_3COO)_2 \cdot 2H_2O$ when heated to 60˚C with water as a medium was shown. The synthesized ZnO was compared with commercial ZnO and characterized by appropriate methods. ZnO shows (hexagonal symmetry) a wurzite structure or (cubic symmetry) rock salt structure, but ZnO crystals are more commonly stable with hexagonal symmetry (**Figure 1**) [13].

2. Material and Methods

2.1. Material

In this experimental research, the following chemicals were used:

- Zink acetate dehydrate ($Zn(CH_3COO)_2 \cdot 2\ H_2O$, Sigma Aldrich
- Methanol (CH_3OH) Sigma Aldrich
- Sodium - hydroxide (NaOH), Sigma Aldrich
- Distilled water.

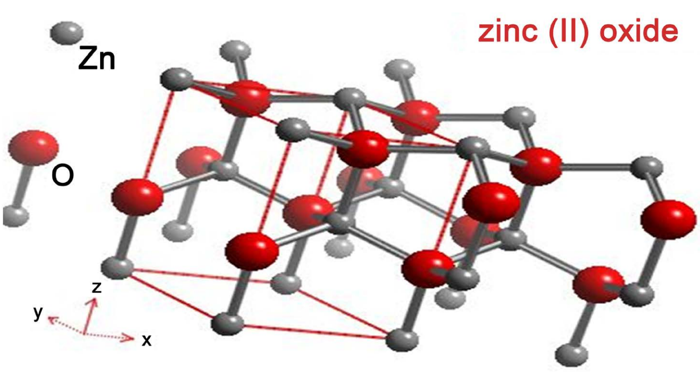


Figure 1. Hexagonal structure of ZnO This figure is reproduced from reference [11].

2.2. Methods

Analysis of ZnO molecule is done utilizing the following methods:

- Interpretation of spectrum of synthetized ZnO samples is analyzed at Perkin Elmer BX FT-IR spectrophotometer at 2 cm^{-1} resolution and wave length range from 4000 to 560 cm^{-1}.
- Analysis of UV spectrum of ZnO is done at Perkin Elmer Lambda UV/VIS 25 spectrophotometer at wave length range from 200 to 400 nm.
- Samples are analyzed at microscope Leica, model 2500D, working on the principle of transmitted polarized light. While making micro photographs, Nikolo's prisms were placed vertically (XPL). Samples are diluted in water and DMSO.
- Scanning electronic microscope (SEM), model JEOL JSM IT 200LA, maximum resolution 3 nm at voltage of 30 KV, power resolution of EDS detector: 129 eV.
- Analysis of synthesized ZnO is done at X-ray diffractometer XRD Bruker D8 Advance, 40 kV, 40 mA.

2.3. Experimental Procedure

ZnO nanoparticles were synthesized by the sol-gel method utilizing 2 g of $(Zn(CH_3COO)_2 \cdot 2H_2O$ dissolved in 15 ml of distilled water with stirring on a magnetic stirrer for 5 minutes, while at the same time a mixture of 8 g of NaOH was prepared in 10 ml of distilled water with the same time stirring for 5 minutes while heating to 60˚C with 100 ml of methanol which was added drop by drop until the formation of a white precipitate. The mixture was left to rest for 24 hours at room temperature, after which it was filtered. White to light yellow precipitate was dried in an oven at about 80˚C. ZnO synthesis lasted for about 2 hours.

3. Results and Discussion

3.1. FTIR Analysis of ZnO

The analysis of the FTIR spectrum of the synthesized ZnO samples is shown in **Figure 2** in comparison with commercial ZnO. Spectrum of the synthesized ZnO in **Figure 2** shows three absorption bands of 3510.04 cm^{-1}, 3387.37 cm^{-1} and 3228.45 cm^{-1}, which is shown for the stretching vibration of the OH group. The peak at 1434.25 cm^{-1} corresponds to the bending of the C-H stretching vibration. The peak present at 686.86 cm^{-1} with methanol solvent corresponds to the Zn-O bond compared to the peak present for commercial ZnO, which is at 689.27 cm^{-1}, where there is a slight shift of the band compared to commercial ZnO.

