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ChemTech

International Journal of ChemTech Research
CODEN (USA): IJCRGG ISSN : 0974-4290
Vol.6, No.3, pp 2141-2143, May-June 2014

ICMCT-2014 [10th – 12th March 2014]
International Conference on Materials and Characterization Techniques

Quasi one dimensional ZnO Nano Structures using Micro wave heating

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Abstract: Quasi one dimensional ZnO nanostructures using amines of different chain lengths, namely hexyl amine, octyl amine and dodecyl amine were prepared. Characterization of the products was carried out using powder X-ray diffraction, UV-visible spectroscopy, Infrared Spectroscopy, Field Emission Scanning electron microscopy and Transmission Electron Microscopy.
Key words- One dimensional structure; Zinc oxide; Amines.

Introduction

A wide range of methods are reported in the synthesis of nano ZnO with diverse morphologies[1,2]. One of the methods, which is easy to adopt for the synthesis of quasi one dimensional nano structures is to synthesize by solution method. Long chain amines are known to facilitate the growth of nano structures in one dimension[3]. Z. Zhang et al[4] have synthesised shape controlled ZnO nanocrystals. They have reported ZnO nanowires, nanorods, bullets and triangular nanocrystals by tuning the molar ratio between amine and zinc acetate precursor. The results suggest that amine can play dual role as both the attacking agent and capping agent in this methodology. This process involves long reaction times. Therefore, it is a challenge to develop a facile, mild and rapid synthesis method to prepare ZnO nanostructures. Microwave heating techniques are known to bring about certain reactions at faster rates compared to the conventional heating techniques [5]. Here we report the synthesis of nanosize ZnO using kitchen microwave oven. Amines of different chain lengths were used, like hexyl amine (ZnO-H), octylamine (ZnO-O) and dodecyl amine (ZnO-D) to bring about various lengths of nano ZnO.

2. Experimental

2.1 Synthesis of quasi one dimensional ZnO using different amines

6.6 ml of hexylamine, octylamine or dodecylamine is taken in a round bottom flask with a known weight of zinc acetate (2.2 g) and refluxed in a rotary evaporator at 90° C for 2 hours. The solution of the respective amine is then taken in a beaker, covered with a watch glass and irradiated in a kitchen microwave at 850 watts for 3 minutes. A milky white solution is obtained in each case. This is then centrifuged and thoroughly washed

with Millipore water several times followed by acetone. The sample is then dried in a hot air oven around 50ºC to get ZnO-H, ZnO-O and ZnO-D respectively.

2.2 Characterization

The compounds are well characterized using powder XRD, UV-visible spectroscopy, IR spectroscopy, SEM and TEM. Powder XRD patterns of the prepared sample were taken in Philips XRD ('X' PERT PRO X-ray diffractometer) using Cu K_α radiation (λ = 1.5418 A^o). ZnO powder was well dispersed in distilled water by sonication and UV-visible absorption spectra were recorded with Shimadzu UV-visible spectrometer (Model 2100). Suitable thin pellets of ZnO with pure KBr as dilutent was made and IR absorption spectra were obtained using FT-IR SPECTRUM 1000 PERKIN ELMER SPECTROMETER. ZnO samples were smeared on to a carbon tape and mounted on metal stubs and SEM images were obtained using JEOL JSM 840A scanning electron microscope. Samples were taken on a copper grid and TEM and HREM pictures were obtained in Technai F30.

3. Results and Discussion

3.1 Characterization

Zinc acetate was treated with different amines in a kitchen microwave oven for maximum of 3 minutes to obtain high purity ZnO of different morphologies. The powder XRD patterns for the as-synthesized ZnO nanocrystals prepared from hexylamine, octylamine and dodecylamine respectively are depicted in Fig.1A a, b, c. The patterns show high crystallinity of the samples. All peaks can be indexed to the hexagonal wurtzite structure of ZnO and matches with the values in the database (JCPDS, 36-1451).

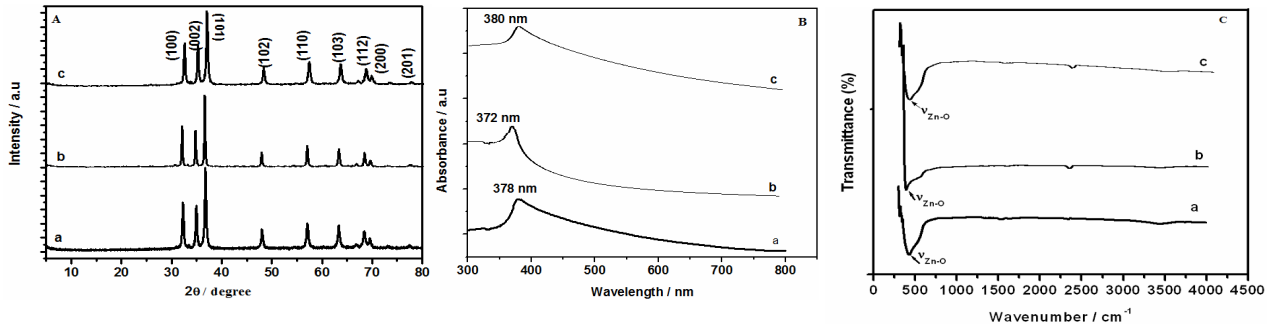

Fig. 1(A) Powder XRD Patterns and (B) UV-visible spectra (C) FTIR spectra of (a) ZnO-H, (b) ZnO-O and (c) ZnO-D.

