Effect of Alkaline Media Concentration and Modification of Temperature on Magnetite Synthesis Method Using FeSO$_4$/NH$_4$OH

M. Tajabadi and M. E. Khosroshahi

Abstract—**Superparamagnetic particles find a great situation in a large number of science. Despite high number of research in the field of magnetic nanoparticle production, there are some drawbacks regarding this kind of materials. In this study in order to overcome some of these drawbacks, nano-sized magnetite fine powder was produced via coprecipitation method under N2 atmosphere by modification of two significant parameters, alkaline media concentration and temperature. Effect of named parameters on the particle size, size distribution was analyzed by transmission electron microscopy (TEM), and in order to evaluate magnetic behaviour of particles, vibrating sample magnetometry (VSM) was used. Crystalline phase of synthesized material were determined by means of X-ray diffraction spectroscopy (XRD). As authors know, there is not any evidence of evaluation of sample synthesized at room temperature for a short period of time and then post modification at higher temperature on particle size and its magnetic properties. Results show the particles synthesized at elevated temperature (70 ℃) and lower alkaline concentration (0.9 M) have highest saturation magnetization, about 68 emu/gr at 70 ℃, compared with 63 emu/gr at 25 ℃ and smallest particle size. On the base of our experimental data we can introduce a modification way for copercipitation method which improves the magnetic properties with reduction of particle size at the same time.**

Index Terms—**Alkaline media concentration, magnetite, particle size reduction, temperature.**

I. INTRODUCTION

Over the past decades Iron Oxide nanoparticles (NPs) which possess extraordinary size- and morphology-dependent physical and chemical properties, have attracted world-wide research attention not only because of these unique properties but also for their biocompatibility and remarkable magnetic properties [1]-[3]. Iron oxides are also present in living organisms (*e.g.* plants, bacteria, molluscs, birds and humans). For the regulation of iron concentration in organism, Fe(II) and Fe(III) species (ferritin) take part in various biomineralization processes, also active center of haemoglobin contains Fe(II) complexes [4].

Among the nanocrystals of different iron oxide phases, magnetite nanoparticles find unrivaled situation in the field

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of science. As magnetite nanoparticles have high magnetization saturation, can easily load with biomolecules and are biocompatible, this kind of material has extraordinary position among iron oxides[5]. Magnetite has inverse spinel structure and its unite cell structure represents with $(Fe^{3+})_A(Fe^{3+}Fe^{2+})_BO_4$ where A and B respectively stand for tetrahedral and octahedral sites coordinated with oxygen ions at face centered cubic array. The unit cell of magnetite contains 32 O^{2-} and 8 Fe^{3+} ions on the tetrahedral sites (A sites) and 16 Fe ions on the octahedral sites (B sites); 8 of which are Fe^{2+} and the other 8 are Fe^{3+}[3], [6]-[8].

Having high saturation magnetization (Ms) as well as small particle size are two determinative parameters in the application of these kind of nanoparticles [5]. Magnetite nanoparticles find a conceivable application in scientific fields such as magnetic memories, optics, production of pigment, electronics, activators, ferrofluids, and biomedical [5], [9].

Various synthetic methods have been used to produce magnetite nanoparticles including coprecipitation, microemulsion, laser pyrolysis, and hydrothermal synthesis [8]-[11]. The major challenges in synthesis procedure of nanoparticle arise from the point that most production methods contain environment-unfriendly ingredients and because of usage of different kind of raw materials these methods are complicated; these factors result in an increase of production cost as well as environmental impact. Among magnetite synthesis methods, coprecipitation technique in water system represents a beneficial method, as the synthesis process is simple and the environmental impact is relatively low. In an alkaline medium such as ammonium or NaOH aqueous solution, coprecipitation of Fe^{2+} and Fe^{3+} ions is done. In this procedure hydroxides of ferrous and ferric ions, ferrous hydroxide (Fe(OH)$_2$) and goethite (α-FeOOH), are coprecipitated as precursors in an alkaline solution. By setting molar ratio of ferrous/ferric equal to 0.5, the solid phase reaction between hydroxides results in formation of Fe$_3$O$_4$. As this method is one of the most efficient ones and doesn't use any surfactant, many researcher attempts to optimize this magnetite production strategy [5], [12].

