



**SYNTHESIS AND COMPARATIVE STUDY OF NANO ZINC OXIDE
STRUCTURES WITH AND WITHOUT CETYLTRIMETHYLAMMONIUM
BROMIDE USING SOL-GEL METHOD**

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Abstract

In this research article, we have done comparative study of synthesis of ZnO nanoparticles without using cetyltrimethylammonium bromide (CTAB) and using cetyltrimethylammonium bromide (CTAB) by sol–gel method in aqueous medium as an environmentally benign method in structure directing agents. Sol-gel method is the very simple method and has the capability to regulate the particle size and morphology through systematic monitoring of reaction parameters. The surfactant effect on the systematic arrangement of the ZnO crystals was studied by scanning electron microscopy (SEM) and transmission electron microscopy (TEM) techniques. To study the probable changes in other properties of ZnO, characterizations like X-ray diffraction (XRD), Fourier transfer infrared spectroscopy (FT-IR) and UV-visible spectroscopy analysis were studied and discussed.

Keyword: ZnO nano particles, Sol–gel method, CTAB, aqueous medium.

Introduction

Zinc oxide (ZnO) is a multipurpose material. Nano sized ZnO crystals have attracted a great deal of attention because of their size-dependent optoelectronic properties. A chemical synthesis of nano-sized ZnO crystals in aqueous solution, which was suitable for a large-scale production, has been developed¹.

The synthesis of various structures of ZnO have increased rapidly, viz., rods, wires, tubes, towers, stars, dendrite and flower like, etc. These structures are likely to have more potential applications in building functional electronic devices with special architectures and distinctive optoelectronic properties. Therefore, it is tried to synthesize ZnO nano or

microstructures in a controllable shape and size to get the demand and to explore the potentials of ZnO. It is still a challenging task for material scientists, to directly fabricate large scale ZnO crystals with controlled morphologyⁱⁱ. Many methods are applied for the synthesis of zinc nano structures like hydrothermal/solvothermal process^{iii-vii}, Micro-emulsion^{viii}, etc. by researchers. J. B. Zhong *et al.* done study of improved photo-catalytic performance of ZnO by sol-gel method^{ix}. Recently M. Meddouri *et al.* worked on Effect of co-solvent on structural and morphological properties of ZnO aerogel prepared by a modified sol-gel process^x.

Since a decade, most of the researchers are inclined to prepare ZnO crystals at a low temperature to reduce energy consumption, improve large-scale production, and obtain special properties^{xi}. The market demand for the ZnO nanoparticles is increasing and extensively used in industries due to their ultraviolet filtering, catalytic, anti-corrosion and anti-bacterial properties. Recently, they have mainly been used in sunscreens as an ultraviolet-resistant additive. Other applications of zinc oxide nano-powder include electrophotography, photo-printing, capacitors, protective coatings, anti-microbial, and conductive thin-films in LCDs, solar cells, and blue laser diodes^{xii}.

Therefore, in this research article we focused on the comparative study of zinc nano structure with CTAB and without CTAB as a surfactant. The investigation of effects of CTAB as surfactants on the surface morphology and other properties of ZnO nano material was done. The new technology of sol-gel method was adopted in the preparation of nanomaterial. The structural changes of material are preferably measured by using X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), infrared spectroscopy (IR) and energy dispersive spectroscopy (EDS) have been characterized and the structure and morphology of ZnO nanomaterial was studied. The synthesis of zinc nano structures have been performed by using distilled water as a core solvent.

Experimental

All the chemicals were analytical grade and purchased from SD Fine chemicals, Mumbai. These chemicals were used without further purification. All the aqueous solutions were prepared using distilled water.

1. Synthesis of nanomaterial

1.1 Zn-O nano-particle synthesis (Synthesis of nano structure without CTAB)

It was synthesized by using new sol-gel method. 0.05 M of zinc acetate was dissolved in 50 ml distilled water and 0.05 M acetic acid dissolved in 50 ml water. The given both solutions were mixed each other drop wise with constant stirring. The pH of mixture solution adjusted up to 7.6 by adding NH₄OH using pH-meter. After the addition of NH₄OH with constant stirring the several minute white precipitate formed. It was separated by centrifugal method and washing with distilled water for 2-3 times. The product was dried at 500 °C for 3 hr.

