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Synthesis and characterization of zinc oxide nanoparticles (ZnO-NPs) via precipitation method

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Abstract
ZnO nanoparticles (ZnO-NPs) were synthesized by precipitation method in three different way *i.e.* synthesis through mixing the of 1M of zinc sulphate [$ZnSO_4$] and 2M of potassium hydroxides [KOH] (M_1), through mixing of 0.2M of zinc nitrate [$Zn(NO_3)_2$] and 0.4M of potassium hydroxide [KOH] (M_2), and by 0.5M of zinc nitrate $Zn(NO_3)_2$] and 1M of urea [$CO(NO_2)_2$] (M_3). The synthesized particles were characterized by energy dispersive x-ray spectrometry (EDaX) and UV-Vis spectroscopy. The results of EDaX found that average elemental analysis of nano ZnO particles which were 74.86 weight % and 45.88 atomic % of ZnO-NPs in M_1; 72.11 weight % and 45.51 atomic % in M_2, and 75.04 weight % and 45.69 atomic % of ZnO-NPs in M_3**.** The optical characterizations of the sample were recorded on UV-Vis indicated three various sample (M_1) red color peck; (M_2) yellow color peck and (M_3) green color peck of synthesis of ZnO-NPs. The band gap energy of the sample is measured by the linear portion of the graph which was confirmed by the presence of excitonic absorption at 380 nm.

Keywords: Characterization, EDaX, precipitation method, UV-Vis and ZnO nanoparticles

Introduction
Zinc oxide a chemical compound found naturally in the mineral called zincite has attracted much attention in recent times due to its low cost and because it can be obtained by simple techniques (Koudelka *et al.*, 2004) [1]. Chemical synthesis is one of the most important techniques which can be performed by using a range of precursors and different conditions like temperature, time, concentration of reactants, and so forth. Nanoparticles synthesis has become the great interest in recent time due to its desirable properties and application in various agriculture and non agriculture field.

Among the nanoscale metal oxides, zinc oxide is a common host material that has been widely used due to its excellent chemical and thermal stability, low cost and environmental-friendliness (Nural *et al.*, 2013) [2]. ZnO nanostructures have a great advantage to apply to a catalytic reaction process due to their large surface area and high catalytic activity (Chen and Tang, 2007) [3]. In general physical and chemical methods are more popular for nanoparticles synthesis. The present study focuses on the preparation of ZnO nanoparticles by precipitation method by using different-different compounds and characterized the synthesized ZnO nanoparticles.

Materials and Methods
ZnO nanoparticles were synthesized at Central Instrumentation Laboratory of Navsari Agricultural University, Navsari by using various chemical compounds viz. mixing of 1M of zinc sulphate [$ZnSO_4$] and 2M of potassium hydroxides [KOH] (M_1); 0.2M of zinc nitrate [$Zn(NO_3)_2$] and 0.4M of potassium hydroxide [KOH] (M_2) (Ghorbani *et al.*, 2015) [4] and by 0.5M of zinc nitrate $Zn(NO_3)_2$] and 1M of Urea [$CO(NO_2)_2$] (M_3) (Sabir *et al.*, 2014). [5] The detail procedures for synthesis of ZnO nanoparticles are given below.

Synthesis of ZnO-NPs (M_1) by zinc sulphate and potassium hydroxides
First of all prepared the aqueous solution of 1M zinc sulphate ($ZnSO_4$) and 2M of potassium hydroxide (KOH) solution. After this, added drop wise potassium hydroxides (2M) slowly in zinc sulphate (1M) under vigorous stirring. The stirring was continuous for 12 hrs. The precipitate obtained was filtered and washed thoroughly with deionised water. The precipitate was dried in the oven at 100 °C and ground to fine powder using agate mortar. The powder obtained from the above method was calcined at 500 °C temperature.

Synthesis of ZnO-NPs (M_2) by zinc nitrate and potassium hydroxides

The aqueous solution (0.2M) of zinc nitrate ($Zn(NO_3)_2$ and (0.4M) of KOH were prepared with deionized water. The potassium hydroxides solution was slowly added into zinc nitrate solution at room temperature under vigorous stirring. White precipitate as obtained which was centrifuged at 5000 rpm for 20 min and washed three times with distilled water. The obtained product was calcined at 500 °C for 3 hrs.