Some problems regarding coprecipitation method are its uncontrollability of particle size and size distribution and sometimes simultaneous presence of different iron oxide phases such as magnetite and maghemite [13]-[15]. There are some articles on investigation of parameters affecting the production of magnetic nanoparticles including pH, alkaline species [15]-[16], reaction temperature [5], [13], [17], kind of iron salts and concentration of ingredients. In order to improve the properties of magnetite nanoparticles which are

synthesized via copercipitation method, we evaluate the impact of alkaline medium concentration and temperature on substantial properties. To the best of our knowledge, there is no report concerning the effect of extra stirring time (30 minute in our case) at elevated temperature on the synthesis method and properties of resultant materials.

II. EXPERIMENTAL

A. Synthesis of Magnetite Nanoparticles

Solutions of 0.012 M ferrous chloride hexahydrate ($FeCl_3.6H_2O$, 99%, Merck) and 0.006 M ferric sulfate heptahydrate ($FeSO_4.7H_2O$, 99%, Merck) used as iron source were prepared by dissolution of proper amount of material in double distilled water. After the ferric and ferrous solution were mixed together, the mixture was then deoxygenated by bubbling N_2 gas and sonicated for 30 minutes to prevent from oxygenation. Ammonia solutions with 0.9-2.1 M concentration was used as alkaline source and vigorously stirred under N_2 bubbling at room temperature. The mixture of ferric and ferrous solution was dropwisely added to the stirring ammonia solution, the color of reaction mixture immediately turned to black. In this experiment, group 1 and 2 are defined as the groups containing the different alkaline media concentration at two different temperatures i.e. 25 ℃ and 70 ℃ respectively. The reaction temperature was kept constant at 25 ℃ for 1 hour then the mixture of group 1 purified. After an hour, having been transferred group 2 to 70 ℃ water bath, the mixture was strongly stirred 30 minutes. For both group 1 and 2, the black precipitation were purified using magnetic separation five times and sedimented by centrifugation. The resultant material was dried by freeze dryer for 24 hours.

B. Characterization

Crystalline phase of magnetite nanoparticles was studied using X ray diffraction using Cu Kα radiation (XRD, λ= 1.5406 Å, FK60-40 X-ray diffractometer). Saturation magnetization of samples was measured by means of vibrating sample magnetometer (VSM-PAR 155) at 300 K under magnetic field up to 8 kOe. Particle size and morphology of these nanoparticles were determined by transmission electron microscopy (TEM, Philips CM-200-FEG microscope, 120 KV).

III. RESULT AND DISCUSSION

A. XRD Analysis

XRD results obviously confirmed the formation of magnetite phase of iron oxide with inverse spinel structure (JCPDS file no.19-0629). Thermodynamic modeling represents in oxygen free environment, *pH* 7.5-14 and $Fe^{2+}:Fe^{3+}$ ratio equal to 1:2, precipitation of magnetite is completed as follow:

$$Fe^{2+} + Fe^{3+} + 8OH^- \rightarrow Fe_3O_4 \text{(black precipitation)} + 4H_2O \tag{1}$$
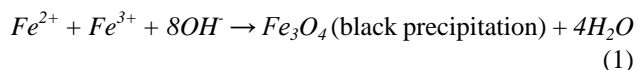
If this reaction takes place in oxygen containing media, the reaction condition will be ready to oxidation of Fe_3O_4. Oxidization of Fe^{2+} to Fe^{3+} ions ($Fe^{2+}+O_2 \rightarrow Fe^{3+}$) declines

