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SYNTHESIS AND CHARACTERIZATION OF ZnO NANOPARTICLES AND 50% ZnO-BENTONITE NANOCOMPOSITE

SWATI PATIL* and S. J. RAUT

Department of Chemical Engineering, Bharati Vidyapeeth University College of Engineering, PUNE – 43 (M.S.) INDIA

ABSTRACT

ZnO nanoparticles and 50% ZnO-Bentonite nanocomposite have been prepared by a precipitation method using zinc nitrate, bentonite clay and NaOH as precursors. X-ray diffractometry (XRD), Scanning electron microscopy (SEM) and energy dispersive X-ray analysis (EDX) were used to characterize the structural and the chemical features of the nanoparticles. XRD pattern of prepared ZnO were compared with commercial ZnO.

Key words: Pure ZnO, 50% ZnO-Bentonite, Nanoparticles, X-ray diffraction, Scanning electron microscopy, Energy dispersive X-ray analysis.

INTRODUCTION

Over the years, a large number of semiconductors have been used as photocatalysts[1]. As the semiconductor particles exhibit size-dependant properties like scaling of the energy gap and corresponding change in the optical properties, they are considered as the front runners in the technologically important materials. The most commonly studied photocatalysts are TiO_2 and ZnO[2]. As a semiconductor oxide, TiO_2 has been investigated extensively since 1980s and found as a photocatalyst due to its abundant availability and chemical stability[3]. However, widespread use of TiO_2 is uneconomical for large scale water treatment operations. It has become an imperative to find a suitable alternative. Some studies have confirmed that ZnO can also be used as a very efficient photocatalyst[4,5]. ZnO is of a lower cost and exhibits higher photocatalytic efficiencies for the degradation of several organic pollutants than TiO_2 [6,7]. Therefore, ZnO has been found to be a suitable alternative to TiO_2.

*Author for correspondence; E-mail: swatty82@yahoo.com

Zinc oxide is attracting tremendous attention due to its interesting properties like wide direct band gap of 3.3 eV at room temperature and high excitation binding energy of 60 meV. It has potential applications in a variety of fields such as catalysis[8], gas sensors[9], field effect transistors[10], resonators[11], short-wave optics[12], optoelectronic devices and functional materials[13].

Several studies have shown that isolated semiconductor nanoparticles have several toxic effects and therefore may pose environmental risk. Decrease of environmental risks of nanoparticles can be achieved by their anchoring to a suitable solid clay matrix[14]. Bentonite clay, because of their low permeability, plays an important role, as physical barriers, for the isolation of metal-rich wastes. The fixing of the semiconductor nanoparticles on the surface of bentonite clay enables to obtain nanocomposites, which have photocatalytic properties after exposure of UV light. These properties are given by photodegradation processes on surface of nanoparticles (on which nanoparticles of semiconductor are securely anchored)[15]. Chemical formula for bentonite clay is $Al_2O_3.4SiO_2.H_2O$. It contain SiO_2 of 61.3% (wt. %) and Al_2O_3 of 19.8 % (wt. %). In this work ZnO nanoparticles and 50% ZnO-bentonite nanocomposite were prepared by precipitation method. Microscopic and spectroscopic methods have been used for product characterization.

EXPERIMENTAL

Material and methods

Reagents and chemicals

Zinc nitrate, NaOH and Bentonite clay used were purchased from Thomas Baker Company. Solutions were prepared by using Millipore water.

Preparation of ZnO nanoparticles

Precipitation method was used to synthesize ZnO nanoparticles. 0.05 moles of zinc nitrate were dissolved in 500 mL of water. 0.75 moles of NaOH were dissolved in 500 mL of water. Zinc nitrate and NaOH were used in the ratio of 1 : 15. 500 mL of NaOH was added dropwise to zinc nitrate solution with vigorous stirring at 70^{o}C. After complete addition of NaOH, the reaction was allowed to proceed for next two hrs. After completion of reaction, solution was allowed to settle for overnight and the supernatant was then discarded carefully. The remaining solution was centrifuged for 10 min. and supernatant was discarded. Thus obtained nanoparticles were washed three times using water. This was followed by distilled acetone and the nanoparticles were dried at 80^{o}C. Then they were calcinated at 300^{o}C for 3 hrs.

