Synthesis and Characterization of ZnO Powder by Sol-Gel Method

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Abstract

Zinc oxide which plays an important role in current industry due to its special characteristics such as anti-corrosion, anti-bacteria, has low electrons conductivity and excellent heat resistance. Therefore, the objective of this study is to synthesize zinc oxide nanostructures with the most practical ways by using sol-gel method and characterize the nanostructures. Sol-gel method is the simplest method and has the ability to control the particle size and morphology through systematic monitoring of reaction parameters. ZnO particles were synthesized via sol gel method using Zinc acetate dehydrate (Zn $(CH_3COO)_2.2H_2O$) as a precursor and ethanol (CH_2COOH) was used as solvent, Sodium hydroxide (NaOH) and distilled water were used as medium. ZnO particles were characterized by using XRD analysis and SEM analysis. According to XRD results, ZnO particles was obtained the average crystallite sizes between were found 45 nm to 47 nm by 400°C, 500°C and 600°C. The shape of grain of ZnO particle was spherical feature and grain sizes of the ZnO particle were measured to be 0.700 µm at 400°C, 0.990 µm at 500 °C and 0.810 µm at 600 °C.

Keywords: ZnO, Sol-gel, XRD, SEM

INTRODUCTION

Synthesis of metal nanoparticles with specific properties is a newly established research area that attracts a great deal of attention. There are several methods that have been put forward for synthesis of these materials, namely chemical vapor condensation, arc discharge, hydrogen plasma-metal reaction, and laser pyrolysis in the vapor phase, microemulsion, hydrothermal, sol-gel, and microbial processes taking place in the liquid phase, and ball milling carried out in the solid phase. The properties of metal nanoparticles depend largely on their synthesis procedures. The variety of metal oxide is great and their range of properties and possible applications appear to be enormous. Zinc Oxide is very suitable for sensor and transducer usage with its relatively bio-safe and biocompatible material. Besides, nanostructured metal oxide has been found to display appealing nano-morphological, functional, biocompatible, non- toxic and catalytic properties. The market demand for the ZnO powder is increasing and widely 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 powder include electrophotography, photoprinting, capacitors, protective coatings, anti-microbial, and conductive thin-films in LCDs, solar cells, and blue laser diodes.

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MATERIALS AND METHOD

In this work, precursor of zinc oxide nanoparticles was synthesized by Sol-Gel method. The chemicals used for synthesis are 2g of Zinc acetate dehydrate, 8g of sodium hydroxide 100ml of ethanol and 25ml of distilled water. Instruments used for synthesis are Muffle furnace, Magnetic stirrer, X-ray diffractometer and Scanning electron microscope.

Synthesis of ZnO powder

The zinc oxide nanoparticles was synthesized by Sol-Gel method. In order to prepare a sol, 2 g of Zinc Acetate Dihydrate and 8 g of Sodium Hydroxide were weighted using a weighting balance. Then, 10 ml and 15 ml of distilled water were measured by beaker. After that, 2 g of zinc acetate dihydrate was dissolved with a 15 ml of distilled water and 8g of sodium hydroxide was dissolved in a 10 ml of distilled water. Each solution was stirred with a magnetic stirrer for about five minutes and 600 rpm. After mixed solution, sodium hydroxide solution and zinc acetate solution with a constant stirring by magnetic stirrer for about 15min and 600rpm. Then, a glass tube was filled with 100 ml of ethanol dropwise and tiltrate to the solution containing both sodium hydroxide solution and zinc acetate. The mixed solution stirred with a magnetic stirrer with 600rpm at increase 50℃-250℃ at once 15min to obtain gel. The obtained gel was dried at 100℃ for 2hr then ground into a fine particle. The temperature of the dried precursor powder was annealed at 400℃, 500℃ and 600℃ for 2 hours to obtain the final product (i.e., ZnO powder).