the effective ratio to less than 0.5 and if Fe^{3+} ions is kept on elevated pH, goethite can be produced[18-19]:

$$Fe_3O_4 + 0.25 \, O_2 + 4.5 \, H_2O \rightarrow 3 \, Fe(OH)_3 \tag{2}$$
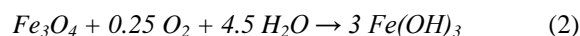
As usage of strong alkaline medium (such as *NaOH, KOH,* and *LiOH*) as a hydrolyzing agent can cause formation of nonmagnetic iron components [5], [12] in order to avoid this matter, NH₄OH was used as alkaline media in present study. Also, since in this study coprecipitation reaction was performed under N_2 atmosphere, the single magnetite phase without any interference of other iron oxide phase was obtained. In addition, the interplanar spacing of prepared sample was 2.534 which is close to the standard value of Fe_3O_4 (=2.532) compared with that of maghemite (=2.51).

The particle size of nanoparticles could be determined using Debay-Sherrer's equation (eq. 3) which measures the size of particles according to broadening of the most intense peak (311) in XRD graph (Fig. 1) of prepared materials [20].

$$D = \frac{K\lambda}{\beta \cos\theta} \tag{3}$$

where *D* is mean diameter of particles, β is full width at half maximum (FWHM), λ is wavelength of incident X-ray and the dimensionless shape factor, *K*, is equal to 0.9. Based on this equation, particle size of samples synthesized at 25 ℃ and 70 ℃ were determined 10.10nm and 9.98 nm, respectively.
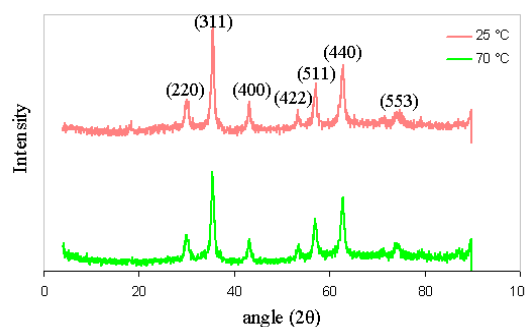

Fig. 1. XRD pattern of synthesized magnetite nanoparticles under A) 25°C B)

70°C

B. TEM Analysis

Morphology and mean particle size of prepared nanoparticles at 25 ℃ and 70 ℃ were determined by TEM (Fig. 2). Respect to different synthesis conditions, particles are quasi sphere and have sizes ranging from 9.5 to 16 nm. Polydispersity and surface aggregation of samples prepared at room temperature could be figured out from Fig. 2, this regards to small size, increased surface-to-volume ratio and thus augmented magnetic dipole-dipole interactions [21]; at synthesizing conditions of prolonged time and elevated temperature because of reduction of surface defects, although particles are smaller than those produced at room temperature, surface aggregation and polydispersity were reduced.

There is a critical size which define the superparamagnetic region, for calculation of this parameter one can use the following expression, Vp≈25kT/K; where k is Boltzmann constant, K denotes anisotropy constant ($Fe_3O_4 = 1.35 \times 10^4$