1.2 Zn-O nano particle synthesis (Synthesis of nano structure with CTAB)

0.05 M of zinc acetate was dissolved in 50 ml of water and 0.05 M of acetic acid was diluted in 50 ml distilled water. 0.1 gm of CTAB was dissolve in 20 ml of water. Zn-acetate solution was added in acetic acid solution drop wise with constant stirring. After the complete mixing of solution drop wise CTAB solution was added as a surfactant. The pH of solution adjusted to 7.6 using NH₄OH. After the addition of NH₄OH, the solution mixture was stirred with constant stirring for the several minutes. The white precipitate was separated by centrifugal method and washed with distilled water for 2-3 times. The product was dried at 500 °C for 3 hours.

2. Result and Discussion

2.1 XRD Analysis

The XRD patterns of the synthesized products without CTAB and with CTAB by sol-gel methods are shown in **Fig-1** and **Fig-2** comparatively. All diffraction peaks can be indexed as hexagonal wurtzite structure (JCPDS card No. 80-0075). The XRD pattern of ZnO nanoparticles indicates the prominent diffraction peaks at 2θ values of 31.90° , 34.50° , 36.25° , 48.90° , 56.95° , 66.80° and 68.10° which are shown to the typical Hexagonal type of ZnO. Samples were synthesized with the addition of CTAB show higher intensity compared to the sample without CTAB (**Fig. 1 and 2**). The sharp diffraction peaks indicated the good crystallinity of the prepared crystals^{xiii-xvi}.

During the study of nano structure of ZnO without CTAB are unequal particle size, but the size of ZnO with CTAB is greater and regular arranged. Due to the increased particle size, the specific surface area reduces, which is in good coordination with the results of the specific surface area. CTAB can diminishes the surface tension of the solution which gives the ZnO particles of good distribution. By adding excess amount of CTAB into the solution system the thickness of colloids becomes more, thus the colloidal particles which formed a nucleus i.e. small particles easier to combine again and then form large particles. The development of ZnO nano particles with uniform particle size increases with the CTAB concentration limits.

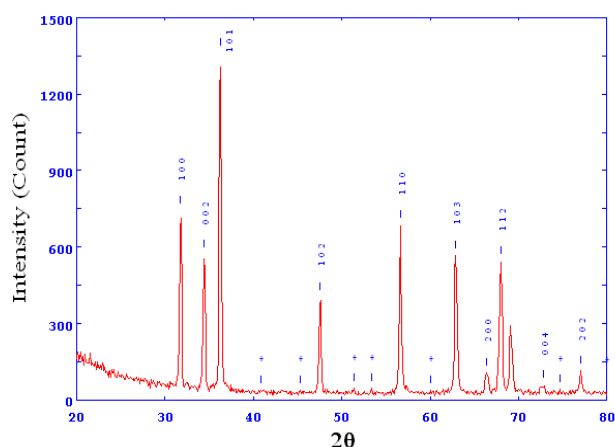


Fig-1. XRD of ZnO without CTAB as a surfactant nano structure

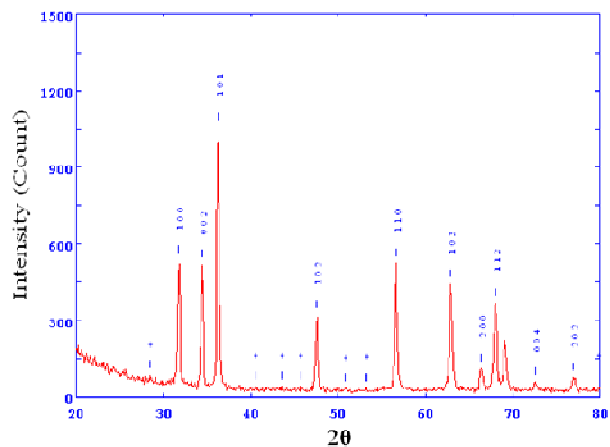


Fig-2. XRD of ZnO with CTAB as a surfactant nano structure

2.2 Morphology

To study the effects of cationic/anionic surfactants on morphology of ZnO nanomaterial, we have done comparison in samples using scanning electron microscopy (SEM). This shows perfect difference between compared samples. Before addition of CTAB some irregular shape of nano particle has been observed, shown in **Fig-3**. After the addition of CTAB remarkably affected the morphology of particles, shows uniform arrangement of ZnO-CTAB particle has been detected in **Fig-4**.