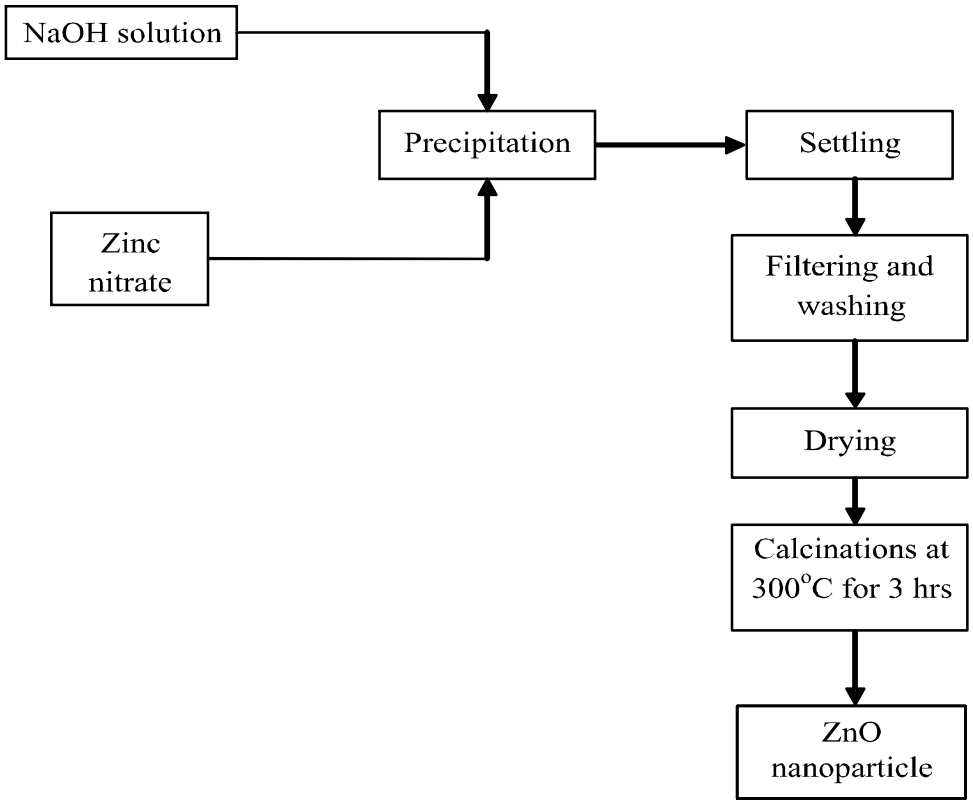

Fig. 1: Schematic of sample preparation using precipitation method

Chemical reactions involved in the formation of ZnO nanoparticles are as follows.

$$Zn(NO_3)_2 + 2\,NaOH \rightarrow Zn(OH)_2 + 2\,NaNO_3 \quad \ldots(1)$$

$$Zn(OH)_2 \rightarrow ZnO + H_2O \quad \ldots(2)$$

Preparation of the 50% ZnO-bentonite nanocomposite

In the preparation of ZnO nanoparticles by precipitation method, 2 g yield was obtained, so 50% ZnO-Bentonite nanocomposite were prepared by in situ deposition of ZnO on bentonite clay. 0.05 moles of zinc nitrate were dissolved in 500 mL of water. 1 : 15 molar ratio of zinc nitrate and NaOH was used. 0.375 moles of NaOH were dissolved in 250 mL of water. 2 g of bentonite clay was added in zinc nitrate solution and stirred for half an hour. 500 mL of NaOH was then added dropwise to the above solution with vigorous stirring at 70°C. After complete addition of NaOH, the reaction was allowed to proceed for next two hrs. After completion of reaction, solution was allowed to settle for overnight and the supernatant was then discarded carefully. The remaining solution was centrifuged for 10 min.

and supernatant was discarded. Then it was washed three times using water. This was followed by distilled acetone and the nanoparticles were dried at 80^{o}C. These were calcinated at 300^{o}C for 3 hrs.

Characterization

The composition of the prepared ZnO nanoparticles and 50% ZnO-Bentonite nanocomposite were analyzed by X-ray diffraction (XRD). The patterns were recorded in the 2θ range of 10-70^{0}. The morphology and dimensions of ZnO nanoparticles and 50% ZnO-Bentonite nanocomposite were observed by scanning electron microscopy (SEM, JEOL JSM-5600LV, 20 KV) and energy dispersive X-ray analysis (EDX).

