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STUDIES ON NANOSTRUCTURED ZnO AS SUN-SCREEN COMPONENT

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ABSTRACT

The popularity of the sunscreens is significantly increased since ultraviolet (UV) radiations have foremost effects in the skin aging. The size of the materials especially nano-sized materials used in lotions also has major contribution. In this nano-particle era, it is desired to synthesis legitimate materials for the sunscreens. Among the category of organic and inorganic materials, inorganic materials provide the best spectrum protection against UV radiations as a physical Sun blocker dominant than chemical sunscreens. Thus, as one of the pass up elements among all is considered to be a zinc oxide and its synthesis. Furthermore efforts can be taken in this direction by synthesizing such inorganic material for skin protection, aging, filters and tumor preventers with the control of its particle size rather than crystallite size and hence has been studied with respect to X-ray diffraction (XRD) and Transmission Electron Microscopic (TEM) images followed by UV-VIS study to obtain 3.185 eV optical band gap.

Key words: Inorganic, Sunscreens, Nano-particles, XRD, TEM, Optical band gap.

INTRODUCTION

A representative II–VI compound semiconductor, zinc oxide (ZnO) has attracted considerable attention due to its many attractive properties when studied it in the past decade. Its excellent UV absorption of ZnO can be utilized in cosmetics, paints, varnishes, and plastics[1,2]. Performance of ZnO powders in the UV absorption applications is determined primarily by these applications. The applications are based on particle size, morphology, agglomeration level, defect structure, and chemical composition. It is to be noted that high UV absorption of it can be determined by the crystallographic and chemical properties mainly. It has the direct wide band gap around 3.37 eV, large exciton binding energy of 60 meV at room temperature, good piezoelectric characteristics, chemical stability and biocompatibility. Especially its biocompatibility suggests a host of possible practical applications, notably in the area of cosmetics. ZnO have been used in sunscreens as it has effective broad-spectrum sunscreen agents particularly in blocking the UVA spectrum, which may not be covered adequately by normal chemical sunscreens. As large microscopic particles, ZnO appear as white pastes, however, if particles are made to nano-size, they become too small to scatter or reflect visible light and become transparent, even as still absorbing UV radiation (UVR), resulting in significant aesthetic improvement[3]. Therapeutic Goods Association of Australia estimated that 30% of ZnO-based sunscreens contained nano-particles[4]. Therefore, the development of materials like zinc oxide is

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highly desirable as sunscreen agent. Hence, here in this article, it is studied with respect to synthesis, XRD and TEM to focus on its nano-structure.

EXPERIMENTAL

Materials and method

Commercially available chemical Zn $(NO_3)_3$ (Zinc nitrate) well crushed in mortar from Merck (India) with 99% purity is magnetically stirred in water for 2 h at 100^{o}C to get homogeneous solution and then dried powder. White powder so formed is crushed well for half an hour to get uniformity. It is then calcined at 600^{o}C for 4 h at the cooling rate of 1^{0}C/min. The powders were structurally characterized by XRD (Cu-Kα radiations) and by Transmission Electron Microscopic studies along with Electron Diffraction (TEM-ED). The purity is ensured.

RESULTS AND DISCUSSION

XRD Studies

XRD pattern of ZnO powder sample is shown in Fig. 1. Hexagonal phase has been observed and confirmed for the powder. It was compared with the patterns of standard JCPDS card file number 79-0208. The major peak positions are indexed. Phase conversion ratio is also calculated for ZnO at calcination temperature 600^{o}C, it is found that the average crystallite size is 52 nm. The percentage phase conversion ratio of the synthesized zinc oxide powder calculated from first three major XRD peaks of experimental data to the standard values reported in JCPDS file was estimated[5]. It was found to be 101.99 %. It shows high phase purity. Nano-crystalline powder has major peak at (101) with space group P63mc (a = b = 3.264 A^o and c = 5.219 A^o) at room temperature. Debye-Scherer equation (1) is used to calculate average crystallite[6].

$$D = \frac{K}{B\,Cos} \qquad \ldots(1)$$

Where, B is the full width at half-maximum intensity (FWHM in radians) of a peak at an angle θ; K is a constant (0.89) depending on the line shape profile; λ (1.542 A.U.) is the wavelength of the X-ray source.