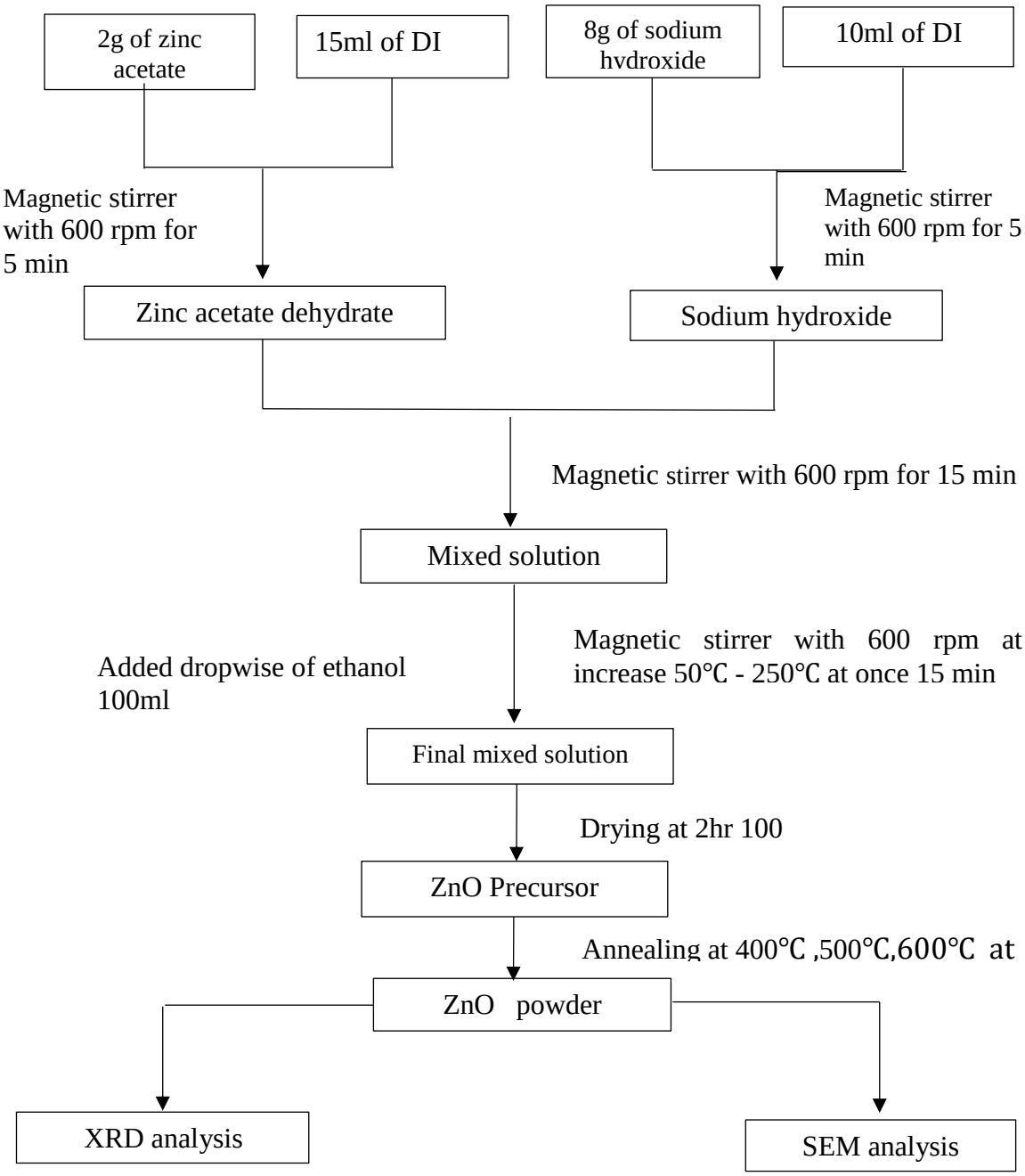

Figure 1. Flow chart of synthesis and characterization of zinc oxide by sol-gel method

RESULTS AND DISCUSSION

XRD Analysis

X-ray diffraction (XRD) was performed using monochromatic Cu-Kα radiation (λ = 1.54060 Å) operated at 40 kV(tube voltage) and 30 mA(tube current) was used to identify crystalline phases and to estimate the crystalline sizes. Specimen was scanned from 20° to 70° in diffraction angle 2θ and sampling pitch 0.02(deg). The XRD spectra of zinc oxide annealed at 400℃, 500℃ and 600℃ for 2hr were shown in fig. From Figure 1(a) to Figure 1(c), nine distinct peaks such as (100), (002), (101), (102), (110), (103), (200), (112) and (201) were formed on the XRD profile of ZnO (pure). All peaks were well matched with the standard