Synthesis of ZnO-NPs (M_3) by zinc nitrate and urea

First prepared 0.5M Zinc nitrate $Zn(NO_3)_2$ and 1M urea solution as precursors. This urea solution was added drop wise into zinc nitrate solution with vigorous stirring at 70 ^{0}C for 2 hrs to allow complete formation of nanoparticles. Added some drop sodium hydroxides for maintaining the basic pH. Precipitating solution turns whitish cloudy. This white precipitating product was centrifuged at 8000 rpm for 10 minutes and washed with distilled water for the removal of any impurities or absorbed ions if present. The product was calcinated at 500 ^{0}C for 3 hrs by using muffle furnace.

Results and Discussion

After the preparation of ZnO-NPs, different characterization techniques such as energy dispersive x-ray spectrometry (EDaX) and UV-Vis Spectroscopy were used to investigate their morphology, crystallite size, optical properties and band gap energies properties.

Characterization

EDaX (Energy dispersive X-ray spectrometer): EDaX spectrum clear that the synthesized of ZnO-NPs through various precipitation methods had recorded the presence of transition metal zinc 74.86 weight percent and 45.88 atomic percent of ZnO-NPs in M_1; Zinc 72.11 weight percent and 45.51 atomic percent in M_2 and Zinc 75.04 weight percent and 45.69 atomic percent of ZnO-NPs in M_3 (Table 1 and Fig. 1 to 3). Similar results were also reported by Yugandhar and Savithramma (2013) [6].

Table 1: Elemental analysis of ZnO-NPs

ZnO-NPs (M_1) $ZnSO_4$ and KOH		
Element	**Weight%**	**Atomic %**
O	21.30	53.34
Au	3.84	0.78
Zn	74.86	45.88
Total	100	
ZnO-NPs (M_2) $Zn(NO_3)_2$ and KOH		
Element	**Weight%**	**Atomic %**
O	20.53	52.95
Au	7.35	1.54
Zn	72.11	45.51
Total	100	
ZnO-NPs (M_3) $Zn(NO_3)_2$ and urea		
Element	**Weight %**	**Atomic %**
O	21.56	53.62
Au	3.40	0.69
Zn	75.04	45.69
Total	100	

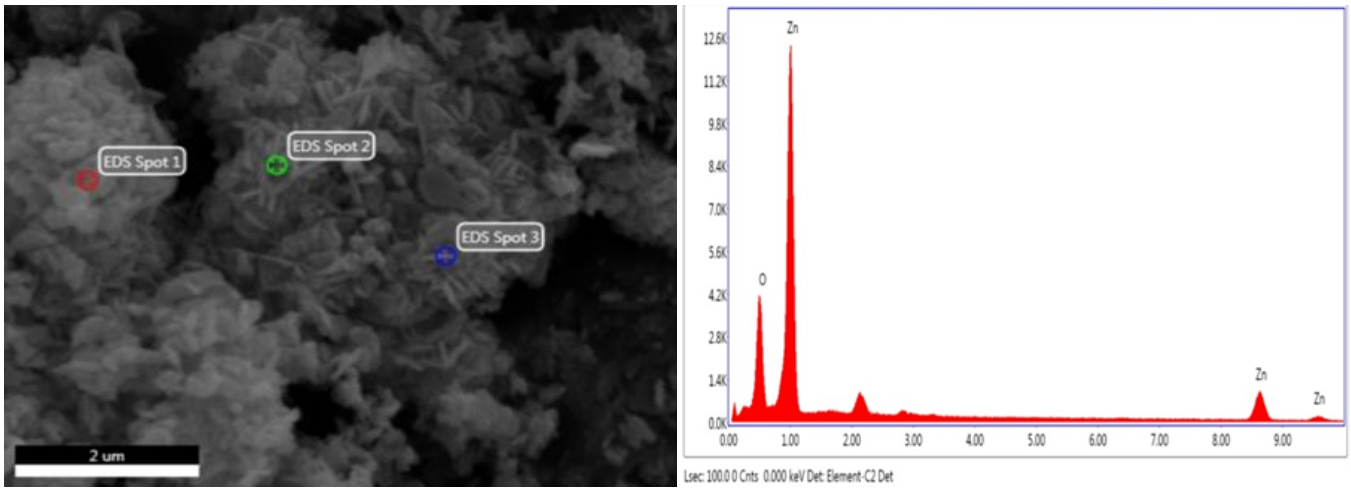

Fig 1: EDaX analysis of ZnO-NPs (M1)