RESULTS AND DISCUSSION

Characterization of the ZnO nanoparticles

The XRD patterns

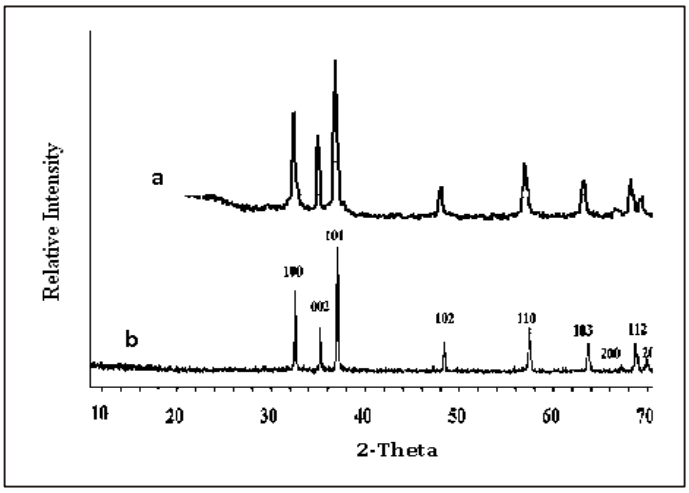

Fig. 2: The XRD pattern (a) prepared ZnO and (b) commercial ZnO

The XRD patterns of the prepared ZnO nanoparticles and commercial ZnO are shown in Fig. 2. No significant difference could be observed between the XRD patterns of the prepared ZnO nanoparticles and the commercial ZnO. All the diffraction peaks of the prepared ZnO nanoparticles were matched well with these peaks of the hexagonal wurtzite structure of ZnO (JCPDS, card no. 36-1451). In addition, no any other impure diffraction peaks were detected from the XRD patterns, which indicated that the prepared ZnO is pure ZnO.

The SEM with EDX analysis

The morphology of the prepared ZnO powder was investigated by scanning electron microscopy (SEM). As shown in Fig. 3, the average size of ZnO particles was estimated to be around 60-70 nm. The SEM photograph shows that the powder was homogeneous and agglomerated.

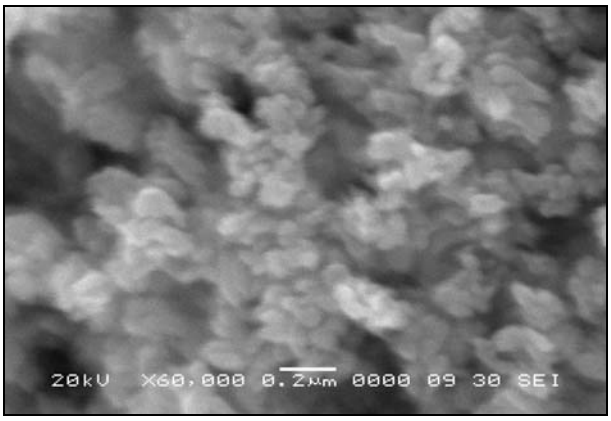

Fig. 3: SEM image of ZnO nanoparticles

Fig. 4 shows the EDX spectrum of ZnO nanoparticles. EDX spectrum shows four peaks which are identified as zinc and oxygen. Hence, it can be seen that pure ZnO nanoparticles can be prepared by precipitation method.

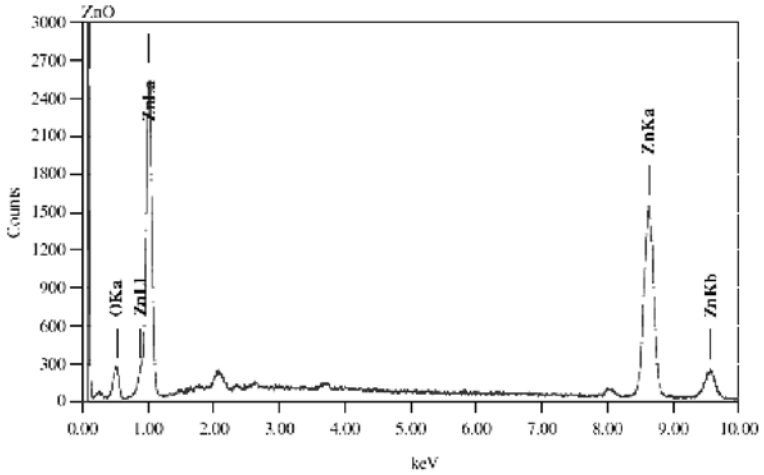

Fig. 4: EDX analysis of ZnO nanoparticles

Characterization of the 50% ZnO-Benzonite nanocomposite

The XRD pattern

The XRD patterns of the prepared ZnO nanoparticles and 50% ZnO-Bentonite nanocomposite are shown in Fig. 5. This indicates that 50% ZnO-Bentonite nanocomposite was formed by precipitation method.