3.2. UV/Vis Analysis of ZnO

The optical properties of synthesized ZnO nanoparticles were measured in the range of 200 - 400 nm and are shown in **Figure 3**. The UV spectrum of ZnO

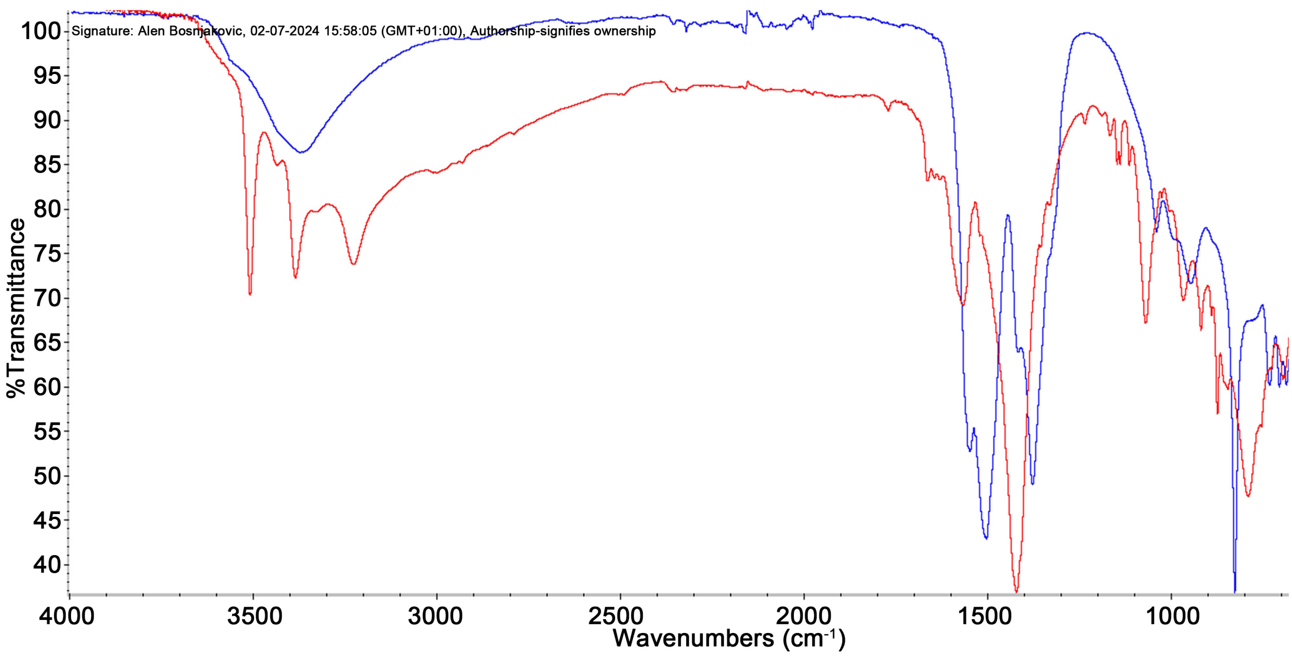


Figure 2. FTIR spectrum (a) commercial ZnO and (b) synthetized ZnO.

nanoparticles in methanol solution shows an absorption maximum at 255 nm and another at 325 nm [14], where we observe a shift of the band towards higher wavelengths (red shift) compared to the spectrum of commercial ZnO whose absorption maximum is at 235.38 nm [15] and which may be due to the influence of particle size and their structure. The results are similar to those of other authors with small differences.