J/m^3) and T is the absolute temperature[22]. At room temperature, 300 K, the critical size is equal to 27 nm. The particles prepared in this study have sizes around 9.5 to 16nm which are below the critical size (27 nm), this matter ensure the superparamagnetic behavior of prepared nanoparticles.
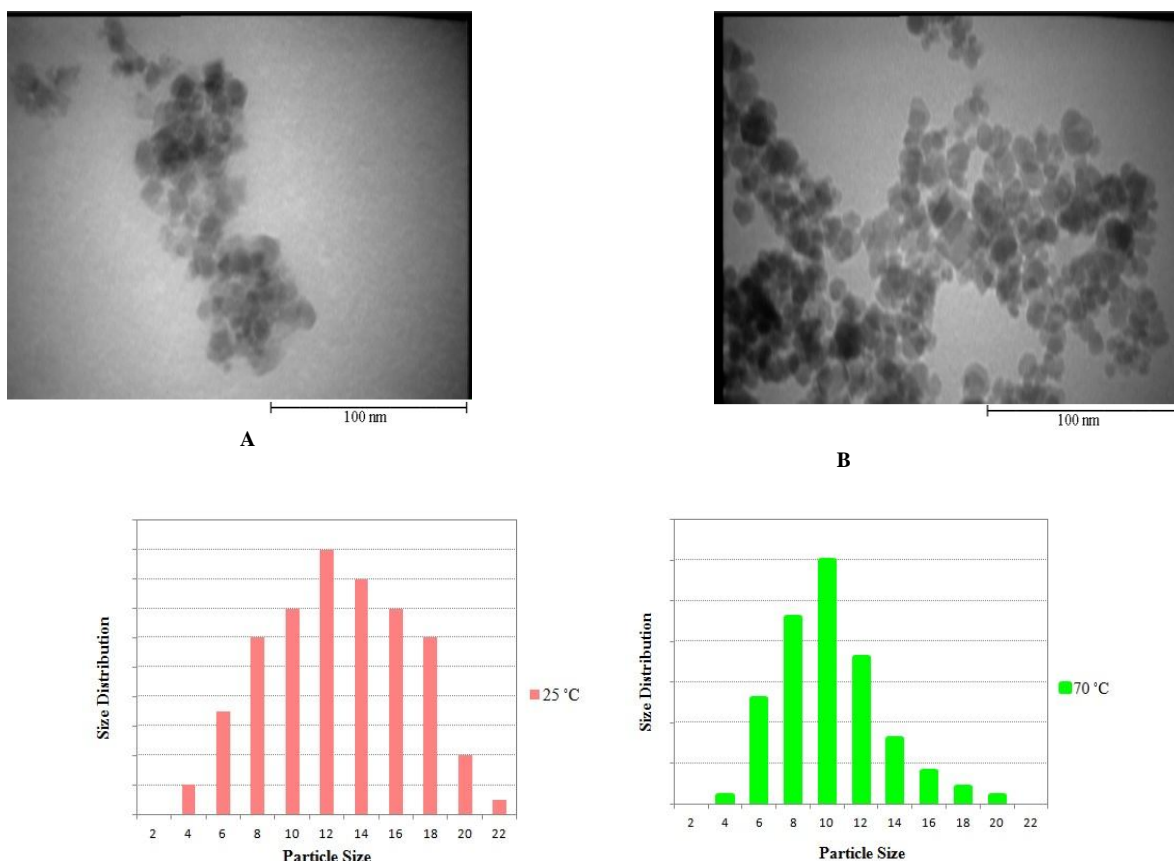

Fig. 2. TEM micrograph of samples prepared at A) 25 ℃ B) 70 ℃

Basic procedure affected the particle sizes and shapes principally depend on the balance of nucleation and growth rates. When ingredients of reaction are added together, the nucleation phenomena happen at supersaturation state. If all ingredients are used no particle growth is happened otherwise Ostwald ripening and aggregation of particles could organize the growth phenomena. Production procedure of nanoparticles contains many intermediate products which may strongly affect the nucleation and growth phenomena. After addition of alkaline species and iron salts together, some cations and anions like SO_4^{2-}, Cl^-, NH_4^+ and some intermediate products such as $Fe(OH)_2$, $Fe(OH)_3$, and $\alpha\text{-}FeOOH$ is produced. The release and usage rate of these materials determine the rate of nucleation and growth of magnetite nanoparticles. Once the magnetite nuclei are produced, ion-to-ion attachment, particle aggregation or Ostwald ripening (growth of larger particle at the expense of dissolving smaller one) cause the particle growth.[23]

