Effect of reducing agents on the structure of zinc oxide under microwave irradiation

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Abstract Zinc oxide was synthesized from zinc sulphate using different reducing agents under microwave irradiation. The influence of sodium borohydride, hydrazine hydrate and urea on the shape and size of the products were investigated by scanning electron microscopy (SEM) and X-ray diffraction (XRD). SEM showed the nano-sized spherical and rectangular shaped structures in case of sodium borohydride and hydrazine hydrate, whereas micro-sized hexagonal structures were formed in case of urea under the same irradiation power. The reducing agents played an important role in forming the various structures. Thus different shapes and size of structures were produced by only varying the reducing agent, which had wide applications in various fields.

Keywords Zinc oxide · Microwave · Sodium borohydride · Hydrazine hydrate · Urea

1 Introduction

Oxides of transition metal have become one of the most interesting focus in many areas, such as chemistry, physics and material sciences [1]. Among which, ZnO has wide applications in the areas such as solar cells [2–4], catalysis [5, 6], sensors [7], laser diodes [8] and varistors [5, 9]. Various techniques had been employed for the synthesis of the nano/micro structured ZnO such as sol-gel process [10], homogeneous precipitation [5], thermal decomposition [11], microwave heating [12], conventional hydrothermal

[13–15], etc. Compared with the conventional heating methods, the microwave heating has many advantages of developing small particles of metal oxides with nano or micro sizes and better purity in short time [16–19]. In this paper, microwave heating has been utilized for the preparation of the zinc oxide in the presence of different reducing agents such as sodium borohydride, hydrazine hydrate and urea. The effect of the reducing agents on the size and shape of the formed products has been studied by scanning electron microscopy (SEM) and X-ray diffraction (XRD). It was found that different structure was obtained using the different reducing agents under the same starting material, ratio and irradiation power.

2 Experimental

2.1 Synthesis of zinc oxide from zinc sulphate and sodium borohydride

The zinc oxide was prepared by stirring 1:2 ratio aqueous solutions of zinc sulphate and sodium borohydride in at 60–80 °C, which produced a clear solution. Then it was heated in a microwave oven at 360 W, which produced a white precipitate after 2–3 min. The precipitate was centrifuged, washed with distilled water and crystallized with methanol. Finally the precipitate was kept for drying at 100 °C for 3–4 h.

2.2 Synthesis of zinc oxide from zinc sulphate and hydrazine hydrate

The zinc oxide was prepared by stirring 1:2 ratio aqueous solution of zinc sulphate and hydrazine hydrate at 60–80 °C. After mixing the solutions, a slight amount of

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Fig. 1 SEM of the spherical zinc oxide with sodium borohydride

white precipitate was observed. Then it was heated in microwave oven under the same irradiation power, which produced thick precipitate in 5–6 min. The precipitate was centrifuged, washed with distilled water and crystallized with methanol. Finally it was kept for drying at 100 °C for 3–4 h.

2.3 Synthesis of zinc oxide from zinc sulphate and urea

Zinc sulphate and urea taking in the same 1:2 ratio was heated under the same conditions. After 10 min, a thick precipitate was obtained. Then it was centrifuged, washed, crystallized, and dried for 3–4 h.

3 Results and discussions

The preparation of ZnO was studied using zinc sulphate and three different types of reducing agents under microwave conditions at the same irradiation power of 360 W. Up to 180 W, the yield was found to be very poor in all the cases. The SEM micrographs of ZnO nanoparticles using different reducing agents were shown in Figs. 1–3.

Fig. 2 SEM of the rectangular shaped zinc oxide with hydrazine hydrate

Figure 1 showed that ZnO possessed the spherical shape when sodium borohydride was used. Figure 2 showed the rectangular shaped nanostructures of ZnO in the presence of hydrazine hydrate. Sodium borohydride was used for the first time for the synthesis of zinc oxide under the microwave irradiation. It was interesting to observe that urea as an organic base produced the micro-sized (see Fig. 3) hexagonal shape structures with zinc sulphate. It could be concluded that different shapes and sizes varying from micron to nano-size of ZnO could be obtained using the different reducing agents under the same starting material and irradiation power. Compared to other heating methods, microwave heating was easy to produce the pure products with good yields.

Figure 4 showed the very sharp peaks in XRD pattern of ZnO nanostructures with sodium borohydride. The XRD pattern indicated the presence of peaks at 2θ values of 31.65°, 34.35°, 36.15°, 47.45°, 56.4°, 62.75°, and 67.7° corresponds to (100), (002), (101), (102), (110), (103) and (200) planes of ZnO respectively. Similar peaks were also observed in the case of hydrazine hydrate (see Fig. 5). The

Fig. 3 SEM of the hexagonal zinc oxide with urea

Fig. 4 XRD of the spherical zinc oxide with sodium borohydride

strongest peak at $2\theta = 36.15^\circ$ belongs to the (101) plane of the products. In Fig. 6, the peaks were in the different position showing the formation of different structures in the case of urea, which further confirmed the formation of micro-sized hexagonal shapes.

4 Conclusions

Different structures of zinc oxide were successfully synthesized using the easily available domestic microwave

Fig. 5 XRD of the rectangular shaped zinc oxide with hydrazine hydrate

Fig. 6 XRD of the hexagonal zinc oxide with urea

oven. Compared to the other conventional heating methods, microwave heating was found to be economic and could produce good yields of products. The reducing agents play an important role during the synthesis process. The SEM confirmed the formation of the different structures of ZnO with the spherical, rectangular and hexagonal shapes. Thus different shapes and sized of micro- and nanomaterials can be prepared by varying these reducing agents, which will have a wide range of applications in various fields.

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