profile of ZnO. The strongest peak of ZnO was found to be (101) plane in all XRD patterns. XRD analysis showed that crystallized in the hexagonal wurtzite structure. The mean size of the ordered ZnO nanoparticles has been estimated from full width at half maximum (FWHM) and Debye-Scherrer formula according to equation the following:

$$G = \frac{k \times \lambda}{\beta \times \cos \theta_B}$$

where β is the peak width measured at half intensity (radian), λ is the wavelength measured in Å, k is the particle shape factor or Scherrer constant (k = 0.9) and G is the diameter of the crystallites (Å). From table (1.1) to (1.8), the average crystallite sizes of the ZnO were found to 47.5405 nm at 400 ºC, 45.4229 nm at 500 ºC, and 46.6122 nm at 600 º C.

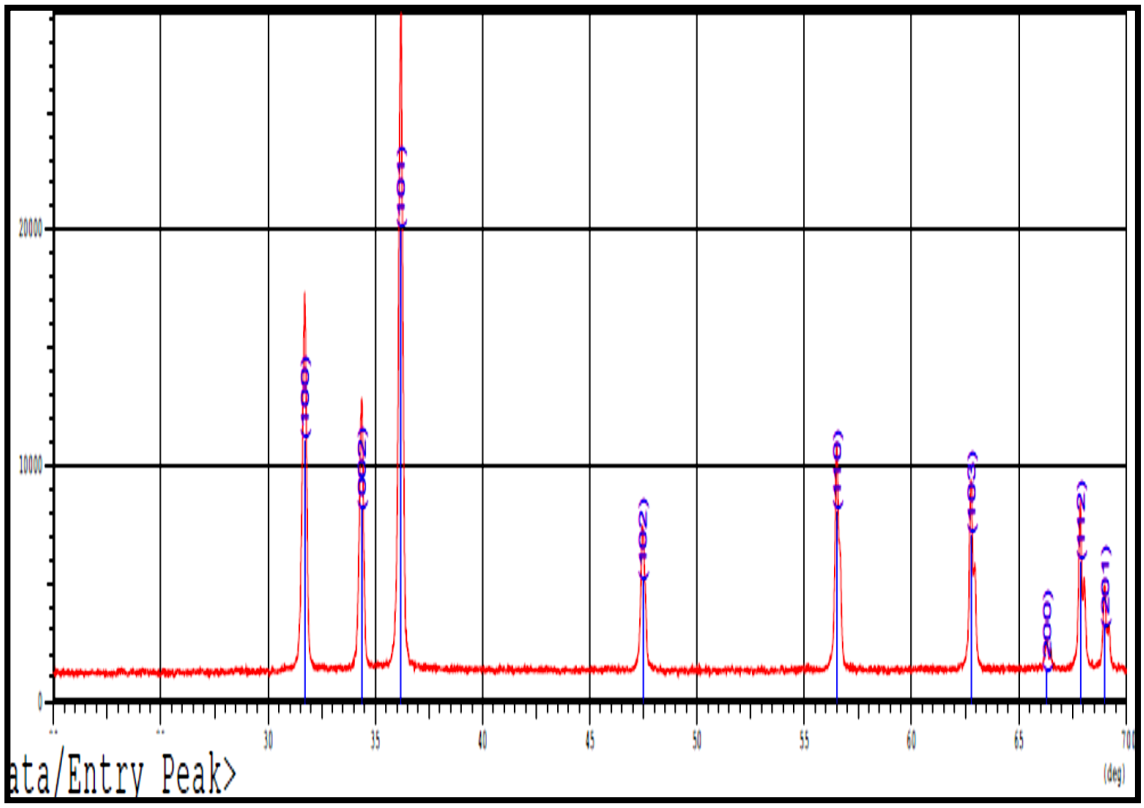

Figure 1(a). XRD pattern of ZnO at 400 º C for 2hr

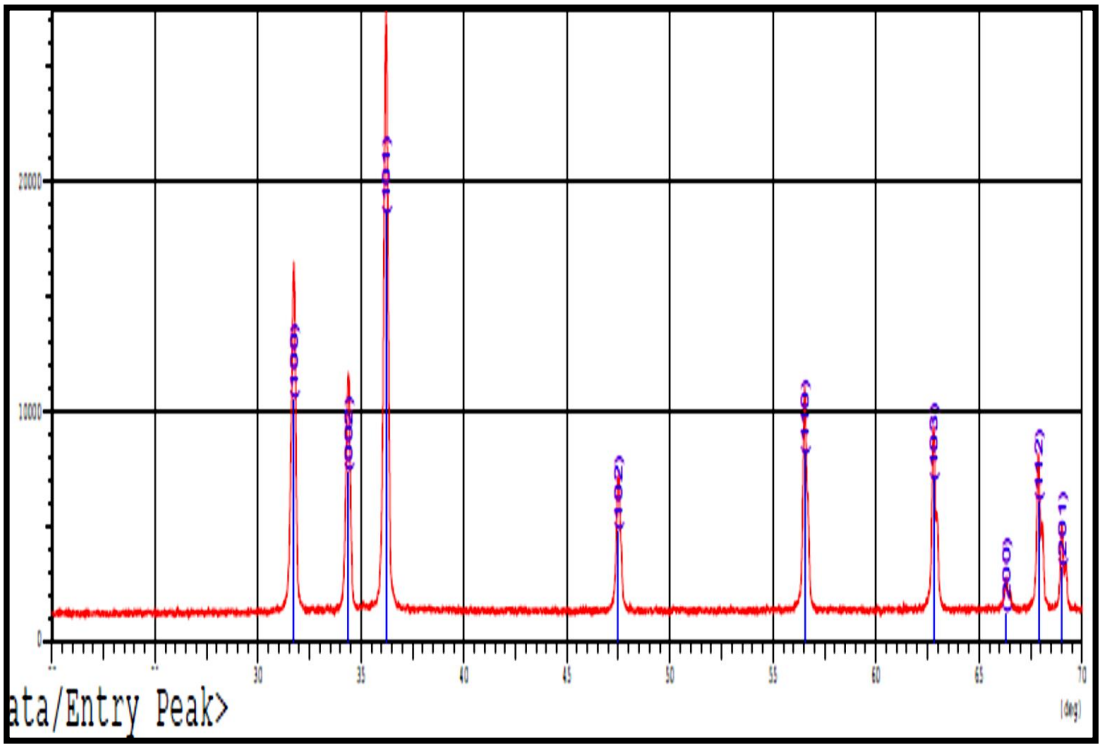

Figure 1(b). XRD pattern of ZnO at 500 º C for 2hr

