УДК: 677.027
DOI 10.30857/2706-5898.2025.2.2

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THE EFFECT OF EXTERNAL PHYSICAL FACTORS ON THE FORMATION AND PROPERTIES OF SYNTHETIC MAGNETIC MATERIALS

Purpose. *Investigation of the effect of external physical factors on the formation of the structure and properties of synthetic magnetic materials for processing textiles of different raw materials.*

Methodology. *The structural characteristics were studied by X-ray diffraction (XRD). The saturation magnetization (Ms) of the created synthetic magnetic samples was investigated using the magnetometry method. To determine the absolute value of the magnetic moment (magnetization) of these samples, calibration was performed using a reference sample with a known saturation magnetization.*

Results. *The values of the saturation magnetization of synthetic nanomagnetite samples when changing the conditions of their synthesis range from 23±1 A·m²/kg to 41±1 A·m²/kg, the average size of magnetite crystallites varies from 6.3±0.1 nm to 10.5±0.1 nm, which confirms the obtaining of nanosized magnetite samples in pure form. The regularity of the influence of synthesis conditions on the size of magnetite nanocrystallites, its structure and properties depending on the action of various external factors has been established. For samples synthesized under ultrasonic treatment conditions without a magnetic field, the saturation magnetization values are, on average, higher than for samples synthesized under ultrasonic treatment conditions and a magnetic field.*

Scientific novelty. *For the first time, the effect of external physical factors (ultrasonic treatment, application of a magnetic field during the formation of nanomagnetite) was studied and the patterns of their influence on the magnetic and structural characteristics of synthetic magnetic materials were established.*

Practical value. *The study of the influence of ultrasonic treatment and the application of a magnetic field on the formation of synthetic analogues of biogenic magnetite is of great importance for the further synthesis of magnetite and the regulation of its properties when processing textile materials of various raw materials. The practical potential of the processed samples lies in the ability to shield microwave energy, the ability to which is provided by the controlled synthesis of magnetite nanoparticles under the action of various external factors.*

Keywords: *synthetic magnetite, nanotechnology, structure, properties.*

ВПЛИВ ДІЇ ЗОВНІШНІХ ФІЗИЧНИХ ЧИННИКІВ НА ФОРМУВАННЯ ТА ВЛАСТИВОСТІ СИНТЕТИЧНИХ МАГНІТНИХ МАТЕРІАЛІВ

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Мета. *Дослідження впливу дії зовнішніх фізичних чинників на формування структури і властивостей синтетичних магнітних матеріалів для обробки текстилю різного сировинного складу.*

Методи. *Дослідження структурних характеристик виконано методом рентгенофазового аналізу (РФА). Методом магнітометрії досліджено намагніченість насичення (Ms) створених* *синтетичних магнітних зразків. Для визначення абсолютної величини магнітного моменту (намагніченості) цих зразків виконувалась калібровка за еталонним зразком з відомою намагніченістю насичення.*

Головні результати. *Значення намагніченості насичення зразків синтетичного наномагнетиту при зміні умов їх синтезу становлять від 23±1 А·м²/кг до 41±1 А·м²/кг, середній розмір кристалітів магнетиту змінюється від 6,3±0,1 нм до 10,5±0,1 нм, що підтверджує отримання зразків нанорозмірного магнетиту в чистому вигляді. Встановлена закономірність впливу умов синтезу на розмір нанокристалітів магнетиту, його структуру та властивості у залежності від дії різних зовнішніх факторів. Для зразків, синтезованих в умовах ультразвукової обробки без магнітного поля, значення намагніченості насичення, в середньому, вищі, ніж для зразків, синтезованих в умовах ультразвукової обробки та магнітного поля.*

Наукова новизна. *Вперше вивчено дію зовнішніх фізичних факторів (ультразвукова обробка, накладання магнітного поля в процесі формування наномагнетиту) та встановлено закономірності їх впливу на магнітні та структурні характеристики синтетичних магнітних матеріалів.*

