A Superficial Solid-State Heating Method for the Synthesis of Zinc- Oxide Nanoparticles in Polymer Matrix

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Abstract - In this study, we report the simple and cost-effective in-situ synthesis of zinc oxide (ZnO) nanoparticles in polymer matrix (PPS). The polymer -polyphenelene sulphide (PPS) plays twofold role in the preparation of ensuing nanoparticles like -a) a chalcogen source and b) a stabilizing matrix. We have studied the effect of molar ratio (1:1) on the reactants- zinc nitrate and PPS. The synthesized nanocomposite were characterised by various physico-chemical techniques as X-ray Diffractometry (XRD) , SEM and UV-Visible spectroscopy. The prima - facie evidence for both type of ratio reveals subsequent entrapment of zinc oxide nanoparticles.

Keywords - ZnO; solid-state; nanoparticles; XRD; PPS.

1. INTRODUCTION

Recently, much attention has been focused on the development of polymer/inorganic nanohybrid materials [1-3]. Inorganic semiconductors like ZnO, TiO_2, MnO_2, and ZrO_2 have been widely examined as hybrids with polymers having synergetic or complementary properties and activities for the fabrication of a array of devices. Among these semiconductors, ZnO has shows potential applications in electrical engineering, catalysis, ultraviolet absorption, photodegradation of microorganisms, and optical and optoelectronic devices [4-8]. Even if ZnO demonstrate many advantages, there are still numerous disadvantages, for example the lack of visible light response, low quantum yield, and lower photocatalytic activity. Furthermore, it is significant to shift the photo activation region of ZnO particles toward visible wavelengths. Prior studies verified that conducting polymers incorporated with ZnO could exhibit rational catalytic activity under light illumination [9-12], and the delocalized conjugated structures of conductive polymers have been verified to arouse a rapid photo induced charge separation and decrease the charge recombination rate in electron transfer processes [13,14]. Nonetheless, ZnO is an amphoteric oxide, and it can react with acid or base to form a water-soluble salt. Consequently, a flourishing inclusion of ZnO into a conducting polymer matrix is the major research topic. Currently, there are numerous reports on the preparation methods of conducting polymer/ZnO composites [15-17], and the methods are mainly electrochemical polymerization and mechanical mixing. On the other hand to the finest of our knowledge, synthesis of nanocomposite/1-D nanostructure using polymer as inherent chalcogen source has not been sincerely attempted [18,19]. In this framework, vide this communication, we projected into the synthesis of addition, PPS with its exceptional mechanical and chemical properties can be processed in any desired form, which could be important aspects from the perspective of nanocomposite based fabrication.

2. MATERIALS AND METHOD

Polyphenelene Sulphide(PPS) with number average molecular weight of 10,000, zinc nitrate andacetone were obtained from Aldrich (99% purity)and were used as received. Zinc nitrate salt were used as Zn source, in both the sets of experiments. The melting temperature (Tm) of PPS is 285 ºC and its thermal decomposition starts at ~ 450 ºC. Therefore, the reaction temperature 285 ºC was chosen for the synthesis of ZnO in PPS matrix as at the melting temperature, sulphur will be loosely bound and some loosely bound sulphur may react with zinc to form ZnO.In-situ synthesis of zinc oxide in polymer matrix via proposed solid state root was carried out with reactants (zinc nitrate :PPS) using 1:1 ratio. For such reactions , the two reactants were admixed in stated molar ratio in a beaker and stirred for half an hour and then this mixture was transferred in an agate pestle- mortar using acetone. The resultant mixture, after

drying at room temperature was subjected to heating at 285 ºC(melting temperature of PPS) in an alumina crucible for 5 hours under an ambient atmosphere. Subsequently, it was naturally cooled down to room temperature. The obtained products with 1:1 molar ratio were of white coloured fine powder sample.

3.THEORY

The choice of the polymers is usually guided mainly by their mechanical, thermal, electrical, optical and magnetic behaviors. The polymer in many cases can also allow easier shaping and better processing of the nanomaterial. Hence we have selected such a polymer which play important role of stabilization. The inorganic salt zinc nitrate provide the metal which get into the polymer matrix . This method suggest superficial generation of ZnO merely heating the precursors at constant temperature of PPS. Also product which we obtained is in sufficient amount as we allowed for reaction. Therefore this method is cost effective.