3.3. ZnO Analysis with Optical Microscope

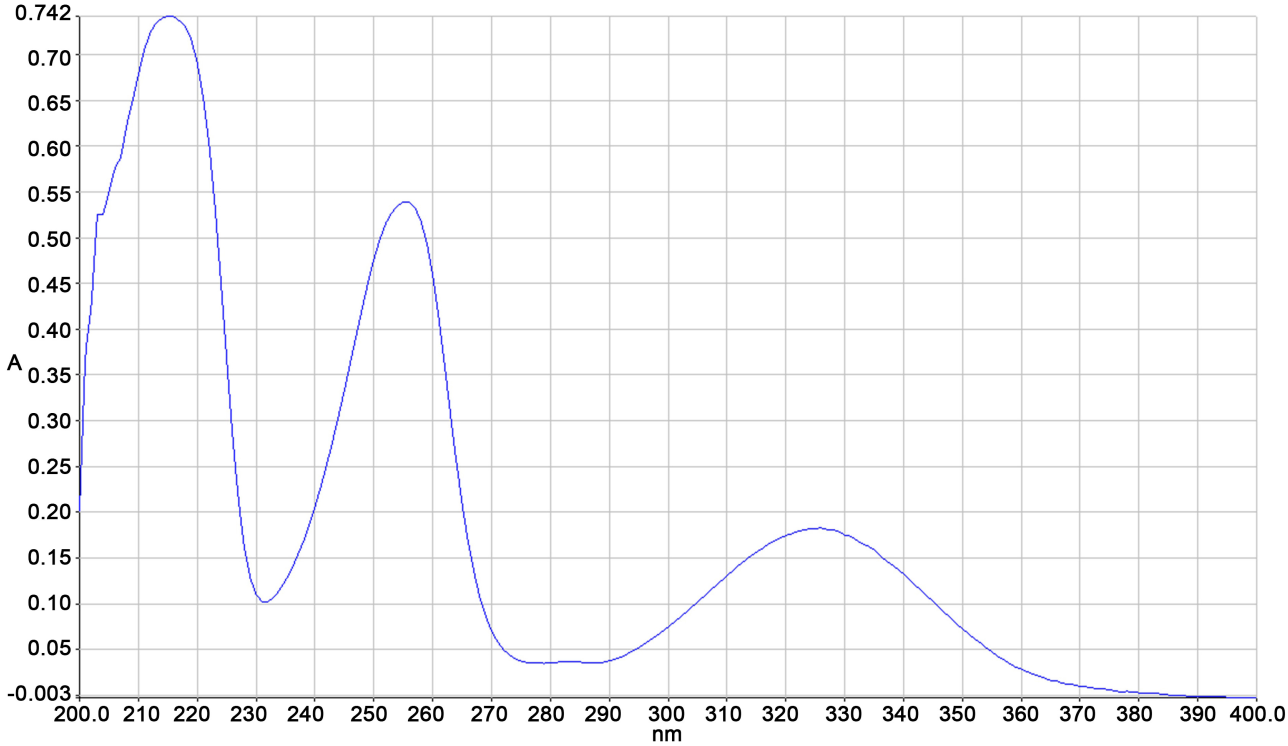
Figure 4 shows needle crystals (A) with a methanol solvent of transparent coloured ZnO with a diameter ranging from 0.02 to 0.2 mm. The second form of crystal (B) is on the edges of the preparation, where the crystals are of an irregular shape, yellow in colour, with a ZnO crystal diameter of 0.15 - 0.25 mm.

3.4. SEM Analysis of ZnO

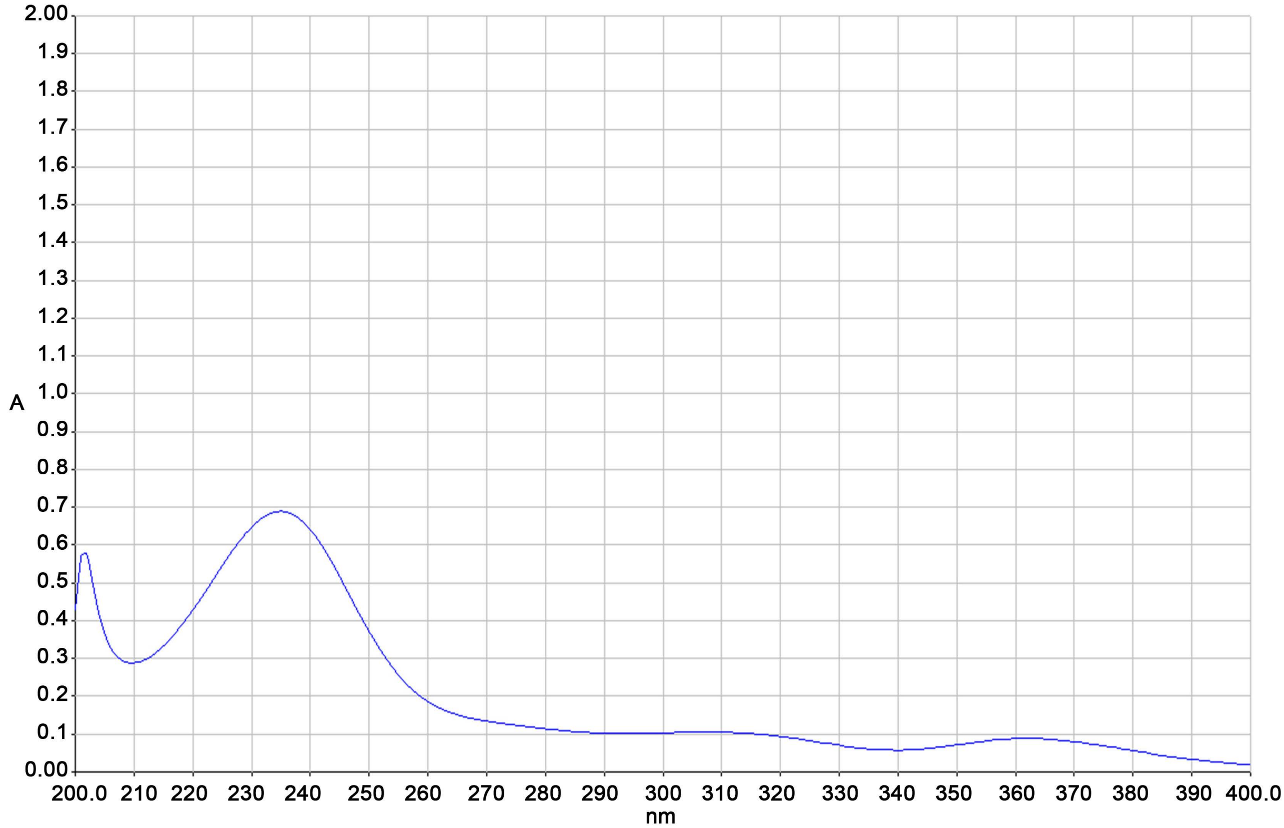
The analysis of the samples utilizing SEM method shows the morphology of the ZnO crystals in **Figure 5**. **Figure 5** shows the recorded nanoparticles of the synthesized ZnO, which have a needle-like structure. Nanoparticles of commercial ZnO show needle-like structure crystals in the form of a ball as it is shown in the picture. The morphology of ZnO nanoparticles was confirmed by the performed analysis.