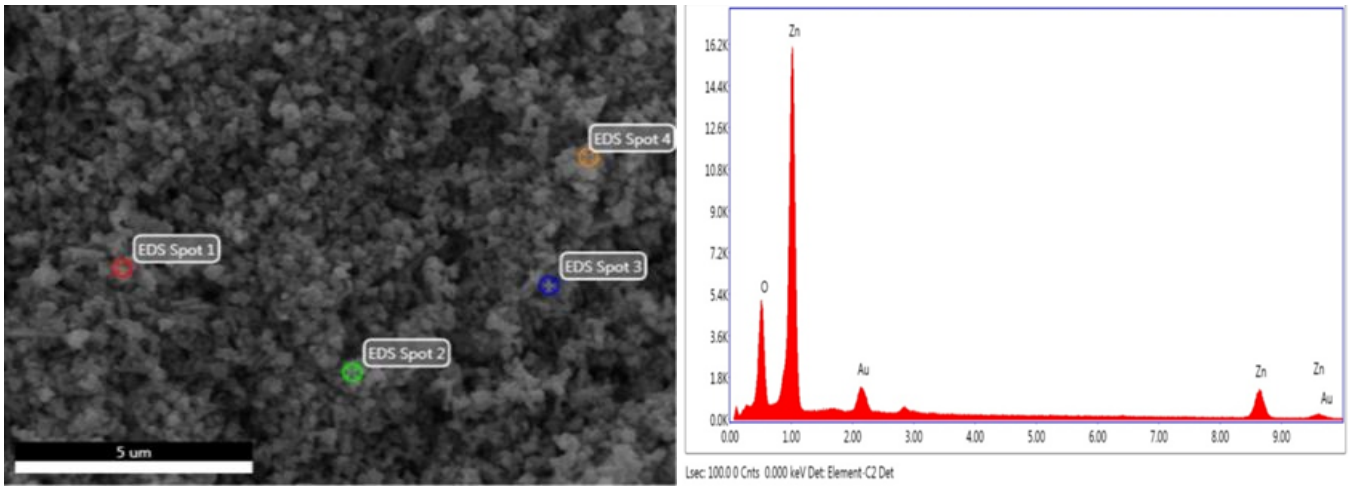

Fig 2: EDaX analysis of ZnO-NPs (M2)

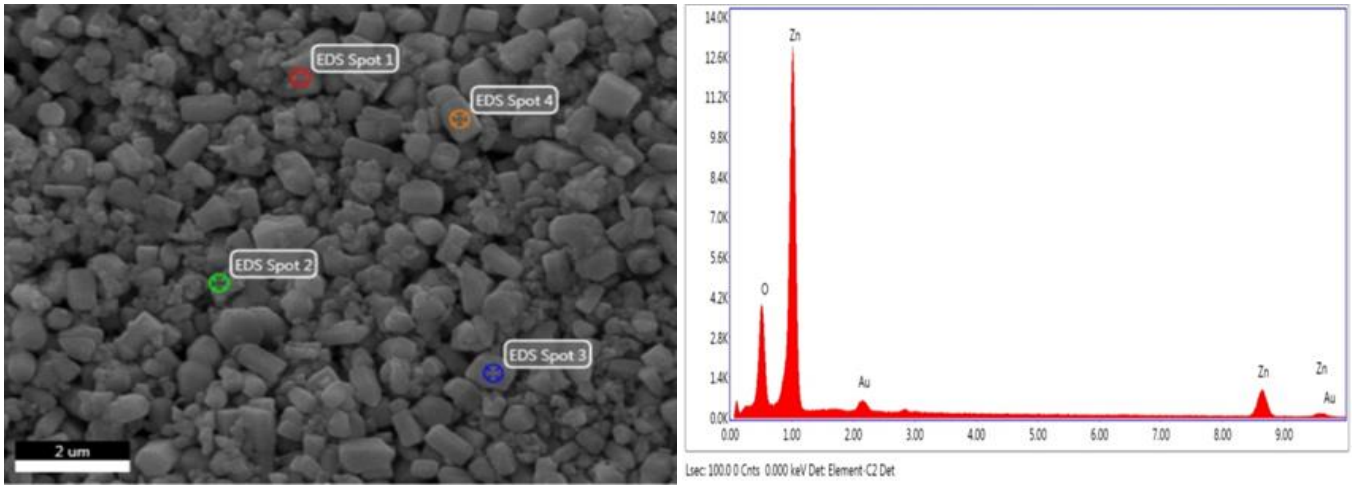

Fig 3: EDaX analysis of ZnO-NPs (M3)