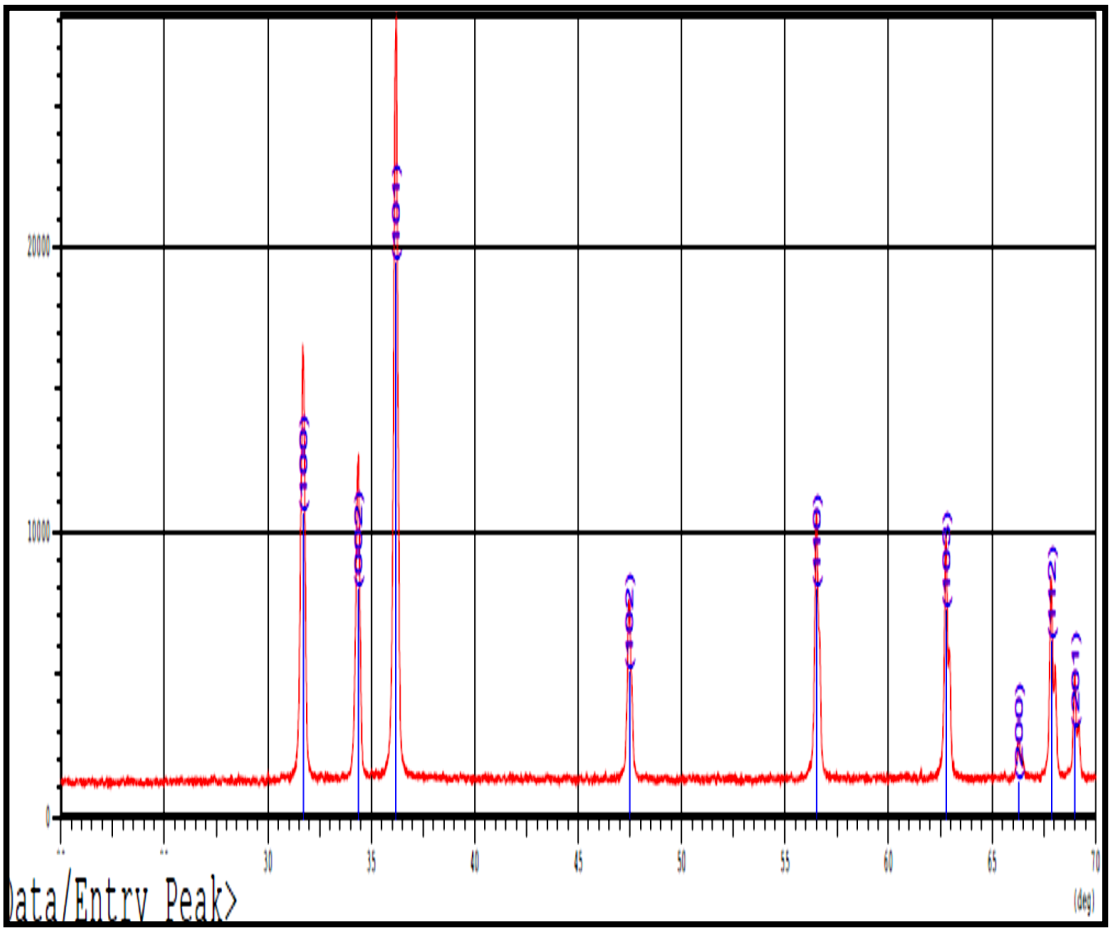

Figure 1(c). XRD pattern of ZnO at 600 º C for 2hr

Table 1.1 Diffraction angles of all Identified peak for ZnO Powder

No	**Peaks**	**Lattice Spacing d(A)**	**Standarded Values 2θ (deg)**
1.	(1 0 0)	2.82009	31.7033
2.	(0 0 2)	2.60816	34.3561
3.	(1 0 1)	2.48039	36.1855
4.	(1 0 2)	1.91395	47.4650
5.	(1 1 0)	1.62693	56.5192
6.	(1 0 3)	1.47887	62.7812
7.	(2 0 0)	1.40859	66.3034
8.	(1 1 2)	1.37982	67.8713
9.	(2 0 1)	1.35983	69.0085

Table 1.2 Crystallite size of Pure ZnO Powder

No	Peks	FWHM (deg)	Crystallite size (nm)
1.	(1 0 0)	0.2242	36.9577
2.	(0 0 2)	0.2090	39.2238
3.	(1 0 1)	0.2080	40.5184
4.	(1 0 2)	0.1895	45.8977
5.	(1 1 0)	0.1771	50.7794
6.	(1 0 3)	0.1813	50.7586
7.	(2 0 0)	0.1771	53.4230
8.	(1 1 2)	0.1725	55.7068
9.	(2 0 1)	0.1741	56.0841
Average Crystallite size			47.7055

Table 1.3 Diffraction angles of all Identified peak for ZnO Powder at 400^{o}C

No	Peaks	Lattice Spacing d (A)	Standard Values 2θ (deg)
1.	(1 0 1)	2.81941	31.7111