4. XRD Analysis of ZnO

The diffraction lines are consistent with the values listed in the ZnO database, providing clear evidence of the formation of the hexagonal Wurtzite-type structure of the synthesized ZnO. No diffraction peaks from any other impurities



(a)



(b)

Figure 3. UV/Vis spectrum (a) commercial ZnO and (b) synthesized ZnO.

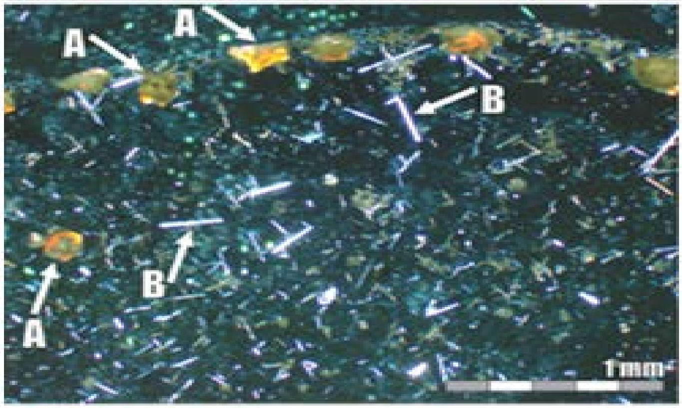
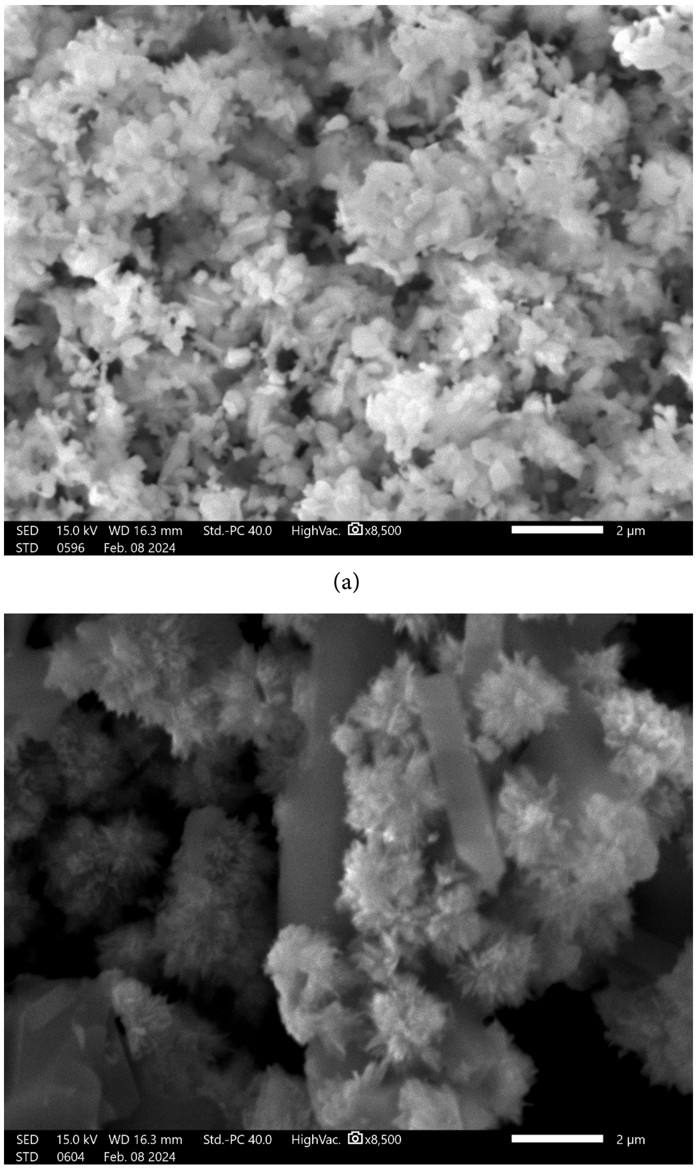