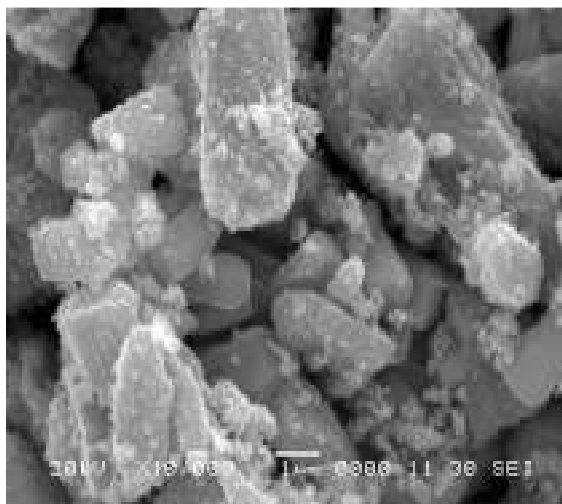


Fig-3. SEM image of ZnO without CTAB as a surfactant nano structure

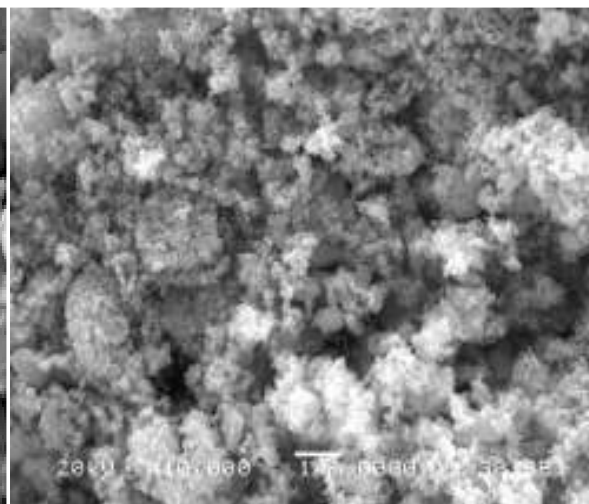


Fig-4. SEM image of ZnO with CTAB as a surfactant nano structure

To get more information about ZnO nanoparticles, TEM were taken on inorganic residues remaining after burning the sample at 500⁰C. **Fig- 5** shows irregular shape and size of nano structures in the investigated TEM of zinc oxide nano particles without cetyltrimethylammonium bromide (CTAB). Whereas the uniform crystal size and shape of the nano structures were shown by using transmission electron microscopy when studied ZnO with CTAB as shown in **Fig-6**.