2.	(0 0 2)	2.60772	34.3621
3.	(1 0 1)	2.47998	36.1916
4.	(1 0 2)	1.91365	47.4727
5.	(1 1 0)	1.62680	56.5242
6.	(1 0 3)	1.47879	62.7852
7.	(2 0 0)	1.40861	66.3027
8.	(1 1 2)	1.37974	67.8758
9.	(2 0 1)	1.35974	69.0140

Table 1.4 Crystallite size of ZnO Powder at 400 ºC

No	Peaks	FWHM (deg)	Crystallite size (nm)
1.	(1 0 0)	0.2151	37.9310
2.	(0 0 2)	0.2076	40.3140
3.	(1 0 1)	0.2056	40.5191
4.	(1 0 2)	0.1888	45.8991
5.	(1 1 0)	0.1813	49.1937
6.	(1 0 3)	0.1774	52.3971
7.	(2 0 0)	0.1756	53.4228
8.	(1 1 2)	0.1767	53.9112
9.	(2 0 1)	0.1792	54.2767
Average Crystallite size			47.5405

Table 1.5 Diffraction angles of all Identified peak for ZnO Powder at 500ºC

No	Peaks	Lattice Spacing d (A)	Standard Values 2θ (deg)
1.	(1 0 0)	2.81676	31.7417
2.	(0 0 2)	2.60597	34.3859
3.	(1 0 1)	2.47816	36.2192
4.	(1 0 2)	1.91264	47.4995
5.	(1 1 0)	1.62611	56.5504
6.	(1 0 3)	1.47827	62.8097
7.	(2 0 0)	1.40819	66.3249
8.	(1 1 2)	1.37930	67.9005
9.	(2 0 1)	1.35930	69.0395