Figure 4. Synthesized ZnO.



(a)

(b)

Figure 5. SEM photos of (a) commercial ZnO and (b) synthesized ZnO.

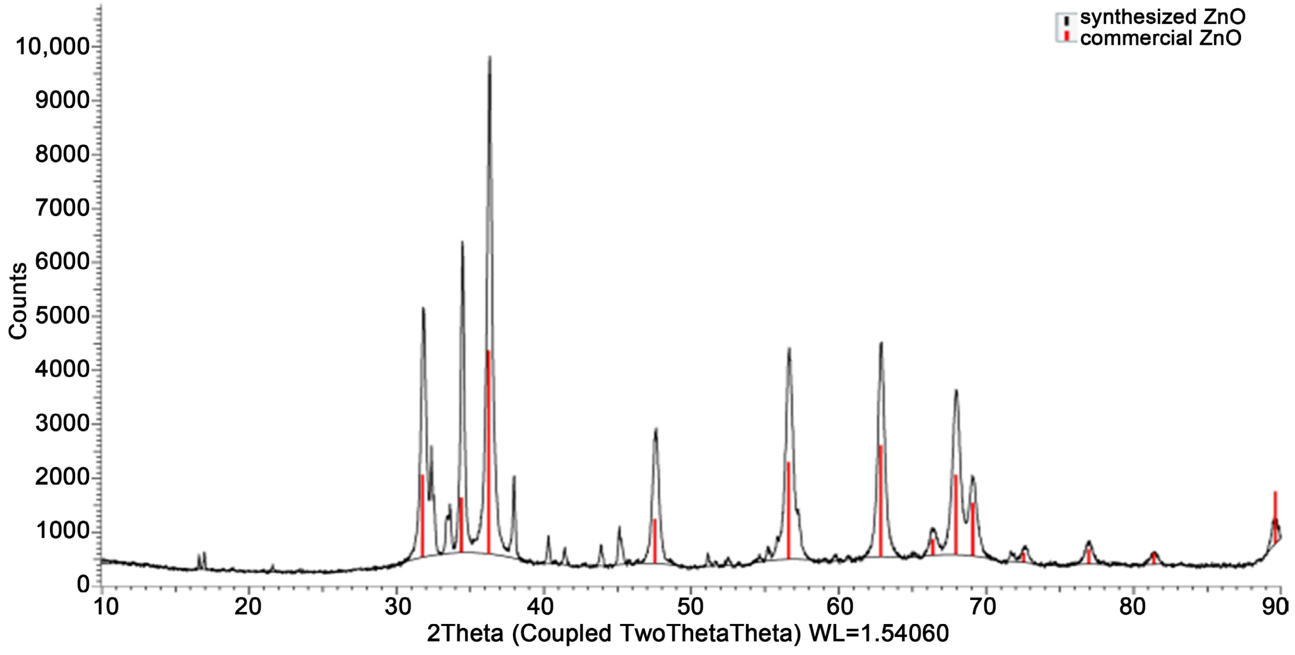


Figure 6. XRD spectar commercial ZnO and synthesized ZnO.

were detected. All the diffraction peaks are quite sharp, indicating that the ZnO sample has a high degree of crystallinity. Using the Debye-Scherrer equation, we have obtained information on the diameter of the needle-like structures, which is 17.76 nm (**Figure 6**).

5. Conclusions

ZnO nanoparticles were synthesized utilizing sol-gel method in this experimental research.

In this study, the influence of solvents on the morphological and optical properties of ZnO nanoparticles was investigated. The properties of zinc oxide nanoparticles were confirmed by SEM, FTIR, XRD, UV characterization methods and optical microscopy. ZnO nanoparticles can be used in various fields of medicine to treat different diseases such as cancer. In this research, the solvent methanol was used, which opens up the possibility of further research in terms of the influence of the solvent on the very structure of ZnO nanoparticles.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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