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# SYNTHESIS OF MAGNETITE UNDER MICROWAVE INFLUENCE

***Purpose.*** *The aim of this work was to synthesize magnetic nanoparticles, using iron (II) sulfate and alkaline reagents under the influence of microwave radiation, elevated temperatures and pressure; to investigate the mineral composition of the obtained magnetic material using the method of thermomagnetic analysis.*

***Keywords****: magnetic nanoparticles, synthesis, thermomagnetic analysis, magnetometry*

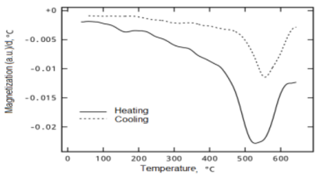
***Objectives.*** Nowadays, synthesis of magnetic nanoparticles is very important for solving both fundamental problems and many technological applications. Due to their unique characteristic, as high saturation magnetization, one could easily operate them by the magnetic field, so they are widely applied in different medical-biological applications. For these applications, magnetite must be finely dispersed with a grain size of less than 100 nm [1]. The methods of magnetite nanoparticles synthesis with such characteristics are rapidly developing. Obtaining magnetite nanoparticles depends mainly on such parameters as reaction temperature, pH of the suspension, initial molar concentration, etc. The selection of reagents plays not the least role, since the physical and chemical properties of the obtained magnetite nanoparticles are highly dependent on the choice of the method of their synthesis. Therefore, the topic is currently quite relevant.

In this work, a method based on the chemical co-precipitation method was used, the latter is widely used for obtaining magnetic nanoparticles. Magnetic nanoparticles are produced under the influence of microwave radiation, elevated temperatures and pressure. The difference of this method is as follows. Microwave radiation increases the speed and efficiency of mineral formation processes, due to the direct effect of radiation on water molecules in the solution. An oxygen-free environment (synthesis in a closed system under pressure) promotes minimal oxidation of the magnetic material and the production of particles with a grain size of less than 100 nm.

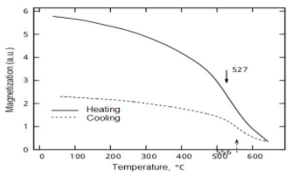
***Methodology.*** The obtained samples were investigated by thermomagnetic analysis and magnetometry methods. Magnetic characteristics of synthesized magnetic nanoparticles were determined using magnetometer with Hall sensor. An external magnetic field of magnetometer varied in the range of 0 to ± 0.45 T. Nickel carbonyl with saturation magnetization at room temperature of 54,4 A·m$^2$/kg, was used as reference sample [2]. Curie temperature and composition of obtained magnetic nanoparticles were determined by thermomagnetic analysis [3]. The temperature range of sample heating was from room temperature to 650 ºC. The rate of sample heating/cooling was 65°/minute.

***Research results***. Iron (II) sulfate heptahydrate ($FeSO_4 \cdot 7H_2O$) and ammonium hydroxide ($NH_4OH$) were used as chemical reagents for the synthesis of magnetite. Magnetite nanoparticles were obtained by the method of iron co-precipitation in an alkaline medium. The preparation of the reagents was performed as follows: $FeSO_4 \cdot 7H_2O$ was dissolved in distilled water and 3M alkali was added to the salt's solution. The solution was incubated in Microwave Reaction System (Anton Paar Multiwave PRO). The samples were synthesized at elevated temperatures (120 °C), pressure (60 bar) and time for 30 min. The formed samples were washed with distilled water and dried in a thermostat at 90 °C for 1 h.

The specific saturation magnetization of the obtained samples were ~ 88 A·m$^2$/kg. According to thermomagnetic analysis data, magnetic samples consist of magnetite, most likely with a small admixture of maghemite (Fig.1). TMA and especially DTMA curves show a sharp decrease in magnetization at ~530 °C, which indicates the presence of magnetite, and a small peak in the temperature range of 100 – 200 °C can be attributed to the main inversion of maghemite.




a



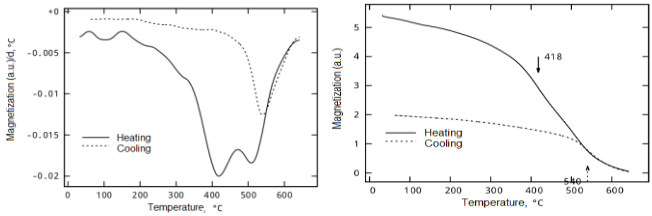

b

Fig. 1. **DTMA (a) and thermomagnetic (b) curves of the samples synthesized at 120 °C.**

We assume that after several more heatings, the sample will oxidize and the Curie temperature of the ferromagnetic residue will approach the temperature of pure magnetite (580 °C). Therefore, the obtained samples were reheated in the temperature range from room temperature to 650 ºC. The resulting thermomagnetic curves are shown in Fig. 2.

Upon reheating, a solid solution of maghemite in magnetite was formed. During the first heating in air, the inversion of maghemite into hematite occurs (T-inversion somewhere at 410-415 °C), in addition, there is a phase of maghemite-magnetite solid solution with a Curie temperature of about 510 °C. Upon cooling, only a part of the maghemite-magnetite solid solution with a

Curie temperature of 540 °C remains, with a significant predominance of the magnetite mineral.




**a** **b**

Fig. 2. **DTMA (a) and thermomagnetic (b) curves of the samples synthesized at 120 °C after reheating.**

The process of the magnetite formation we could describe as follows: iron (II) sulfate salt is unstable in aqueous solution, and $Fe^{2+}$ is partially oxidized to $Fe^{3+}$. After that, adding of alkali starts the co-precipitation reaction that takes place because of temperature and microwave treatment and leads to magnetite formation. We could conclude that obtained samples are promising for different application.

***Conclusion.*** Incubation of Fe (II)-containing solution under the influence of microwave radiation, pressure and elevated temperatures leads to the synthesis of magnetized magnetic nanoparticles with a specific saturation magnetization of 88 $A \cdot m^2/kg$. It was shown that in an aqueous medium, using iron (II) sulfate and alkaline reagents under the influence of microwave radiation, magnetite with maghemite admixture is formed. Upon reheating, the magnetization will decrease even more, and the residual magnetite will increase the Curie temperature to 580 °C. The magnetization of obtained samples is rather high and makes them promising for different applications (adsorbents of radioactive waste, carriers for magnetic drug targeting, etc.). The results could be used to solve the fundamental problems associated with the transformation of iron oxides and hydroxides in aqueous medium.

## References

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