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PRODUCTION OF IRON-COBALT COMPOUND NANOPARTICLES USING REVERSE MICELLAR SYSTEM

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Abstract. Synthesis of nanoparticles using surfactant systems such as water-in-oil micro emulsion systems or reverse micellar systems brings perfect particle control over particle size. In this study, reverse micellar system was formed with anionic surfactant AOT in isooctane to produce $CoFe_2O_4$ with particle size ranging with 15 – 150 nm. In addition, effects of reaction time, concentration of $FeSO_4{\cdot}7H_2O$ and AOT on the nanoparticle formation were investigated. The molar concentration of $FeSO_4{\cdot}7H_2O$ was found to be an important parameter in control of nanoparticle size. A decrease in the concentration of $FeSO_4{\cdot}7H_2O$ resulted in larger $CoFe_2O_4$ particles.

1. INTRODUCTION

In the recent years, metal particles of nanometer dimensions have been investigated because of their present applications and perspectives in various fields of science, technology and pharmaceutical. For example, in the electronic industry, semiconductors resulted in the transition from the vacuum tubes to diodes and transistors and eventually to miniature chips. This process is limited by the capacity of information storage devices, which in most cases work on the magnetic recording principle. Increasing of the storage density requires more strict control over morphology of the magnetic material and strong reduction of its dimensions, down to the sizes of single domain [1-3].

A lot of methods provides manufacturing nanocrystalline objects from inorganic materials; micro-emulsion systems and reverse micelle systems are widely used here since it is no need for expensive devices, moreover, particle formation and their dimensions are easily controlled by these methods. Note that particle size distribution is critical for application of the materials in actual devices.

'Water-in-oil' micro-emulsions consist of nanometer sized water droplets that are dispersed in an oil medium and stabilized by surfactants. These reverse micellar systems are heterogeneous on a molecular scale, nevertheless they are thermodynamically stable. Reverse micellar systems are suitable reaction media for the synthesis of nanoparticles, because tiny droplets of water are encapsulated into reverse micelles. Water pools of these reverse micelles act as micro-reactors for performing simple reactions of synthesis, and the size of micro-crystals of the product is determined by the size of these pools. The size of the water pools is known to be controlled by the molar water/surfactant ratio in the system [2–4]. This method offers several advantages: it is a soft technology, which can be used to perform several chemical reactions. On the other hand, it does not require extremely high temperature and pressure conditions as well as any special equipment [4].

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Table 1. Solution compositions used in the experiments.

Experiments	*Concentration of* $FeSO_4 \cdot 7H_2O$(M)	*Concentration of* $CoSO_4 \cdot 7H_2O$ (M)	*Concentration of* AOT (M)	*Reaction time* (min)
1	0,92	0,11	0,50	90
2	0,92	0,11	0,50	120
3	1,44	0,11	0,50	120
4	0,92	0,11	0,65	90

The magnetic properties of the metallic nanoparticles were studied in [1-4], however the information on the effect of the operating conditions (water droplet size, surfactant concentration, *etc.*) on the size, shape and crystalline forms of particles prepared in micro-emulsions is unexhaustive. For this reason in this study, we have demonstrated the versatility of the reverse micelle method for the synthesis of cobalt-ferrite compounds. In addition, the effects of reaction time, AOT concentration in iso-octane, and $FeSO_4$ percentage in aqueous medium on the particle size were investigated.

2. MATERIAL AND METHODS

Reagents. Sodium diisooctylsulfoccinate (AOT) was obtained from Aldrich. $FeSO_4 \cdot 7H_2O$, $CoSO_4 \cdot 7H_2O$, NH_3, and chromatographically pure iso-octane and iso-propanol were supplied by Merck.

Synthesis of Nanosized Particles. In this study, reverse micelle systems consisted of iso-octane (the oil phase), AOT (the surfactant) and the aqueous phase, which contained reactants. Initially, two solutions (A and B) were prepared: solution A with metal salts and solution B with reducing agent NH_4OH, which were used to control of the product's morphology. As an example, to prepare $CoFe_2O_4$ nanoparticles with the average diameter of 34 nm we used the following reagents: solution A of 45 ml 0.50 M AOT in i-octane, 5 ml of water with 0.92 M $Fe_2SO_4 \cdot 7H_2O$ as a metal salt, and 0.11 M $CoSO_4 \cdot 7H_2O$; solution B consisted of 45 ml 0.50 M AOT in i-octane and 5 ml NH_4OH as a reducing agent. The list of the solution A and B compositions used in our experiments is given in Table 1. Then both solutions were mixed suddenly and stirred for 90-120 min in the orbital shaker. After that, equal volume of i-propanol was mixed with the solution in order to break down the reverse micelles.