UV-Vis Spectroscopy: The optical characterizations of the sample were recorded on UV-Vis spectroscopy, depicted in Fig. 4 indicated three various sample (M_1) $ZnSO_4$ + KOH show red color peck; (M_2) Zn $(NO_3)_2$+ KOH indicate the yellow color peck and (M_3) Zn $(NO_3)_2$ + urea present green color peck of synthesis of ZnO-NPs. The band gap energy of the sample is measured by the linear portion of the graph which was confirmed by the presence of excitonic absorption at 380 nm.

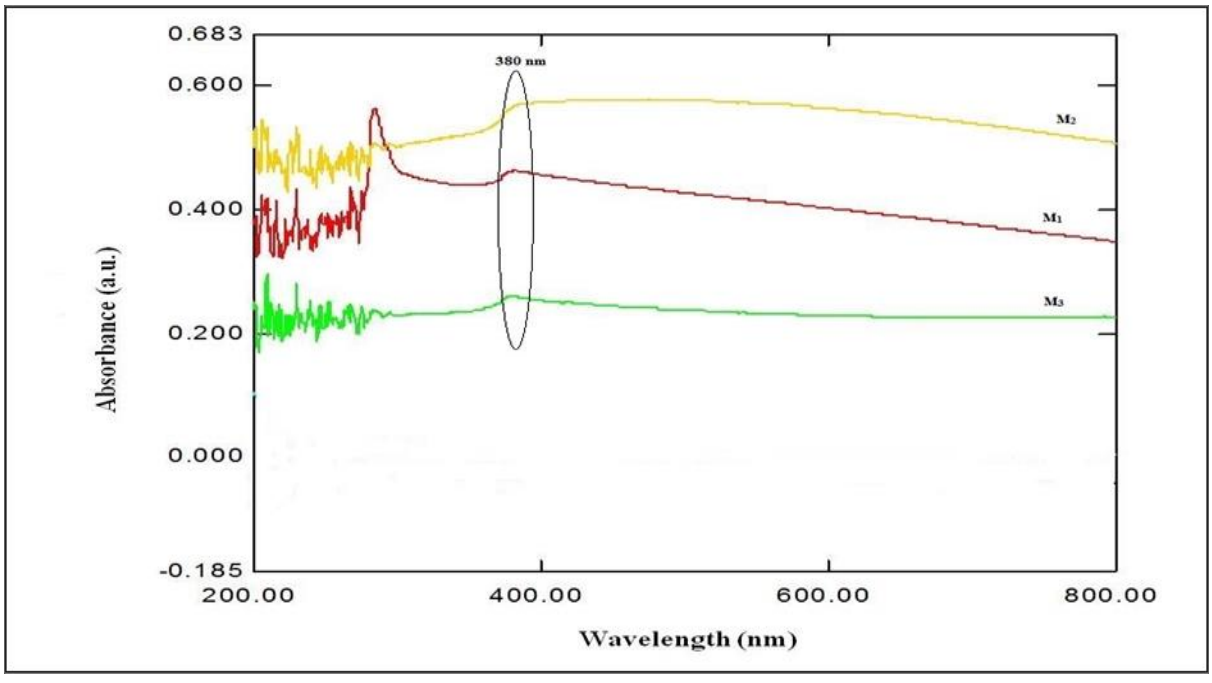

Fig 4: UV-Vis Spectroscopy analysis of ZnO-NPs

Conclusion

The synthesized ZnO nanoparticles by precipitation method are very easy. The characterization of ZnO nanoparticles by using EDaX techniques and UV-Vis spectroscopy shows that synthesized ZnO nanoparticles are good in morphology, high in purity and suitable for agriculture as fertilizers.

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