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PRODUCTION OF MAGNETITE NANOPOWDER BY HYDROGEN REDUCTION FROM α-FeOOH HYDROXIDE COMPOUND UNDER ISOTHERMAL CONDITIONS

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In this work, the process of obtaining magnetite nanopowder Fe_3O_4 by hydrogen reduction from α-FeOOH hydroxide compound under isothermal conditions was studied. The α-FeOOH nanopowder was prepared in advance by chemical precipitation from aqueous solutions of iron nitrate $Fe(NO_3)_3$ (10 wt. %) and alkali NaOH (10 wt. %) at room temperature, pH = 11, under the condition of continuous stirring. The hydrogen reduction process of α-FeOOH powder under isothermal conditions was carried out in a tube furnace in the temperature range from 310 to 375 °C. The study of the crystal structure and phase composition of the powder samples was performed by XRD phase analysis. The specific surface area S of the samples was measured using BET method by low-temperature nitrogen adsorption. The average particle size D of powders was calculated using the data of measuring the specific surface area S. The size and morphology characteristics of the particles were investigated by scanning and transmission electron microscopy method. The calculation of the kinetic parameters of the process of obtaining the Fe_3O_4 nanopowder under isothermal conditions was carried out using the Gray–Weddington model and the Arrhenius equation. It was found that the reduction process of the α-FeOOH nanopowder begins to accelerate strongly in the temperature range from 340 to 375 °C. The reduction rate constant at 375 °C is about 2 times that of the reduction at 310 °C. The value of the activation energy for the process of obtaining Fe_3O_4 nanopowder under isothermal conditions was ~ 36 kJ/mol, which indicates a mixed mode of limiting the kinetics of the process. It has been shown that the time-temperature regime of the reduction of the α-FeOOH hydroxide compound with holding at 340 °C for 3 h makes it possible to obtain a nanosized magnetite powder Fe_3O_4 with high purity at an increased rate of the process. The obtained Fe_3O_4 nanoparticles have a crystalline structure and generally have a polygonal or irregular shape, the size of which ranges from 30–80 nm with an average value of 65 nm, each of them being connected to several neighboring particles by isthmuses.

Keywords: kinetics, magnetite nanopowder, hydrogen reduction, isothermal conditions, reduction degree, rate constant, activation energy.

Introduction

Currently, magnetite Fe_3O_4 nanopowders (NP) have found wide application in various fields of science, technology, and medicine [1–4]. Fe_3O_4 nanoparticles (NPs) functionalized with biomolecules can be used as materials for targeted drug delivery in order to recognize or treat biological systems. Due to their magnetic properties, Fe_3O_4 NPs are also often used in magnetic hyperthermia to destroy tumor cells, in magnetic resonance imaging (MRI) as contrast agents, to create actively antitoxic adsorbents in the field of nanotoxicology. In addition, dispersion, non-toxicity, and superparamagnetic properties make Fe_3O_4 NPs interesting for use in other fields, for example, in the manufacture of effective catalysts, biosensors, ferromagnetic liquids, as a feedstock for the synthesis of Fe NP by reduction [2, 3, 5–13].

The production of metals and oxides NP, including Fe_3O_4 NP, is carried out in various ways, most of which are characterized by a number of disadvantages, such as high energy consumption, reduced productivity, and environmentally uncleanness [5, 6, 9, 14]. Chemical-metallurgy method, which includes the stage of hydrogen reduction of oxygen-containing compounds, is a highly efficient method from a technology and economic point of view, because the method is quite simple in equipment, environmentally friendly and makes it possible to use industrial waste as a raw material, to regulate the dimensional characteristics of oxides and metals NPs in the course of their production [6, 15–17].

A big disadvantage of chemical-metallurgy method for producing oxides and metals NP is low productivity due to the low speed of the reduction process under conditions of holding at low temperatures. At that time, an excessive increase in the reduction temperature is not recommended because it leads to intensive processes of aggregation and sintering of NPs, respectively, to the formation of particles with a particle size outside the nanoscale range [13, 18].

Thus, the study of the kinetic characteristics of synthesizing process of Fe_3O_4 NP in order to find a way to accelerate it while guaranteeing the required properties of the obtained products is an important scientific-practical problem.

In this regard, the aim of this work is to study the kinetics of synthesizing process of Fe_3O_4 NP by hydrogen reduction from α-FeOOH NPs under isothermal conditions, as well as to analyze the structural and dimensional properties of the obtained magnetite nanoparticles.