Analysis. The dimension of the synthesized particles was measured by Zetasizers 3000 HSA, Malvern Instrument, in the organic media. Note that this analyzation technique prevents particle agglomeration, so it prevents the increase in the particle size.

3. RESULTS AND DISCUSSIONS

Effect of some parameters such as reaction time, concentration of $FeSO_4 \cdot 7H_2O$ and AOT were investigated. Table 2 presents the average particle size obtained in the series of experiments, see Table 1 for operating condition details.

Effect of the reaction time on the size of nanoparticles is presented in Fig. 1. One can see that the particle size increases with the reaction time. Reaction time is the most effective factor for increasing reverse micelles in the organic phase. As expected, with increasing of the reaction time, bigger diameters of $CoFe_2O_4$ particles are produced in the water droplets.

Fig. 2 illustrates the effect of $FeSO_4$ concentration on the particle size; it decreases with the increase of $FeSO_4$ percentage. Note that the production of reverse micelle in the inter-phase might be increased by increasing $FeSO_4$ percentage. In other words, the percent of particles may be increased, while the particle size will be decreased.

The effect of the AOT concentration on the particle size is shown in Fig. 3. As seen from this figure, the increase in the AOT concentration leads to

Table 2. Average size of $CoFe_2O_4$ particles.

Experiments	*Average particle size* (nm)
1	34
2	43
3	27
4	25

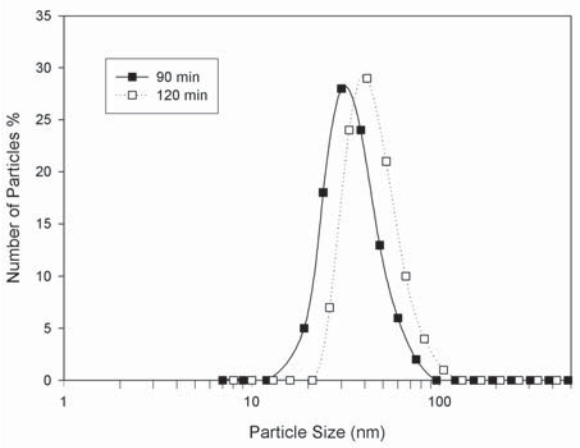

Fig. 1. The effect of the reaction time on the particle size.

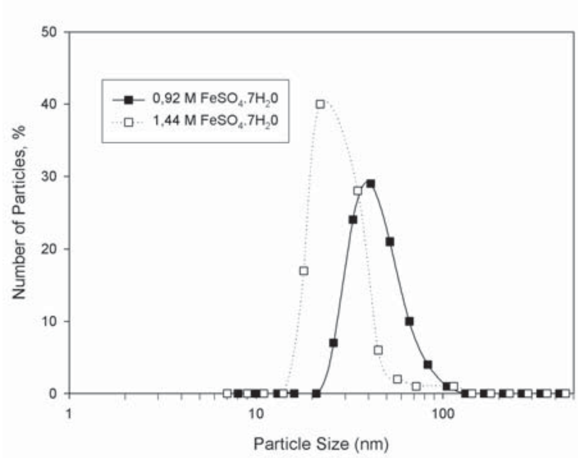

Fig. 2. The effect of the $FeSO_4 \cdot 7H_2O$ concentration on the particle size.

the decrease in the particle size; at the same time the number of reverse micelle increases enlarging the volume of the reaction media.

4. CONCLUSIONS

In this study, $CoFe_2O_4$ nanoparticles with the diameter from 15 to 150 nm were synthesized by reverse micelle system. The effects of the reaction time, $FeSO_4 \cdot 7H_2O$ concentration and AOT percentage on the particle size were investigated. It was shown that the particle size of $CoFe_2O_4$ increases with the reaction time and decreases with the increase in $FeSO_4$ and AOT concentrations. The percent of particles increases with the concentration of $FeSO_4$ and AOT.

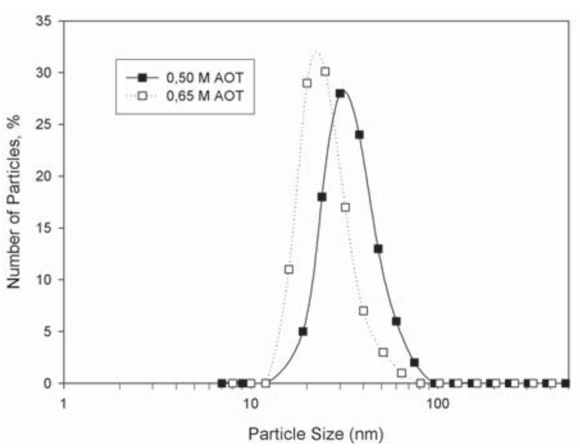

Fig. 3. The effect of the AOT concentration on the $CoFe_2O_4$ particle size.

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