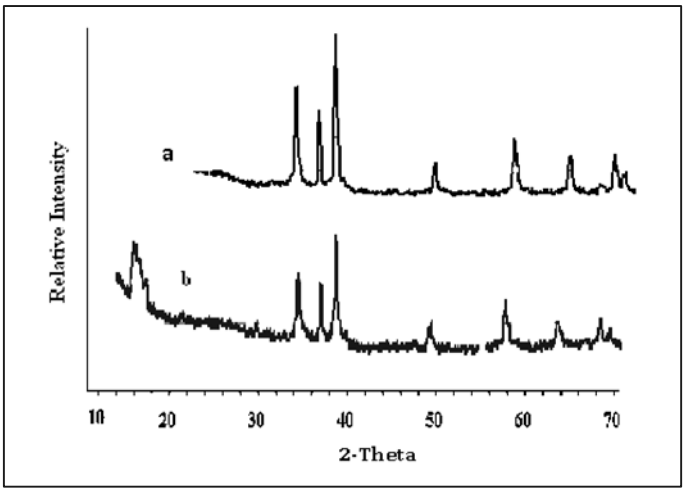

Fig. 5: XRD pattern for (a) ZnO nanoparticles and (b) 50% ZnO-Bentonite nanocomposite

The SEM with EDX analysis

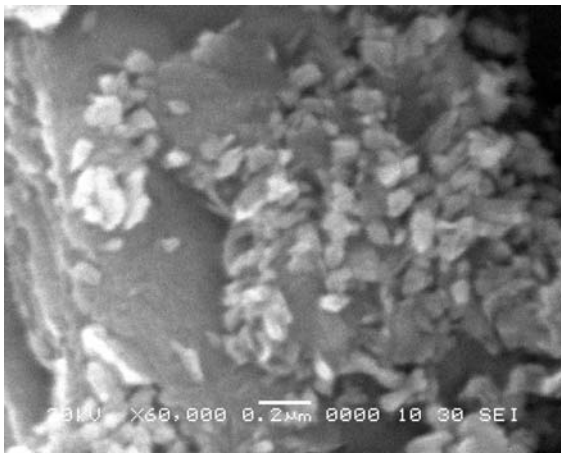

Fig. 6: SEM image of 50% ZnO-Bentonite nanocomposite

The morphology of the prepared 50% ZnO-Bentonite nanocomposite powder was investigated by scanning electron microscopy (SEM). As shown in Fig. 3, the average size of composite was estimated to be around 60-70 nm. The SEM photograph shows that the powder was homogeneous and agglomerated.

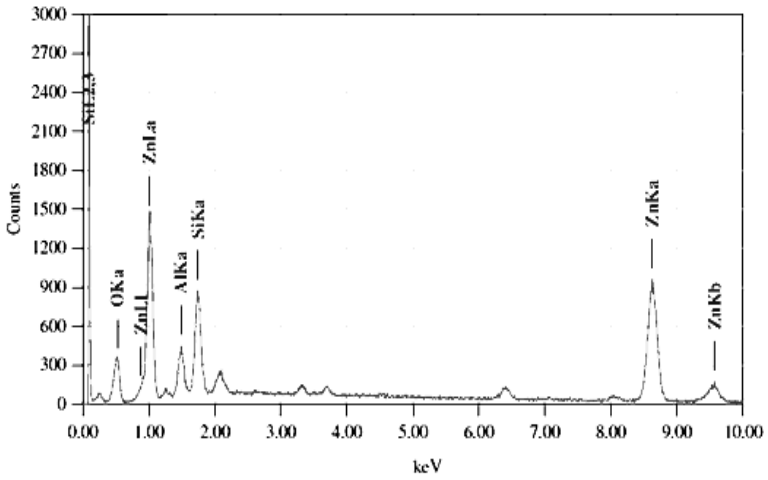

Fig. 7: EDX analysis of 50% ZnO-Bentonite nanocomposite

Fig. 7 shows the EDX spectrum of the 50% ZnO-Bentonite nanocomposite. EDX spectrum shows seven peaks which are identified as zinc, oxygen, silicon and aluminium. Hence, it can be seen that the 50% ZnO-Bentonite composite can be prepared by precipitation method.

CONCLUSION

Nano-sized pure ZnO particles and ZnO-Bentonite composite were successfully synthesized via direct precipitation method. XRD, SEM and EDX analysis confirmed the formation of nanoparticles of pure ZnO and ZnO-Bentonite composite. The XRD patterns for pure ZnO showed only the presence of pure wurtzite crystal structure in the sample and SEM analysis showed that average particle size of nanoparticles synthesized is about 60-70 nm. ZnO-Bentonite composite also prepared successfully with 50% ZnO loading.

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