4. RESULTS AND DISCUSSION

The X-Ray powder Diffraction pattern of the synthesized sample were recorded with Rigaku Miniflex X-ray Diffractometer using Ni filtered CuKα radiation. The external morphology and particle size of the sample were characterized by Scanning Electron Microscopy (SEM: Hitachi S 4800). For SEM analysis, the sample were prepared by (dispersing the obtained powder in methanol followed by ultrasonication treatment for 10 min) mounting a drop of nanoparticle solution on an aluminium stub allow it dry in air. The XRD pattern of the synthesized ZnO nanoparticles is shown in figure-1, corresponding to 1:1 molar ratio of $ZnNO_3$ with PPS heated at 285 ºC with constant reactant time of five hours. As shown in the fig 1., the peaks at 2θ =31.766, 34.463, 36.259, 47.545, 56.600, 62.841, 66.370, 67.990, 69.141, 72.527, 76.974 can be readily indexed as Hexagonal ZnO structure in the agreement with the reported JCPDS Card No. 36-1451. The strong and broadened peak at 2θ =36.259 indicates that the material has good crystallinity and small size. Also peak at 101 is much stronger than the other diffraction peaks. The key particle size has been calculated from FWHM values of strongest peak for 1:1 molar ratio is in the range of 27nm for sample corresponding to zinc nitrate with PPS. The FESEM photomicrographs of sample product corresponding to 1:1 molar ratio are resented in Fig 2(a,b).

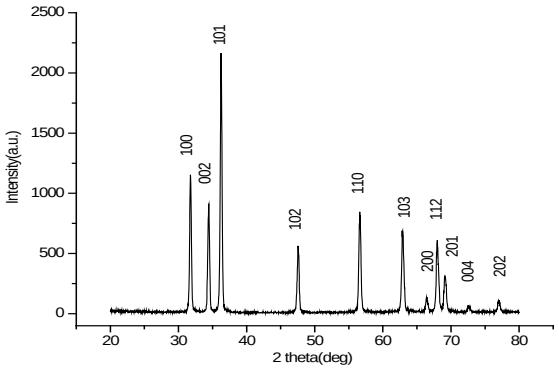

Fig. 1. X-ray diffractogram correspond to heated admixture of PPS and zinc nitrate in molar ratio of 1:1.

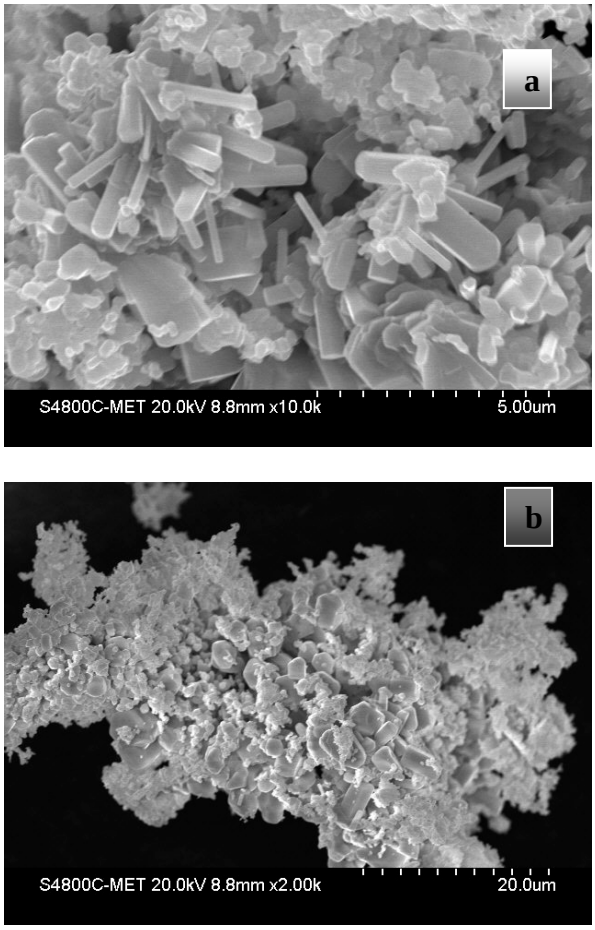

Fig. 2. FESEM images (a) and (b) correspond to heated admixture of PPS and zinc nitrate in molar ratio of 1:1

The hexagonal structure like morphological feature (of size 5-20μm) are observed in Fig 2(a). Such features appeared to be characteristics of ZnO nanoparticles indexed by X-ray

diffraction data. Also flower like morphology appeared in Fig 2(b) photomicrogaphs which can be attributed with ZnO nanoparticles in polymer matrix. The chunk like morphologcal features in fig 2(b) appeared to be characteristics of PPS.

5.CONCLUSIONS

We have demonstrated superficial solid-state method for the synthesis of ZnO nanoparticle in polymer matrix using metal salt $ZnNO_3$ with PPS as precursors. The strong diffraction peaks are observed, confirming the formation of ZnO nanoparticles which arestrongly supported by FESEM images. Such ZnO nanoparticles may be useful in electrical engineering, catalysis, ultraviolet absorption.

7. FUTURE SCOPES

This research demonstrated that we have future scope to study various kind of reactions with different inorganic salt and PPS

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