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Effects of Oxalic Acids on Surface Morphology of Synthesized ZnO Nanomaterials

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ABSTRACT: - *Nano-sized crystals of zinc oxide (ZnO) were synthesized, with a size of 26 nm. The products have been subjected to thorough characterization using SEM (Scanning Electron Microscope, and EDX (Energy Dispersive X ray). These investigations confirm the formation of zinc oxide nanoparticles with different surface morphology. Two samples were synthesized; one with Oxalic acid and the other without Using SEM and EDX, their morphology was investigated. The images obtained by SEM showed rod shaped clusters of nanoparticles which were distributed well within a range of 100 nm which is a favorable property to exhibit better adsorption and photo catalytic activity. The EDX results showed elemental composition of ZnO nanoparticles which showed 54% Zn, 44.07% O and 1.93% Mn impurities for L_1 and 55.34% Zn, 42.3% O and 2.37% Mn impurities for L_2.*

Keywords: - Characterization, Adsorption, ZnO, Nanoparticles, Morphology

INTRODUCTION

Most organic stabilizers for semiconductor nanoparticles such as organic thoils oleic acids, urea, oxalic acids and others are water soluble. Oxalic acid is an organic compound which is used during synthesis of ZnO nanoparticle. It is used as a chemical capping in which it is used to passivate the surface of particles so that that they do not agglomerate or ripen to form larger particles. The capping agent provides protective organic shell to particles to prevent the nanoparticles from aggregating in solution. It also promotes formation of fewer larger nuclei and thus nano crystal particle sizes (Gnanasangeetha and Sarala, 2013). Stabilizers enable the nanoparticles to be resistant to deactivation and thus its performance. The crystal structure of nano ZnO without as well as with oxalic acid has no effect on ZnO's structure as revealed by X-ray diffraction. SEM reveals different nature of surfaces and microstructures for nano ZnO obtained with and without stabilizers (Rajesh and Raychaudhuri, 2013). Nanoparticles have two key properties that make them particularly attractive as sorbents. On a mass basis, they have much larger surface areas than bulk particles. Nanoparticles can also be functionalized with various chemical groups to increase their affinity towards target compounds. Photo-catalytic nanomaterials allow ultraviolet light also used to destroy pesticides, industrial solvents and germs (Yantasee *et al*., 2007).Zinc oxide nanoparticles have been used to remove arsenic from water, even though bulk zinc oxide cannot adsorb arsenic.

EXPERIMENTAL

ZnO nanoparticles were synthesized using precipitation method by synthesizing two sets of ZnO nanoparticles. In the first set, 100 ml of 1M ZnSO4 solution was added to 100 ml of 2M NaOH solution in drops. When the addition was complete, the mixture was kept at room temperature under constant stirring using magnetic stirrer for a period of 2-4 hours (Zhang et al., 2010).

The resultant precipitate obtained was filtered then rinsed with distilled water. The formed white precipitate of $Zn(OH)_2$ was allowed to settle, filtered using filter paper of pore size 0.4 µm in a suction pump, washed with distilled water several times and dried in hot oven at 150^0C for 45 minutes. The synthesized ZnO nanoparticles were further irradiated at 180 W with microwave radiation in a microwave oven for 30 minutes and the sample was named L_1. The procedure was repeated to synthesize ZnO nanoparticles in different experimental conditions. $ZnSO_4$, NaOH and oxalic acid were used as stabilizing agents. Thus one more sample was obtained and referred to as L_2.

Morphological characteristics of ZnO nano particles

The synthesized ZnO nanoparticles were subjected to, SEM and EDX methods of analysis.

RESULTS

SEM Analysis

Figures 1 and 2 show the SEM diagram for samples $L_1$and L_2 at high magnification These images show that the ZnO nanoparticles obtained formed rod shaped clusters distributed within the range of 100 nm. The diagrams also show that the surface was not uniform but porous in nature. Thus the nanocatalyst has considerable number of pores hence a good possibility for the heavy metals to be trapped and adsorbed onto these pores and it is a good sign for effective adsorption of heavy metals (Joshi & Shrivastava, 2012).The photographs also show different surfaces for $L_1$and L_2. The L_1showed round ended while L_2 showed sharp ended nanoclustures. Therefore the stabilizing agent had an influence on the morphology of the samples. Similar studies were made by Soltaninezhad and Amrnifar (2011). They studied surface morphology of ZnO nanoparticles produced by spray pyrolysis. The pictures observed showed particles that were spherical in shape. However, Joshi and Shrivastava (2012) determined the surface texture which was found to be rough and porous in nature. Also Shanthi and Muthuselvi, (2012) under a similar study, characterized nano ZnO synthesized by precipitation and the SEM pictures which indicated sphere and cube like nanoparticles which were distributed within the range of 100nm. Due to these close similarities, the ZnO nanoparticles were confirmed.