Materials and methods

As materials for the preparation of Fe_3O_4 NP was used iron hydroxide (III) α-FeOOH NP, synthesized by chemical precipitation from aqueous solutions of iron nitrate $Fe(NO_3)_3$ (10 wt. %) and alkali NaOH (10 wt. %) at room temperature, pH = 11, and continuous stirring. Getting iron hydroxide proceeds by the reaction:

$$Fe(NO_3)_3 + 3\,NaOH = \\ = FeOOH\downarrow + 3\,NaNO_3 + H_2O. \qquad (1)$$

The pH was monitored with an “Expert-001” pH meter; the measurement error was ±0.03. Using a Buchner funnel, the obtained α-FeOOH precipitate was washed until the ions of the dissolved salt were completely washed off, which was controlled by the pH of the solution over the precipitate. Then the precipitate was dried at 40 °C for 48 h, the thickness of the filling of the hydroxide paste in the plates was no more than 10 mm. The time-temperature regime of drying was determined experimentally for the complete removal of the precipitate of adsorbed water molecules from the sample while ensuring a high dispersion of α-FeOOH particles. Thereafter, the dried α-FeOOH was milled using a laboratory mill “Fritsch Pulverisette 2”. The obtained α-FeOOH powder was analyzed for structural and dispersed properties and was used for further research.

The study of the kinetics of the process of obtaining Fe_3O_4 NP was carried out in a tubular furnace “SNOL 0.2/1250” in a hydrogen atmosphere at various temperatures. During the process, the following reaction took place:

$$6\,FeOOH + H_2 = 2\,Fe_3O_4 + 4\,H_2O. \qquad (2)$$

The hydrogen generator “CAM-1” was used as a source of hydrogen; the relative humidity of the obtained hydrogen did not exceed 1%. To prevent the ignition of the resulting powders in air, after reduction, their surface was passivated in a stream of nitrogen taken from the dewar of liquid nitrogen.

The calculation of the degree of conversion (α) (u.fr.) (the ratio of the amount of the reagent that entered the reaction to its initial amount) was carried out according to the formula:

$$\alpha = \frac{m_0 - m_t}{m_0} \cdot \frac{6M_{\alpha\text{-FeOOH}}}{(3M_{H_2O} + M_O)} = \\ = 7.6 \cdot \frac{m_0 - m_t}{m_0}, \qquad (3)$$

where m_0 is the initial weight of the sample, kg; m_t is the mass of the sample over time t, kg; M_{H_2O} is the molar mass of water; M_O is the molar mass of atomic oxygen.

The kinetics of synthesizing process of Fe_3O_4 NP by hydrogen reduction was studied using the model of Gray–Weddington [19]. The use of this model makes it possible to determine the values of the rate constant by the formula:

$$k \cdot t = 1 - (1 - \alpha)^{1/3}, \qquad (4)$$

where k is the rate constant, s^{-1}; t is the reaction time, s.

The calculation of the activation energy (E_a) (J/mol) was carried out according to the experimental data obtained under isothermal conditions using the integral form of the Arrhenius equation:

$$\ln k = -\frac{E_a}{R} \cdot \frac{1}{T} + \ln A, \qquad (5)$$

where A is a constant (pre-exponential factor); T is temperature, K; R is gas constant, J/(mol·K).

The phase composition of powder samples was determined by XRD phase analysis on a diffractometer "Difrey-401" (Russia) (Cr-K_α radiation) at room temperature. The size and shape of the obtained nanoparticles were investigated on scanning and transmission electron microscopes "TESCAN VEGA 3B" (SEM) (Czech Republic) and "LEO 912 AB OMEGA" (TEM) (Germany).

The specific surface area (S) (m^2/kg) of the samples was measured by the BET method based on low-temperature nitrogen adsorption on a "NOVA 1200e" analyzer (USA). The average particle size of the powders was calculated from the measurement data of the S value using the formula:

$$D = \frac{6}{\rho \cdot S}, \qquad (6)$$

where ρ is the pycnometric density of the material, kg/m^3; D is average particle size, m.

Results and discussion

The XRD and SEM results for the NP sample of the initial α-FeOOH are shown in Fig. 1.

The result of XRD (Fig. 1a) showed that the initial sample is single-phase, containing only the hydroxide α-FeOOH phase. Analysis of the SEM image (Fig. 1b) revealed that α-FeOOH NPs mainly have an acicular shape with a length of up to 200 nm. The specific surface area S of α-FeOOH NP is 39.5 m^2/g, respectively, the average size D of α-FeOOH NP is 44 nm.

In order to study the kinetic characteristics of synthesizing process of Fe_3O_4 NP under isothermal conditions, the dependences of the degree of conversion α on time t were obtained at different temperatures: 310, 325, 340, 355, 375 °C. These temperatures are in the range of intensive course of the reduction process, which was shown by the TG data in the work [18]. In Fig. 2 shows the curves of the dependence α(t) at different temperatures.