Table 1.6 Crystallite size of ZnO Powder at 500 ºC

No	Peaks	FWHM (deg)	Crystallite size (nm)
1.	(1 0 0)	0.2338	35.1583
2.	(0 0 2)	0.2282	36.2849
3.	(1 0 1)	0.2212	37.4052
4.	(1 0 2)	0.1978	43.2808
5.	(1 1 0)	0.1839	49.1998
6.	(1 0 3)	0.1803	50.7663
7.	(2 0 0)	0.1901	50.1914
8.	(1 1 2)	0.1824	52.2341
9.	(2 0 1)	0.1774	54.2850
Average Crystallite size			45.4229

Table 1.7 Diffraction angles of all Identified peak for ZnO Powder at 600ºC

No	Peaks	Lattice Spacing d (A)	Standard Values 2θ (deg)
1.	(1 0 0)	2.81956	31.7094
2.	(0 0 2)	2.60782	34.3608
3.	(1 0 1)	2.48000	36.1914
4.	(1 0 2)	1.91384	47.4677
5.	(1 1 0)	1.62681	56.5238
6.	(1 0 3)	1.47881	62.7842
7.	(2 0 0)	1.40874	66.2955
8.	(1 1 2)	1.37979	67.8729
9.	(2 0 1)	1.35972	69.0151

Table 1.8 Crystallite size of ZnO Powder at 600 ºC

No	Peaks	FWHM (deg)	Crystallite size (nm)
1.	(1 0 0)	0.2285	36.0343
2.	(0 0 2)	0.2150	38.1920
3.	(1 0 1)	0.2089	39.4240
4.	(1 0 2)	0.1878	45.8982
5.	(1 1 0)	0.1834	49.1936
6.	(1 0 3)	0.1783	52.3968
7.	(2 0 0)	0.1886	50.1830
8.	(1 1 2)	0.1780	53.9104
9.	(2 0 1)	0.1775	54.2772
Average Crystallite size			46.6122

SEM analysis

The surface morphologies of ZnO particle were studied using scanning electron microscope (SEM). The SEM image of all of ZnO particle were seemed to be crack-free and uniform grain distribution. Some grains were separated by holes and some were in continuity[9]. The grain size was measured by using well known bar code system, drawing cross bars of some dimensions as provided scale and measuring number of interesting grains across them. From Figure 2(a) to Figure 2(c), the shape of grain of ZnO particle was spherical feature and grain sizes of the ZnO particle were measured to be 0.700 µm at 400 °C, 0.990 µm at 500 °C and 0.810µm at 600 °C.