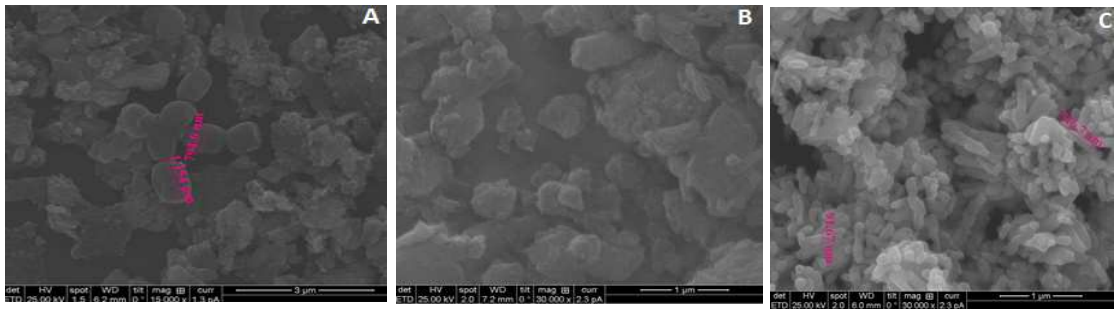

Fig. 2. SEM images of (A) ZnO-H, (B) ZnO-O and (C) ZnO

The UV-visible spectra (Fig. 1B a, b, c) show the absorption edge at 378 nm (3.28 eV), 372 nm (3.33 eV) and 380 nm (3.26 eV) for ZnO-H, ZnO-O and ZnO-D, respectively. ZnO-O shows the highest blue shift. The infrared absorption spectrum of the samples, ZnO-H, ZnO-O and ZnO-D recorded in the region from 300 to 4000 cm^{-1} and is shown in the Fig. 1C a, b and c respectively. The band around 420 cm^{-1} corresponds to Zn-O stretching frequency and absence of any other band indicates the high purity of the samples synthesized. Surface morphology of ZnO-H, ZnO-O and ZnO-D reveals variation in crystal sizes as depicted in the SEM pictures Fig. 2 (A, B, C) respectively. The morphology and structure of ZnO-H, ZnO-O and ZnO-D at different magnifications were further characterized by TEM and are depicted in Fig. 3 (A, B; D, E; F and G) respectively.

The Zn-O shows a well defined larger needle like structure, compared to ZnO-H and ZnO-D. Fig. 3 (C, F and I) shows the corresponding SAED patterns and all the diffraction patterns could be easily indexed to the hexagonal structure, which further matches with the powder XRD pattern (Fig. 1A).

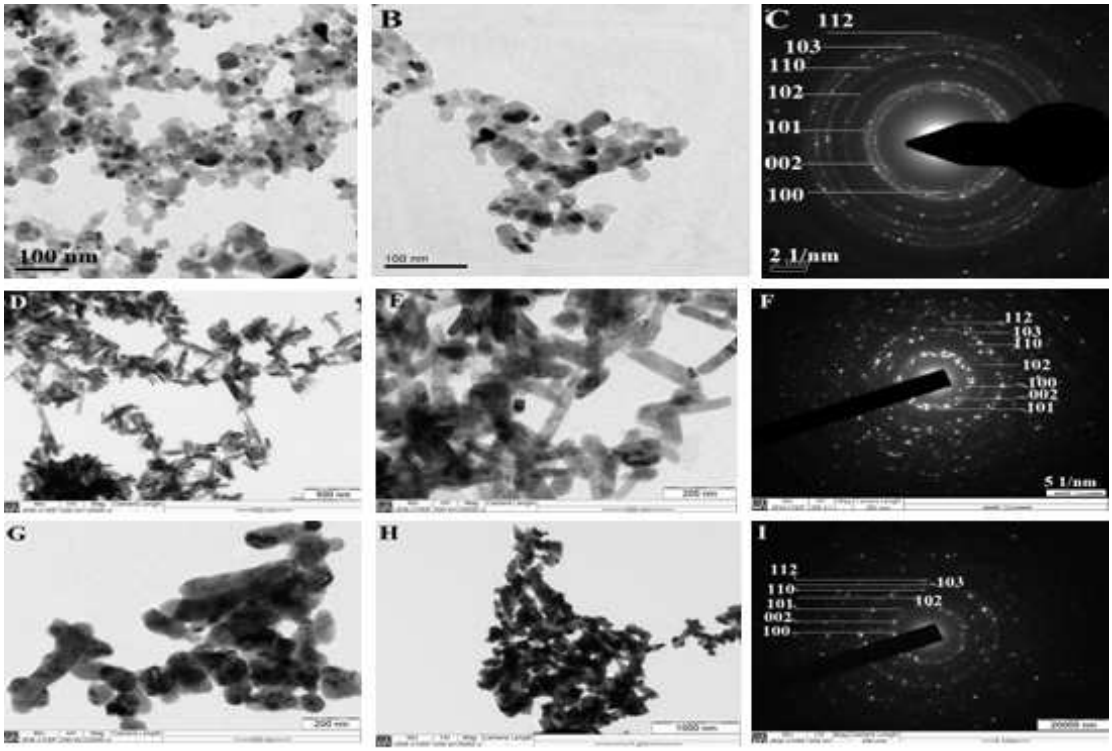

Fig. 3. (A),(B) are TEM images and (C) SAED pattern of ZnO-H; (D), (E) are TEM images and (F) SAED pattern of ZnO-O; (G),(H) are TEM images and (I) SAED pattern of ZnO-D.

Conclusion

The method described here is clean, simple and fast. Zinc oxide of various morphologies using amines of different chain lengths were obtained by micro wave heating.

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