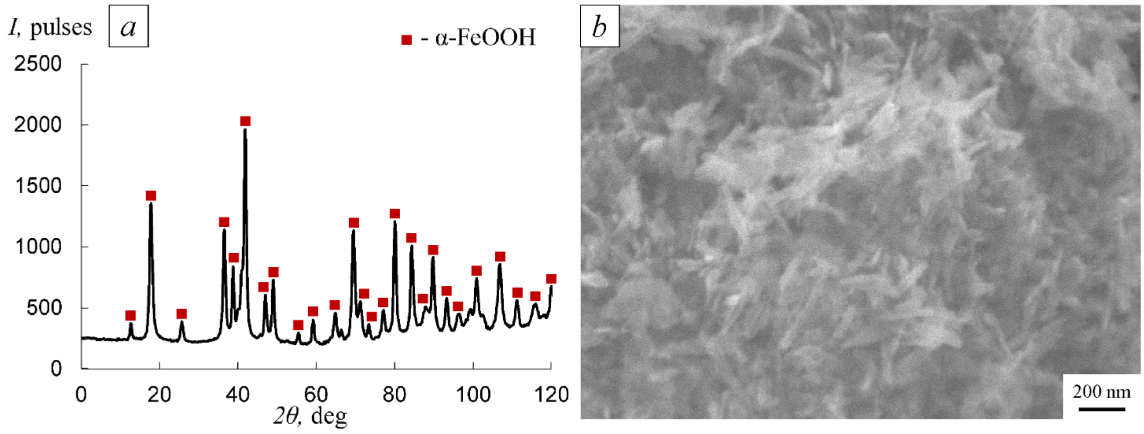

Fig. 1. XRD pattern (a) and SEM image (b) of the α-FeOOH NP sample

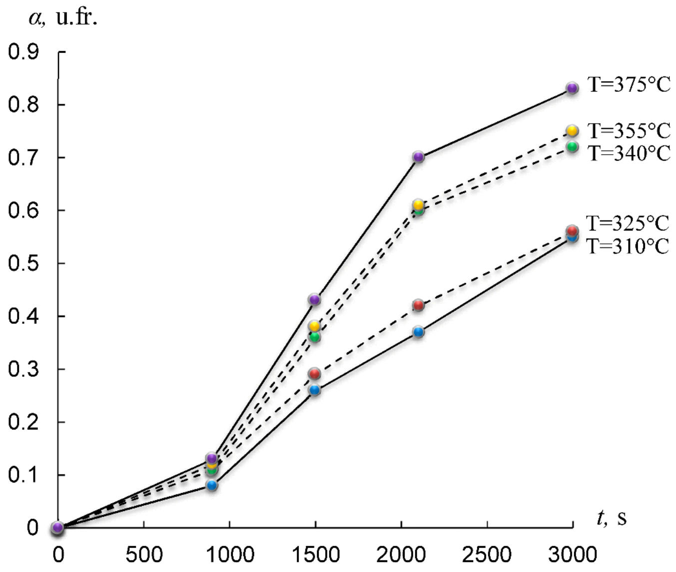

Fig. 2. Dependence α(t) at different temperatures

It can be seen that the reduction process of α-FeOOH NP begins to accelerate strongly in the temperature range from 340 to 375 °C. At temperature T = 375 °C for 50 min of reduction, the value of α was 83%, which is approximately more than 1.5 times compared to the case at 310 °C (for the same time).

The rate constant k of synthesizing process of Fe_3O_4 NP was calculated using the Gray–Weddington equation (4). The result of calculating the values of $k(T)$ is presented in Table 1.

According to the data in the table, it can be seen that the rate constant of reduction at 375 °C is about 2 times higher than the value obtained in the case of reduction at 310 °C.

To determine the activation energy E_a of synthesizing process of Fe_3O_4 NP by hydrogen reduction from α-FeOOH under isothermal conditions, the graphs of the dependence of the logarithm of the rate constant on the reciprocal temperature were plotted (Fig. 3) according to the Arrhenius equation (5).

The calculated E_a value was ~ 36 kJ/mol. Comparison of the obtained E_a value with the limiting values in the literature [20], it can be concluded that the synthesizing process of Fe_3O_4 NP by hydrogen reduction from α-FeOOH proceeds in a mixed mode of kinetics limiting. In this case, the rate of the general process of obtaining Fe_3O_4 NP is determined both by the rate of its own chemical reaction and by the rate of diffusion of reagents and reduction products through the powder layers; accordingly, an expedient way to accelerate the process can be both an increase in temperature and the elimination of diffusion layers by intensive mixing.