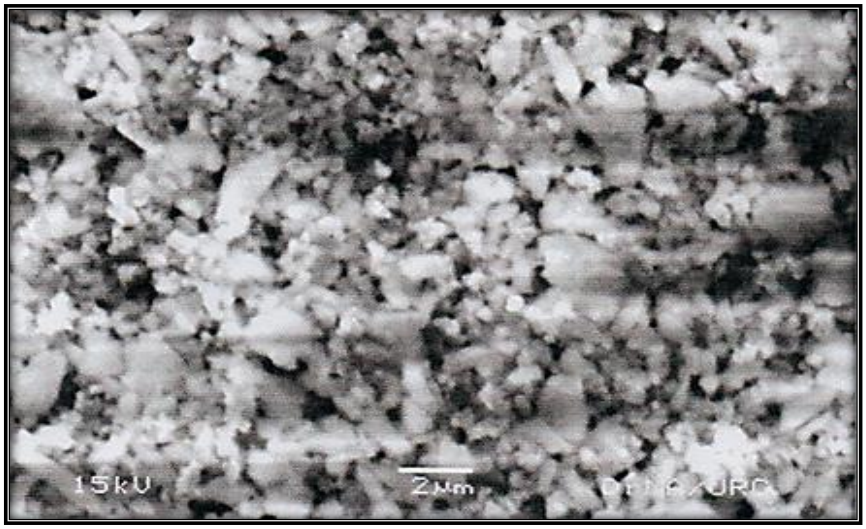

Figure 2(a). SEM image of ZnO powder at 400 °C

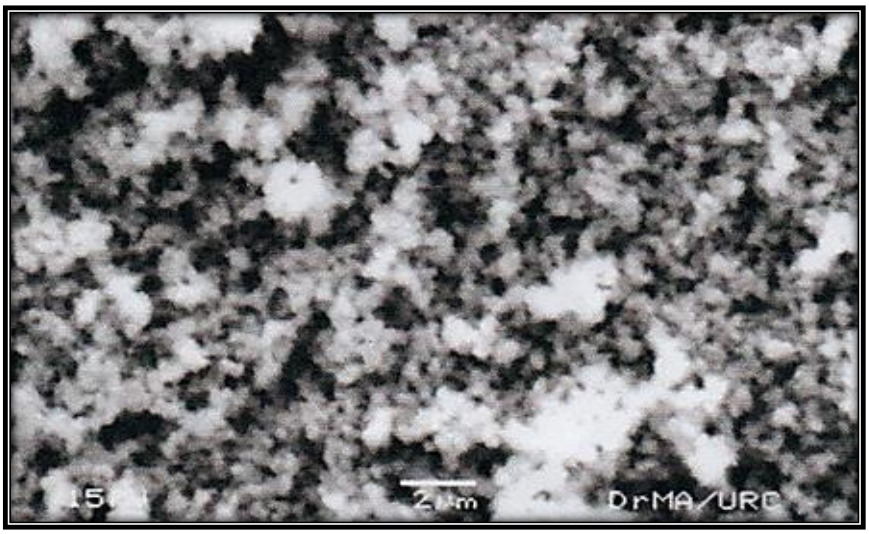

Figure 2(b). SEM image of ZnO powder at 500 °C

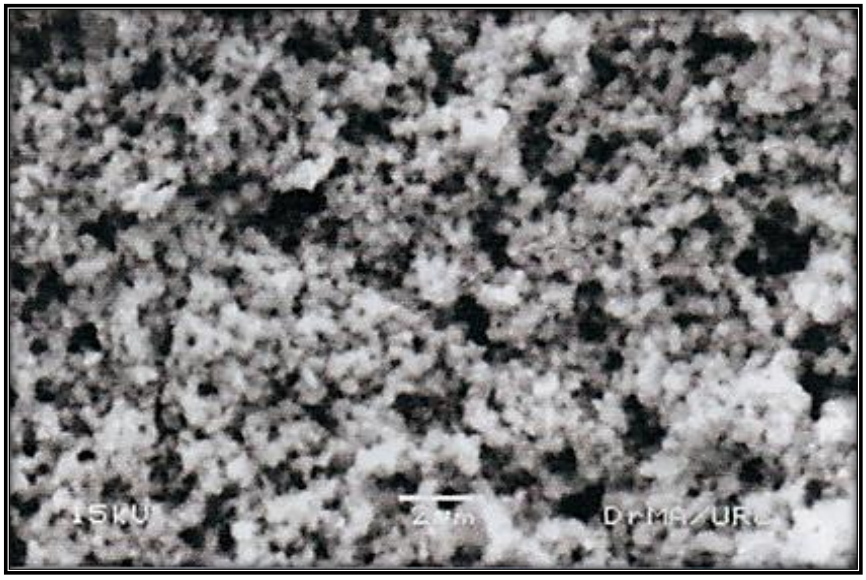

Figure 2(c). SEM image of ZnO powder at 600 °C

CONCLUSION

ZnO particles have been successfully synthesized by simple Sol-Gel method using zinc acetate dehydrate and sodium hydroxide. The prepared ZnO particles were spherical in shape and were characterized using XRD, and SEM techniques. The average crystallite sizes of the ZnO were found to 47.5405 nm at 400ºC, 45.4229 nm at 500ºC, and 46.6122 nm at 600ºC. According to XRD result, crystallite size well matched 45.4229 nm at 500ºC more than at 400ºC and 600ºC. The synthesized ZnO powder obtained exhibit good crystallinity. The shape of grain of ZnO particle was spherical feature and grain sizes of the ZnO particle were measured to be 0.700 µm at 400°C, 0.990 µm at 500°C and 0.810 µm at 600°C. From SEM image, it is clear that with increasing temperature the particles shape were changes to the spherical and less agglomerate.

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