As some separate articles recommended, in order to produce tiny nuclei, the initial temperature of this study set to 25 ℃ and after 1 hour group 1 was purified which resulted in particles with larger size (average diameter is about 14 nm). Depending on reaction time, the diffusion and growth process of samples could be completed [24]-[25]. At elevated temperature and prolonged time, the reaction condition set to be completed and samples with less crystal defects and smaller particle size (average diameter is about 10 nm) were produced. With increase of ingredient (alkaline media) concentration more materials are available on the growth phase and thus particles with higher diameter could be obtained (Fig. 3).
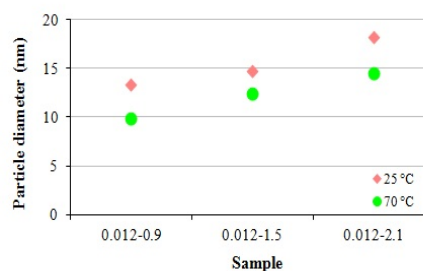

Fig. 3. Variation of particle size with alkaline media concentration and temperature measured by TEM
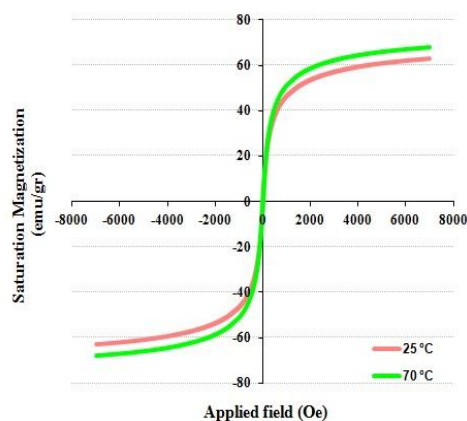

Fig. 4. VSM result of samples prepared at A) 25 ℃ B) 70 ℃

C. *Magnetic Measurements*

Magnetic properties of nanoparticles were determined by VSM at room temperature, results show that these materials have strong magnetic properties of 63.1emu/gr and 67.8emu/gr for samples synthesized at 25 ℃ and 70 ℃, respectively.

Bulk material of magnetite has higher saturation magnetization , the small particle size and breaking of a large number of exchange bonds for surface atoms cause this reduction [9, 26]. As figure 4 shows, complete reversibility of magnetization process confirms the superparamagnetic behaviour of prepared nanoparticles. Because of higher amount of alkalin media, with increase of alkaline media concentration, the probability of non-magnetite layer production (magnetically dead layer) increased which decrease the amount of saturation magnetization (see the Fig. 5.) revealing that the positive effect of alkaline media on saturation magnetization has a limit.
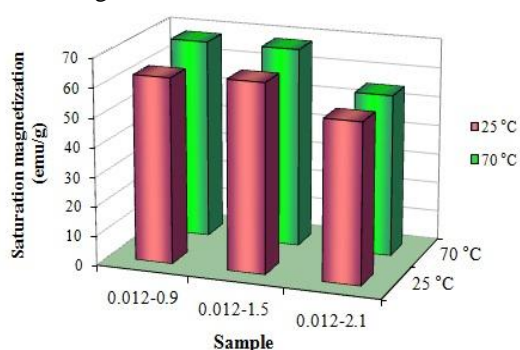

Fig. 5. Change of Ms as change of alkaline media concentration and temperature

Using Langevin's equation and magnetic experimental data, the average magnetic particle diameter were calculated[3]:

$$a_M^3 = \frac{18 K_B T}{\pi \mu_0 M_b M_s} (\frac{dM}{dH})_{H \to 0} \qquad (4)$$

where a_M is the magnetic particle diameter, k_B is Boltzmann's constant, μ_0 is the vacuum magnetic permeability, and M_b is magnetization of bulk magnetite. Figures 5 and 6 represent increase of magnetic properties and slight decrease of particle size with respect to elevated temperature. Owing to prolonged reaction time and elevated temperature, synthesis reaction could be completed [20] thus particle size was reduced and because of loss of surface defects, magnetic saturation was increased.
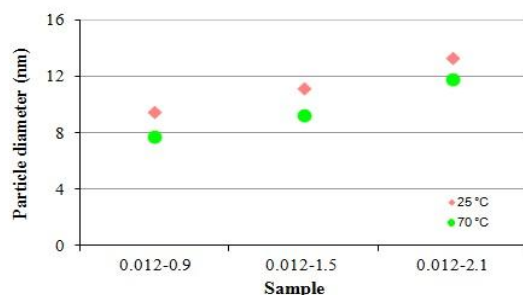

