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Fast Microwave-Assisted Synthesis of Uniform Magnetic Nanoparticles

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In recent time, magnetic nanoparticles have become widely used for preparation of advanced magnetic materials and also for biomedical applications. Requirement for preparation of particles of suitable shape and size has appeared, hence, various methods have been developed. Here we present rapid and energy saving one-pot solvothermal synthesis using microwave pressurized system. This method allows tuning the size of the particles as well as their magnetic properties. Spherical Fe_3O_4 nanoparticles are obtained in 30 min; they are uniform with average dimensions of 200 nm and exhibit ferromagnetic behavior dependent on synthesis temperature.

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1. Introduction

In the past decades, the interest in synthesis of uniform magnetic particles in micro- or nanodimensions expanded due to their potential application in many fields. They can be applied in medicine for controlled drug delivery, hyperthermia or medical diagnostic for magnetic resonance imaging. These particles are also used as fillers in magnetic composites or in electromagnetic shielding materials. They are investigated as a key component of magnetorheological fluids, too. Moreover, external magnetic field can be used for preparation of oriented anisotropic structures from this particular system thus achieving novel properties [1–6]. Lots of methods have been introduced for preparation of magnetic particles, employing both dry and wet chemistry. The most popular among them have become simple one-pot solvothermal methods [7–10].

2. Experimental

In a standard experiment, 5 mmol of $FeCl_3{\cdot}6H_2O$ was dissolved in 60 mL of ethylene glycol, followed by the addition of nucleating agent (50 mmol of NH_4Ac, 25 mmol of $(NH_4)_2CO_3$ or 200 mmol of aqueous NH_3). This mixture was placed in a teflon reaction vessel (XP-1500 Plus), heated in pressurized CEM Mars 5 microwave system (CEM Corporation) to a required temperature (200, 210 or 220 °C) and maintained at this temperature for 30 min. After the reaction, the vessel was cooled to a room temperature and the as-obtained product was filtered off, washed with water and ethanol for several times and dried naturally on air. The structure of the final product was characterized by X-ray diffraction method (XRD; PANalytical X'Pert PRO). The particle size and shape was visualized by scanning electron microscopy (SEM; VEGA\\LMU, Tescan) and magnetic properties were identified by a vibrating sample magnetometer (VSM; VSM 7400, Lake Shore).

3. Results and discussion

Magnetic particles were obtained by a simple microwave-assisted solvothermal method in 30 min in nearly 100%. As can be seen from XRD pattern in Fig. 1, all the diffraction peaks are attributed to cubic Fe_3O_4, although we cannot distinguish whether it is magnetite or maghemite. Key factor for obtaining well crystallized structures are synthesis time and temperature. The required product can be obtained if the time of synthesis is about 30 min. Experiments with different synthesis temperatures showed that the crystallic structure of particles improves with the temperature increment. The typical SEM image of the product prepared with NH_4Ac at 220 °C in Fig. 2 shows that the prepared particles are spherical, uniform and their dimension are about 200 nm. Figure 3 gives magnetization curves of particles prepared with NH_4Ac at different temperatures and that these particles exhibit ferromagnetic behavior. A strong effect of the synthesis temperature is manifested: with increase of the temperature used for preparation of particles, saturation magnetization and coercivity of prepared material are significantly higher.

4. Conclusion

To conclude, in this work there is presented an efficient, rapid and facile one-pot solvothermal microwave-assisted

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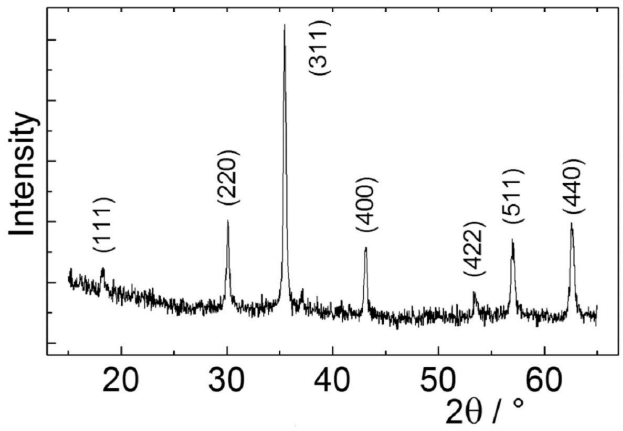

Fig. 1. XRD pattern of Fe_3O_4 particles prepared with NH_4Ac at 220 °C for 30 min.

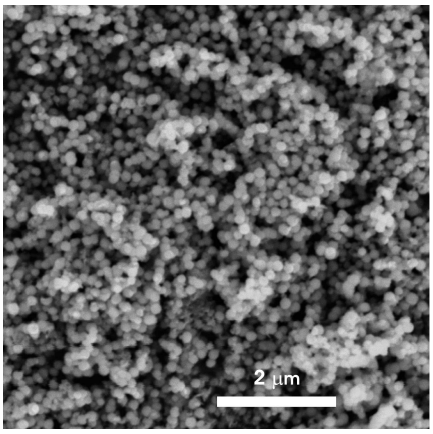

Fig. 2. SEM image of Fe_3O_4 particles prepared with NH_4Ac at 220 °C for 30 min.

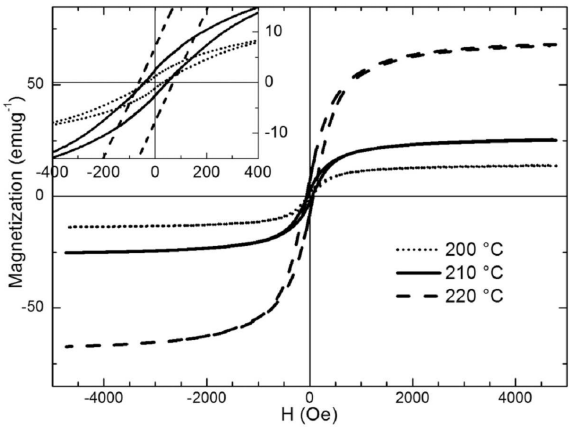

Fig. 3. Room temperature magnetization curves of Fe_3O_4 particles prepared with NH_4Ac at 200, 210 and 220 °C for 30 min. In the inset is given a detailed view of magnetization curves is given, shoving variation of coercivity while different temperatures are used within the synthesis.

synthesis of magnetic Fe_3O_4 nanoparticles by which they can be obtained in 30 min without requirement of further treatment such as calcination, which can cause disruption of particles. The as-obtained spherical particles are uniform with an average dimension of 200 nm and exhibit ferromagnetic behavior. The method allows particle size tuning and refinement of magnetic properties by simple changing the synthesis parameters, i.e. temperature or nucleating agent, which can be profitable in future medical application as well as in formation of advanced magnetic materials.

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