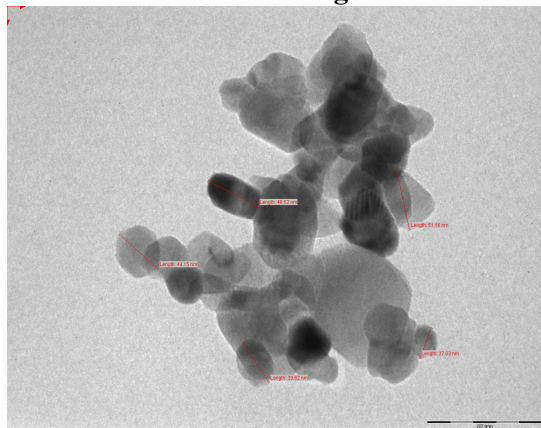


Fig-5. TEM of ZnO without CTAB as a surfactant nano structure

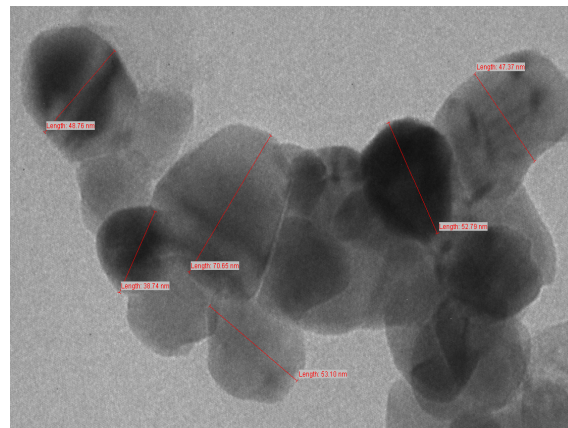


Fig-6. TEM of ZnO with CTAB as a surfactant nano structure

2.3 Fourier Transfer Infrared Spectroscopy (FT-IR)

The FTIR spectrum of synthetic of ZnO nano particles with CTAB shows absorption bands due to O–H stretching at 3,530 cm⁻¹, asymmetric and symmetric C=O stretching of Zinc acetate at 1,630 and 1,510 cm⁻¹, O–H bending of hydroxyl group at 546.66 cm⁻¹, and Zn–O stretching of ZnO at 507.70 cm⁻¹. These results as approximately match with another study^{xvii}.

2.4 The Energy Dispersive Spectroscopy (EDS)

The EDS was performed with the apparatus JOEL (Jed-2300). The formation of the ZnO nano structures was due to the presence of a CTAB as surfactant. The energy dispersive spectroscopy (EDS) shown in **Fig-6** showed the presence of Zn^{xviii}.

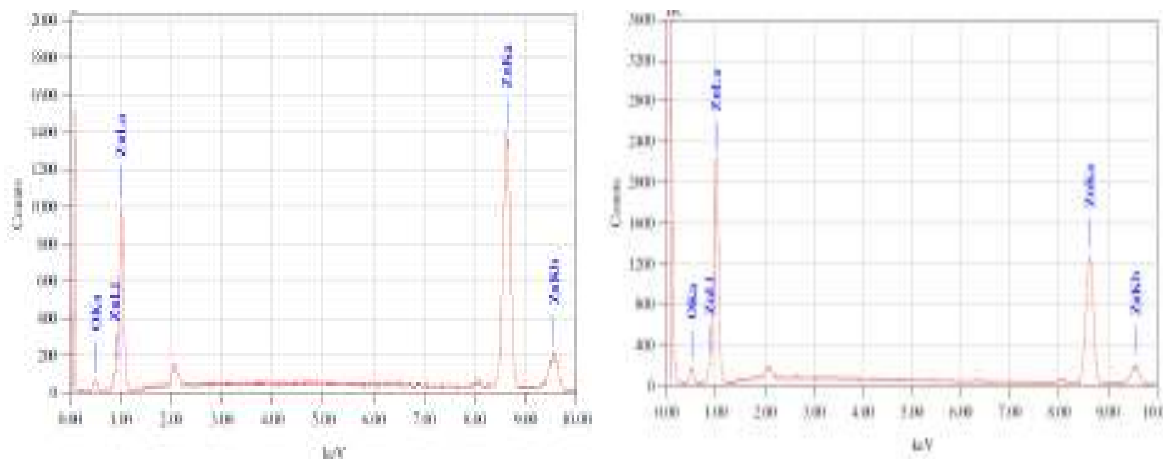


Fig-6. EDS spectrum of ZnO nanoparticles with and without CTAB as a surfactant nano structure.

Conclusion

The comparative study of ZnO nano materials were successfully done by using sol-gel method using zinc acetate dihydrate sodium hydroxide as a reactant and CTAB as the surfactant. The sol-gel method was performed in aqueous medium, hence, we called it as environmentally benign method. The XRD pattern gives that the synthesized materials were crystalline in nature. The energy dispersive spectroscopy shows the presence of Zn as well as TEM gives the information about crystal size changes of ZnO in both the condition (i. e. without CTAB and with CTAB). The morphology of absorption of ZnO nanomaterial were studied by using the spectroscopic analysis.

Conflict Of Interest

The authors confirm that this article content has no conflict of interest.

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