Практична значимість. *Дослідження впливу ультразвукової обробки та накладання магнітного поля на формування синтетичних аналогів біогенного магнетиту має велике значення для подальшого синтезу магнетиту та регулювання його властивостей при обробці текстильних матеріалів різного сировинного складу. Практичний потенціал оброблених зразків полягає в можливості до екранування енергії мікрохвиль, здатність до якого забезпечує контрольований синтез наночастинок магнетиту за умов дії різних зовнішніх чинників.*

Ключові слова: *синтетичний магнетит, нанотехнологія, структура, властивості.*

Introduction. Currently, for the practical implementation of products that are nanostructured materials, it is necessary to develop new technologies for their production in order to successfully realize the potential of nanotechnology in the consumer properties of the resulting product. Today's challenges encourage the search for new methods for creating «smart» materials containing iron-oxide compounds of nanosize, due to the unique properties of magnetic nanoparticles [1–5], for example, their ability to intensively absorb microwaves and exhibit a bactericidal effect [4].

Many chemical methods can be used for the synthesis of magnetic nanoparticles for biomedical purposes [6–12]: synthesis in microemulsions, sol-gel synthesis, chemical reactions using ultrasound, hydrothermal reactions, hydrolysis and thermolysis of precursors, flow-injection synthesis, electrochemical synthesis. These methods are used to produce nanoparticles of homogeneous composition and with a narrow size distribution. However, the most common method for obtaining magnetic nanoparticles was and remains the method of chemical co-precipitation of iron salts [13–16]. It seems relevant to clarify the patterns of the relationship between synthesis conditions and structural characteristics and magnetic properties of iron oxide materials. Solving similar problems is of great importance for the development of nanotechnologies for the creation of «smart» materials for the purpose of controlled synthesis of nanosized magnetite in nanocomposites of various nature.

Analysis of previous studies. Various metal-containing compounds are used as starting materials for the synthesis of magnetic nanoparticles: metal carbonyls, organometallic compounds, salts of carboxylic acids. Most often, the precursor decomposes upon heating or UV irradiation, but other methods have been developed that facilitate the production of nanoparticles: a) thermolysis of metal-containing compounds, which has been studied in detail in connection with the creation of the scientific foundations of the MOCVD (Metal Organic Chemical Vapour Deposition) method, which is successfully used to produce nanoparticles. When the reaction is carried out in liquid media in the presence of surfactants or polymers, it is possible to stabilize the formed amorphous nanoparticles with a diameter of up to 10 nm [6, 18]; b) decomposition of metal-containing compounds under the influence of ultrasound. In this method, metal carbonyls or their derivatives are usually used as metal-containing compounds, although there are cases of successful use of other organometallic compounds. Thus, for the synthesis of Co nanoparticles, the decomposition of a solution of $Co_2(CO)_8$ in toluene under the influence of ultrasound was used [19]. To maintain monodispersity and prevent aggregation during particle formation, the sodium salt of bis-(2-ethylhexyl) sulfosuccinic acid was added to the solution; c) reduction of metal-containing compounds. For the synthesis of magnetic nanoparticles from salts of the corresponding metals, strong reducing dispersions of alkali metals in esters or carbohydrates, complexes of alkali metals with organic electron acceptors (such as naphthalenes) and other complex hydrides were used. When using $NaBH_4$ in aqueous solutions at room temperature, both homo-(Fe, Co, Ni) and heterometallic (Fe-Co, Fe-Cu, Co-Cu) nanoparticles were obtained in the form of amorphous powders containing a significant amount of boron (20 wt.% and more) [18]; d) synthesis in reverse micelles. In recent years, the method of synthesizing nanoparticles in nanoscale "reactors" has been intensively developed and widely used, since it allows, within certain limits, to regulate the sizes of "nanoreactors" in which the synthesis takes place. Such nanoreactors include, in particular, micelles. Reverse micelles are the smallest water droplets stabilized in a hydrophobic liquid phase due to the formation of a surfactant monolayer on their surface. Due to the precise dosing of the amount of metal-containing compounds in each micelle, it is possible not only to regulate the composition and average particle sizes, but also to obtain monodisperse samples with a fairly narrow particle size distribution [13, 14]; e) the sol-gel method, which is widely used in a number of technologies [19, 20]. In nanotechnology, it is usually used to obtain metal oxides, but can also be used for the synthesis of nanoscale metals and "alloy" bimetallic and heteroelement particles. Thus, the reduction of Ni^{2+} and Fe^{2+} ions, introduced into silica gel in a ratio of 3:1, with hydrogen at 733–923 K led to the formation of nanoparticles (4–19 nm) of the Ni_3Fe composition inside the SiO_2 matrix [19]; e) encapsulation and self-organization of magnetite nanoparticles. It is known that nanoparticles of some metals are pyrophoric, i.e. they spontaneously ignite in air at room temperature, therefore the creation of a protective shell on such nanoparticles (encapsulation) is a common method of their protection and stabilization. Carbon is often used as a protective coating [21, 22]. Encapsulation of magnetic nanoparticles makes them resistant to oxidation, corrosion and spontaneous aggregation, which allows them to maintain their single-domain nature. Other methods for the synthesis of encapsulated magnetic nanoparticles are based on the use of high-temperature plasma, laser pyrolysis, thermal evaporation, and chemical encapsulation methods [21–24]. Despite the large number of works performed the considered area of research is only at the development stage.