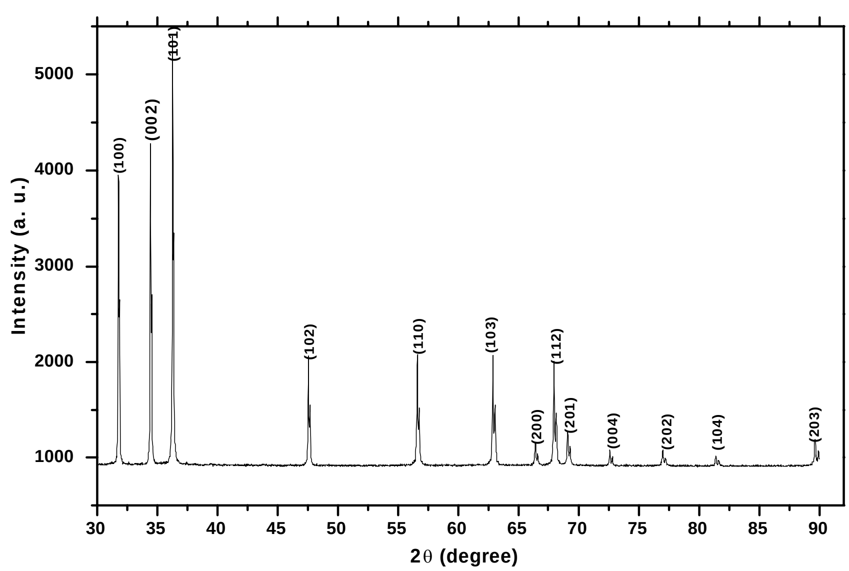

Fig. 1: Indexed XRD pattern of ZnO calcined at 600^{o}C

Transmission electron microscopy and electron diffraction study

Figure 2 shows at 600^{o}C, TEM-ED images of nano-crystalline ZnO along with corresponding electron-diffraction pattern. Size of nano-crystallites calculated from XRD data and particle size from TEM images are having good match. The diffraction spot patterns of hexagonal structure ZnO indicate that the nano-particles are highly crystalline. Some of the selected images are also shown here. The observed grain size and its smaller deviation from crystallite size depict little agglomerations of the crystals of ZnO to form clusters of somewhat larger size. It also shows grain growth within the range of 25 nm to 60 nm. The average crystalline size calculated from XRD is 52 nm, it shows diffused crystallites to form grain coalesce. The indexed ED pattern for the XRD peaks 100, 002, 101, 200, 102 and 103 is shown.

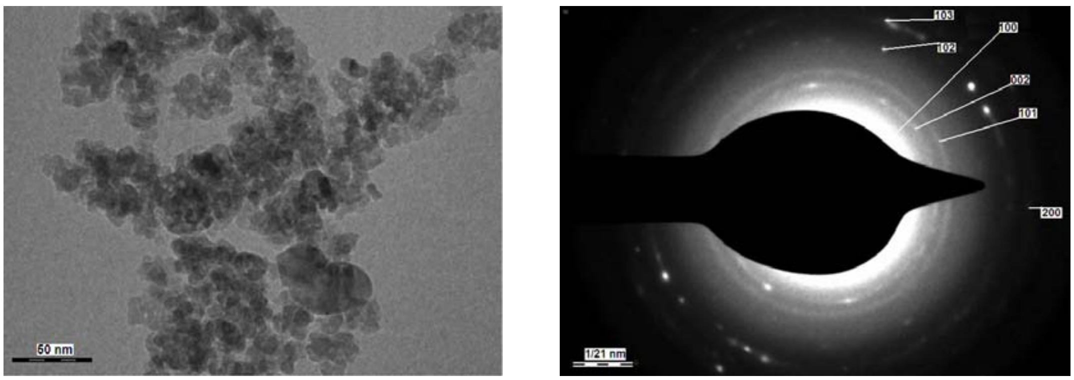

Fig. 2: Selected area TEM image of ZnO powder (with indexed ED)

Purity of ZnO

The purity of ZnO is necessary to check before its use as sunscreen component. The method is routine acid base titration. The purity of synthesized material ZnO is checked by using formula (2) as follows.

$$\text{Zinc oxide percent by mass} = \frac{4.07\,(B - A)}{M} \quad \ldots(2)$$

where, A = Volume in ml of standard sodium hydroxide solution used in the Titration with ZnO.

B = Volume in ml of standard sodium hydroxide solution used in the blank determination i.e. without ZnO.

M = mass in gm of ZnO to be tested

A = 7.39 mL, B = 7.75 mL, M = 1.5 g

Hence, Zinc oxide percent by mass = 0.97 x 100

Zinc oxide % = 0.97 x 100 = 97 %

It is 97% pure.

UV-VIS Studies

The UV spectrum of the synthesized ZnO is shown in Fig. 3. To calculate the optical band gap of ZnO calcined at 600^{o}C is subjected to UV-VIS spectrometric measurements, wavelengths assigned to the peaks are directly plotted as fitment curves of exponential growth of the wavelength vs reflectance, the tangent to this plot of (hν) vs $(\alpha h\nu)^2$ intersecting to X-axis (hν) gives direct optical band gap of ZnO and it is observed to be 3.185 eV as shown in Fig. 3.

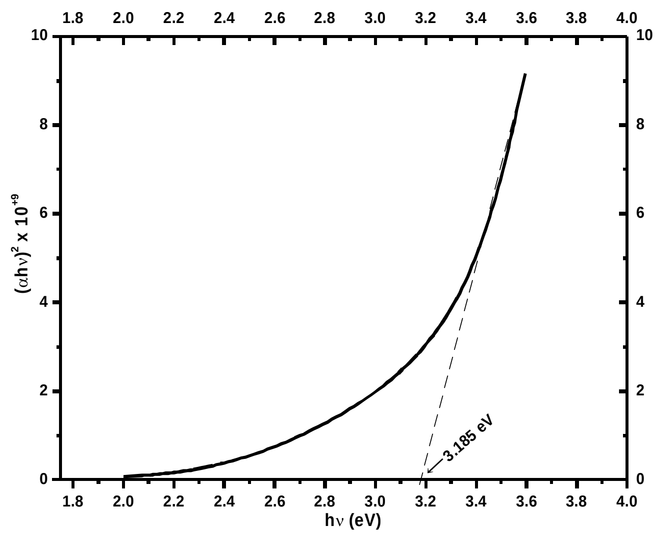

Fig. 3: Plot of $(\alpha h\nu)^2$ vs photon energy (hν) near the absorption edge for ZnO

CONCLUSION

We successfully synthesized hexagonal wruitzit ZnO. Phase purity of ZnO is obtained more than 100% when calculated from phase conversion ratio formula for the powder calcined at 600°C. At the same time mass purity of 97% is ensured from laboratory titration method. The average crystalline size calculated for it from XRD is 52 nm which is supported by TEM-ED observations. UV-VIS studies give optical band gap of 3.185 eV. Hence, it can be recommended as one of the sunscreen agents.

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