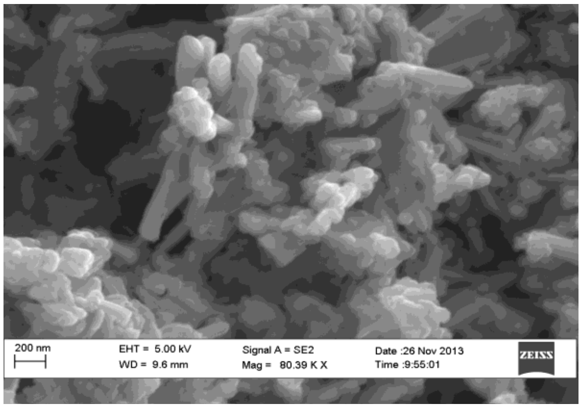

Figure 1: Magnified L_1 SEM diagram

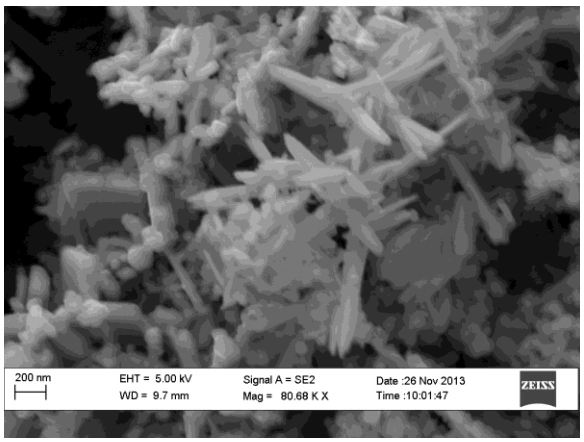

Figure 2: Magnified L_2 SEM diagram

The difference in distribution range is attributed to the level of accuracy during synthesis and also method of synthesis (Joshi & Shrivastava, 2012).

EDX ANALYSIS

Figures 3 and 4 show the EDX spectra for sample $L_1$and L_2. Considerable number of pores hence a good possibility for the heavy metals to be trapped and adsorbed onto these pores and it is a good sign for effective adsorption of heavy metals (Joshi & Shrivastava, 2012).The photographs also show different surfaces for $L_1$and L_2. The L_1showed round ended while L_2 showed sharp ended nano clusters. Therefore the stabilizing agent had an influence on the morphology of the samples. The EDX spectra indicated that the samples were made up of Zn, O and traces of Mn impurities. The peak at the intense peak is assigned to the bulk ZnO and the less intense on to the surface ZnO. The peak at 0.5 Kev can only be attributed to O and not Mn due to overall position of the peaks. The elemental composition of the nanomaterial was found to be 55.34% Zn, 42.3% O and 2.37% Mn for $L_1$and 54% Zn, 44.0% O and 1.93% Mn for L_2.

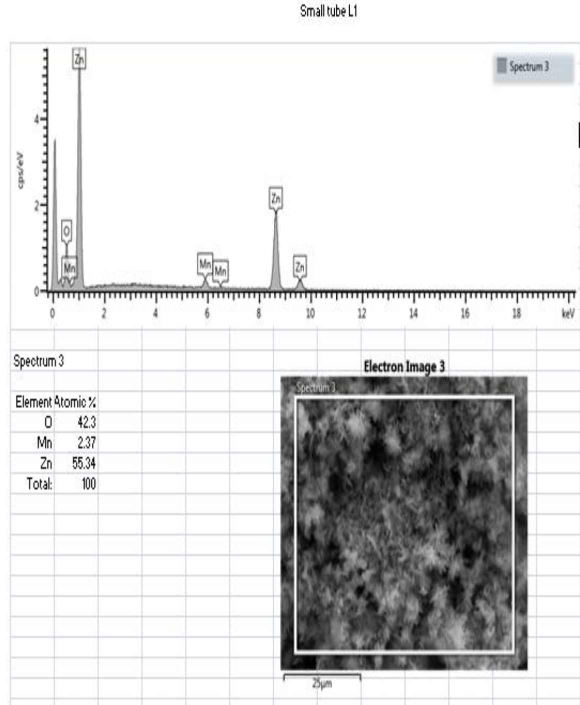

Figure 3 EDX Pattern ZnO L_1

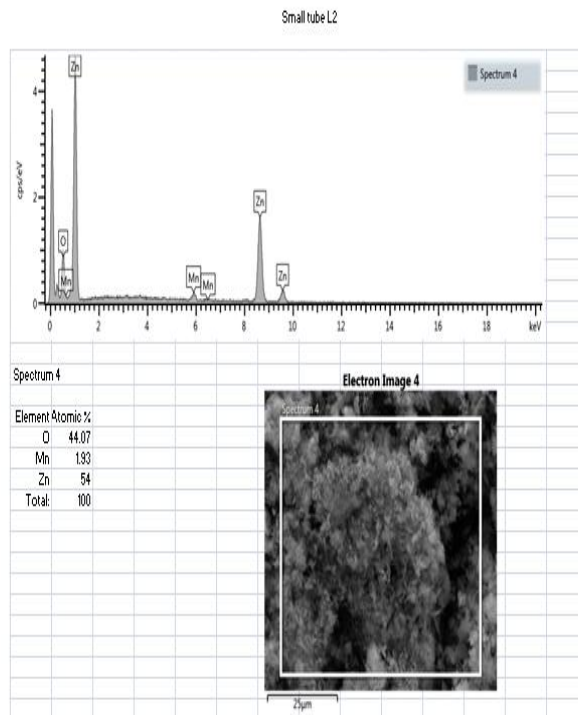

Figure 4 EDX Pattern ZnO L_2

CONCLUSION

These images show that the ZnO nanoparticles obtained formed rod shaped clusters distributed within the range of 100 nm. The diagrams also show that the surface was not uniform but porous in nature. Therefore the stabilizing agent had an influence on the morphology of the samples.

RECOMMENDATIONS

The microbial threats to human health and safety are a serious public concern. Thus, further improvements must be made in the direction of the development of materials with greater stability (resistance to pH changes and concentrations of chemicals present in contaminated water) and the capacity for the simultaneous removal of multiple contaminants, such as toxic metal ions, organic dyes and bacterial pathogens. Treatment technologies should be developed for the purification of water in order to meet the demand of increased environmental pollution. Furthermore green approach should be adopted for stabilizing agents.

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