Problem statement. The influence of synthesis conditions and methods on the formation of the structure and morphology of magnetic synthetic materials is insufficiently studied, therefore, research related to the synthesis of magnetite, its magnetic and structural features depending on the action of various external physical factors (ultrasonic (US) treatment, the action of a magnetic field) is a relevant task.

The purpose of the work is to study the influence of external physical factors on the formation of the structure and properties of synthetic magnetic materials for processing textiles of various raw material compositions.

Methodology. The objects of research were samples of synthetic magnetites formed by the method of chemical coprecipitation of two types: coprecipitation of a mixture of iron (II) sulfate:iron (III) chloride and a mixture of iron (II) chloride:iron (III) chloride at temperatures from 20 °C to 90 °C with a concentration of iron sulfate: 20 (C20), 40 (C40), 60 (C60), 80 (C80), 100 (C100).

To study the effect of ultrasonic treatment and the application of a magnetic field on the properties of synthetic analogues of biogenic magnetite, 4 samples of nanomagnetite were synthesized by the method of coprecipitation of Fe^{3+} and Fe^{2+} salts in an alkaline medium under the influence of two different external conditions (magnetic field and ultrasound). During the synthesis of samples, which took place under the influence of ultrasound, an ultrasonic device UP200S (operating frequency 24 kHz, power 200 W) and a magnetic field of 8 mT were used. The conditions of sample synthesis: sample 1 – synthesis without ultrasound and without a magnetic field, sample 2 – synthesis under magnetic field conditions; sample 3 – synthesis under the influence of ultrasound without a magnetic field; sample 4 – synthesis under magnetic field conditions under the influence of ultrasound.

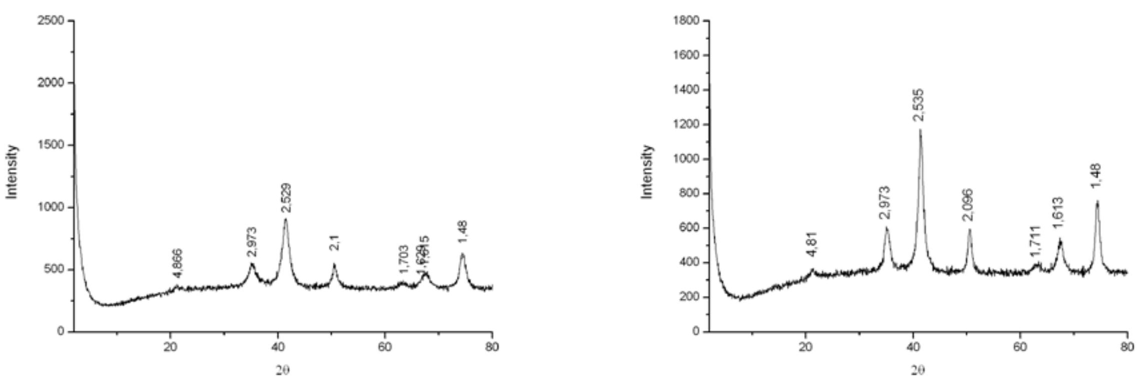
The study of structural characteristics was performed by the method of X-ray phase analysis (XPA). XPA of samples was carried out on a DRON-3M diffractometer, copper radiation (CuKα = 1.54178 Å). The scanning range of samples (2θ) – 10° – 70°. The diagnostic results were compared with reference samples of the PCPDFWIN (PDF-2) 2003 data bank of the American card index.