Fig. 6. Variation of particle size with alkaline media concentration and temperature measured by VSM

Nanoscale materials show great different properties which are highly size dependent. In magnetic materials, saturation magnetization, susceptibility, coercivity and other magnetic properties are tremendously affected by particle size. For materials with sizes greater than magnetic domains, coercivity could be produced by pinning. As in the bulk materials bloch wall can move through the magnetic domains, they may pinned at grain boundaries and for continuing this movement an additional energy, coercivity, is required.

The relationship between coercivity and particle size of materials was studied and quantified by Herzer for the first time. He tries to produce a theory which is reliable for a magnetic particles ranging from 10 to 300 nm, which is known as the grain size dependence of coercivity and permeability (GSDCP) theory. Herzer demonstrated there is a critical particle size above which the relation between size and coercivity defines by the D^{-1} (eq. 5):

$$H_C = P_1 \frac{\sqrt{AK}}{M_s D} \propto 1/D \qquad (5)$$

Here K is magnetocrystalline anisotropy constant, A represents the exchange constant, and P1 is dimensionless factor. This equation represent with reducing the grain size more pining sites could be produced and coercivity is increased. However, below the critical size, the coercivity complied with a D^6 power law (eq. 6).

$$H_c = P_2 \frac{K^4 D^6}{M_s A} \propto D^6 \qquad (6)$$

In the GSDCP theory the critical size is defined as the exchange length which could be calculated by the fallowing equation (eq. 7):

$$L_{ex} = \sqrt{\frac{A}{K}} \qquad (7)$$

Exchange length for magnetite is calculated as 27 nm ($K = 1.35 \times 10^4$ J/m^3, $A = 10^{-11}$ J/m) [27]-[30] .
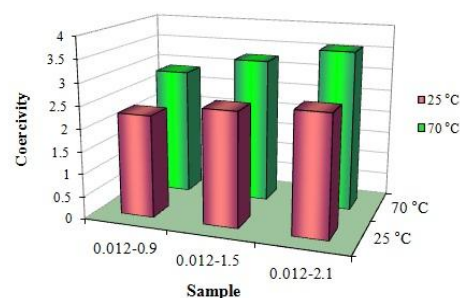

Fig. 7. Dependence of coercivity on alkaline media concentration.

As the samples prepared in this study have particle size below the critical one, the coercivity should be reduced by reduction of particle size as a function of reduction of ingredient concentration, which is obviously observable in Fig. 7.The low coercivity, less than 10 Oe, demonstrates that the particles are superparamagnetic. Fig. 7 shows that all prepared sample have coercivity below the 5Oe and all particles are superparamagnetic.

IV. CONCLUSION

Samples synthesized by modification of temperature and optimization of alkaline media concentration have higher saturation magnetization and smaller particle size compared

to other prepared by common copercipitation method. The results indicate that concentration and temperature have significant effects on magnetic properties, particle size and size distribution. Surface defects are decreased by increasing of synthesis temperature and prolonged reaction time; this matter resulted in improvement of saturation magnetization and other magnetic properties and reduction of particle size. Increasing of alkaline media concentration decreases the saturation magnetization and increases the particle size. Also, decrease of alkaline media causes the reduction of coercive force, this matter is one of the great phenomena happens in nano scale. Optimum synthesis formula which results in highest saturation magnetization and smallest particle size was obtained at alkaline media concentration of 0.9 M and prolonged synthesis time at 70 ℃.

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