The saturation magnetization (Ms) of the obtained samples was investigated by magnetometry using a magnetometer with a Hall sensor. To determine the absolute value of the magnetic moment (magnetization) of these samples, calibration was performed using a reference sample with a known saturation magnetization. Pure nickel was used as the reference sample, the saturation magnetization of which was determined with high accuracy (54.5 A · m²/kg at room temperature).

Research results and discussion. Experimental studies have shown the fundamental possibility of using the principles of nanotechnology in the direction of developing physicochemical processes for creating magnetite nanoparticles under different conditions [1–4]. This work presents nanotechnologies for creating synthetic analogues of biogenic magnetic minerals – synthetic magnetites, synthesized using different technological conditions and methods in order to obtain synthetic materials with magnetic properties.

The work investigates the effect of ultrasonic treatment and the application of a magnetic field on the properties of synthetic analogues of biogenic magnetite, which may be useful for the further synthesis of magnetite with specified and controlled properties.

Using X-ray phase analysis, the structural features of synthetic magnetites synthesized under different conditions were studied. Diffraction peaks in the diffractograms indicate that magnetite nanoparticles were obtained as a result of the synthesis (Fig. 1). The relative crystallite size of the synthesized nanoparticles was calculated using the Debye-Scherrer formula and was 10.5±0.1 nm for nanomagnetite particles synthesized by ultrasonic treatment of the reaction mixture and 6.3±0.1 nm for particles synthesized without ultrasound. It was shown that the saturation magnetization of the nanomagnetite sample synthesized by ultrasonic treatment of the reaction mixture (37 A·m²/kg) is higher than the saturation magnetization of the sample synthesized without ultrasound (24 A·m²/kg). The unit cell parameter a calculated for nanoparticles synthesized using ultrasonic treatment (a = 8.383±0.007) was closer to the unit cell parameter of macroscopic magnetite (a = 8.393±0.001) than the parameter calculated for nanoparticles synthesized without ultrasonic treatment (a = 8.362±0.0009). Thus, ultrasonic treatment of the reaction mixture affects the properties of synthetic nanomagnetite, contributing to an increase in the unit cell parameters, size and saturation magnetization of the resulting nanoparticles.



a) b)

Fig. 1. **Diffraction patterns of a synthetic nanomagnetite sample without US treatment (a) and a nanomagnetite sample synthesized under the action of US (b).**

It is shown that according to the X-ray diffraction data, all the obtained samples are magnetically ordered minerals (magnetite/ maghemite).

According to the obtained curves (Fig. 2), which show the dependence of the magnetization of the substance (Ms) on the magnitude of the magnetic field induction (B), the values of the saturation magnetization were estimated, which are given below (Table 1).

The graph (Fig. 2) shows a rapid initial increase in the magnetization of the particles with increasing field, and then a gradual increase to saturation. In our case, the shape of the hysteresis curves of the synthesized samples is extremely narrow, which is a characteristic feature of superparamagnetic nanoparticles.

Table 1

Values of the saturation magnetization and average particle sizes

Sample	Particle size, nm	Saturation magnetization, $A \cdot m^2/kg$
Sample 1	6.3±0.1	24±1
Sample 2	7.0±0.1	32±1
Sample 3	10.5±0.1	37±1
Sample 4	7.1±0.1	32±1

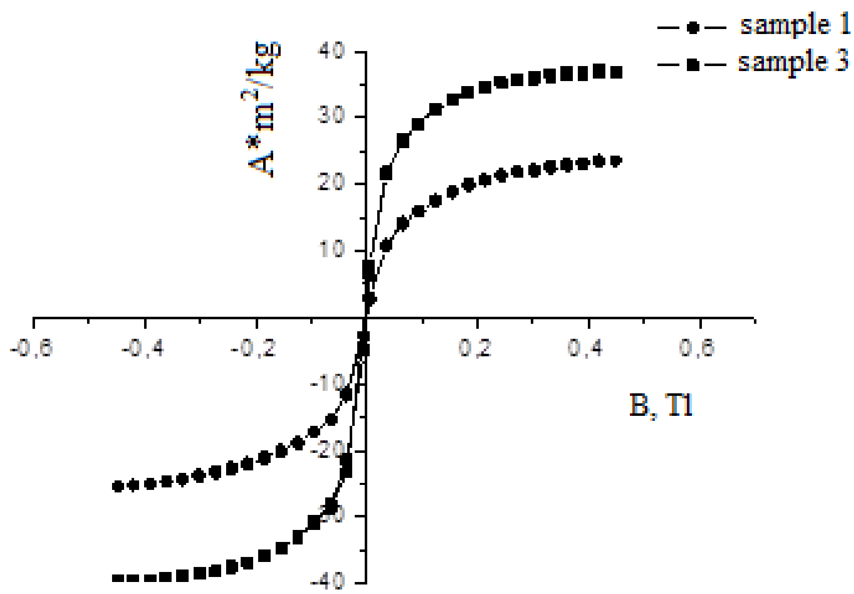


Fig. 2. **Appearance of the magnetization curve for samples 1 and 3**

Magnetic studies were performed using a magnetometer with a Hall sensor, which is designed to measure the parameters of the hysteresis loop in the limit and partial hysteresis cycles of powder isotropic and anisotropic materials. The obtained experimental results of the saturation magnetization of synthesized magnetite nanoparticles obtained in the presence of a magnetic field and ultrasonic treatment show the following. Under conditions of a magnetic field, the particle size increases (sample 2). The action of ultrasound further increases the particle size (sample 3). However, the simultaneous action of a magnetic field and ultrasound leads to a smaller effect than the action of ultrasound without a magnetic field (sample 4). The obtained experimental results allow us to establish the effect of changing the conditions of magnetite synthesis: the action of a magnetic field affects the particle size, which increases, the application of ultrasound further increases the particle size. However, the simultaneous action of a magnetic field and ultrasound leads to a smaller effect than the action of ultrasound without a magnetic field. The obtained data also show another important regularity: with increasing particle size, the magnetization of the samples increases. This dependence may be due to the fact that Fe ions at the boundaries between crystallites do not contribute to the total magnetization. With increasing crystallite size, the relative fraction of such ions decreases and the total magnetization increases.

To confirm the obtained results, additional experiments were conducted on the formation of magnetic nanoparticles at different concentrations of iron salts (C = 20 – 100 g/l) in the presence/absence of ultrasound and the application of a magnetic field depending on the change in the power of ultrasound treatment. The general conditions for the synthesis of magnetic nanoparticles and the product yield are given in Table 2. The study of the influence of the power of ultrasound treatment on the value of the saturation magnetization of synthetic nanomagnetite samples (from 4 to 20 W) (Fig. 3, Fig. 4) shows that the presence of a magnetic field under the conditions of the synthesis of magnetic nanoparticles leads, on average, to a decrease in the saturation magnetization of magnetic nanoparticles.

Table 2

Conditions and results of studies of synthesized samples

Sample	Temperature dynamics, °C	Ultrasonic disperser power, W	Synthesis time, h	Magnetic field	Ultrasound	Saturation magnetization, A·m²/kg	Product yield, g
M 20	24 – 80	4	1	+	+	33	0.4514
M 40		8		+	+	31	0.4872
M 60		11		+	+	30	0.4789
M 80		15		+	+	31	0.4767
M 100		19		+	+	29	0.5114
NM 20	20 – 80	4	1	–	+	23	0.5111
NM 40		8		–	+	40	0.4805
NM 60		11		–	+	40	0.4778
NM 80		15		–	+	41	0.4664
NM 100		19		–	+	37	0.3602

Note: M – samples synthesized with a magnetic field and US;
NM – samples synthesized without a magnetic field, but with US.

Thus, it can be concluded that the power of ultrasonic treatment, when synthesizing magnetite in the presence/absence of a magnetic field, does not affect the saturation magnetization values, therefore, the saturation magnetization values are on average the same for all synthesized samples. For samples synthesized under ultrasonic treatment and in the absence of a magnetic field, the saturation magnetization values are, on average, higher than for samples synthesized under ultrasonic treatment and in the presence of a magnetic field. That is, the application of a magnetic field to the reaction mixture leads to a decrease in the saturation magnetization values of the samples.

Thus, it is shown that the application of ultrasonic vibrations to the reaction mixture leads to an increase in the saturation magnetization of the samples compared to the samples synthesized in the absence of ultrasonic vibrations, and the application of a magnetic field to the reaction mixture, on the contrary, reduces the saturation magnetization of the samples.

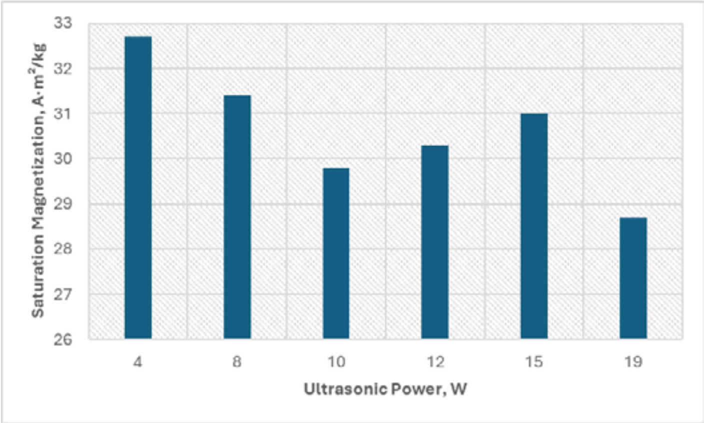


Fig. 3. **Dependence of saturation magnetization on the power of the US disperser (with a magnetic field)**

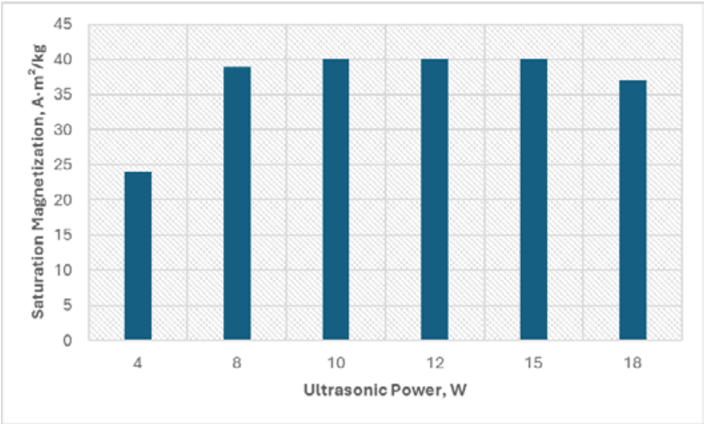


Fig. 4. **Dependence of saturation magnetization on the power of the US disperser (without magnetic field)**

Thus, in the nanotechnology processes of creating textile materials with magnetic properties, it is necessary to take into account the conducted studies and established conditions for obtaining synthetic nanomagnetite in terms of protection from the influence of various external physical factors.

Conclusions. It is shown that magnetite samples exhibit a very rapid initial increase in magnetization with increasing field, and then a gradual very slow increase to saturation. The shape of the hysteresis loops for the synthesized samples is extremely thin, they do not exhibit residual magnetization or coercivity. That is, synthetic magnetite samples are superparamagnetic. The values of saturation magnetization of synthetic nanomagnetite samples when changing the conditions of their synthesis range from 23±1 A·m^2/kg to 41±1 A·m^2/kg, the average size of magnetite crystallites varies from 6.3±0.1 nm to 10.5±0.1 nm, which confirms the obtaining of nanoscale magnetite samples in pure form.

The regularities of the influence of various external physical factors (ultrasound treatment and the action of a magnetic field) on the properties and structure of synthetic analogues of biogenic magnetite have been established, which may be useful for the further synthesis of magnetite with controlled properties for the processing of textile materials of various raw material composition.

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