The next part of the work presents the results of study on the structural-dispersed characteristics of the reduction product of α-FeOOH NP at 340 °C, the holding time was 3 h (Fig. 4). On the one hand, this temperature is in the range of intensive acceleration of the process, and on the other hand, it is not too high, which, without

Table 1

The result of calculation of the rate constant *k* at different temperatures

$T_{reduction}$, °C		310	325	340	355	375
		α, u.fr.				
t, min	15	0.08	0.11	0.11	0.12	0.13
	25	0.26	0.29	0.36	0.38	0.43
	35	0.37	0.42	0.60	0.61	0.70
	50	0.55	0.56	0.72	0.75	0.83
$k \cdot 10^4$, s^{-1}		0.6947	0.7669	1.1121	1.1745	1.4143

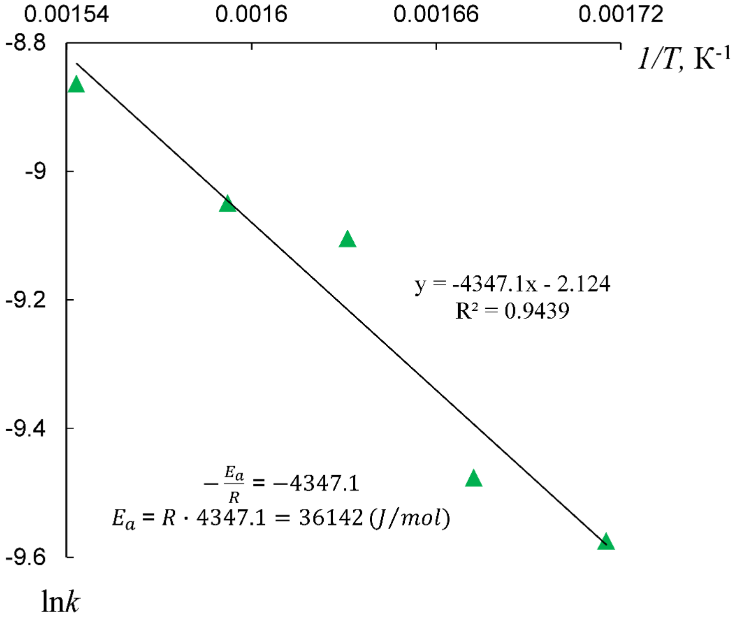

Fig. 3. Calculation of the activation energy in coordinates of the Arrhenius equation

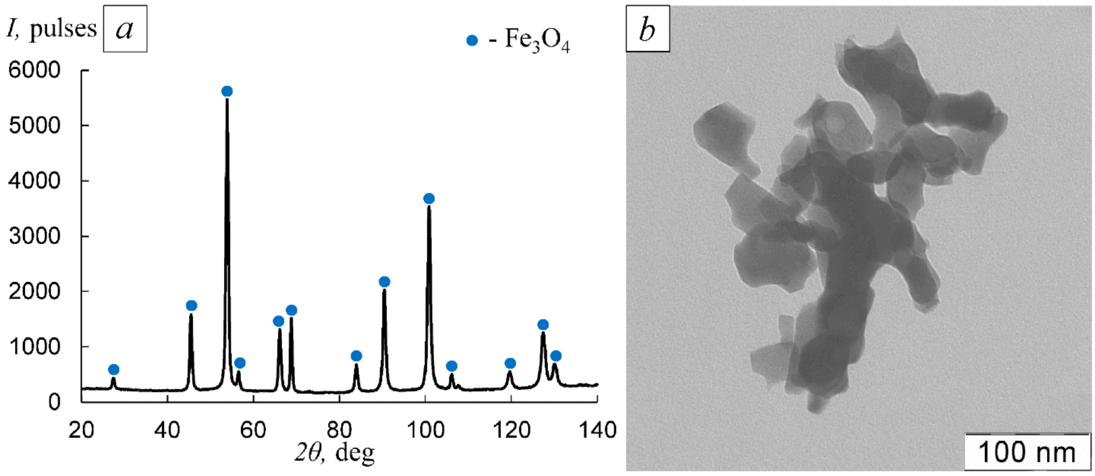

Fig. 4. XRD pattern (a) and TEM image (b) of the Fe_3O_4 NPs obtained by hydrogen reduction from α-FeOOH NP at 340 °C, holding for 3 h

leading to intensive processes of aggregation and sintering of the obtained magnetite NPs, makes it possible to obtain a reduction product with an increased quality. Nevertheless, in practice, depending on the task, in order to accelerate the reduction process while ensuring the required properties of the obtained products, it is desirable to vary the reduction temperature in the considered range from 340 to 375 °C.

The XRD result (Fig. 4a) revealed that the composition of the sample under study is single-phase, consisting only of the Fe_3O_4 phase with a cubic crystal lattice. The pronounced peaks on the XRD diffraction pattern of the sample show the high crystalline nature of the obtained magnetite particles. Analysis of the TEM image (Fig. 4b) of the sample showed that Fe_3O_4 NPs mainly have a polygonal or irregular shape, the size of which ranges from 30–80 nm, each of them is connected to several neighboring particles by isthmuses. The measurement result S of Fe_3O_4 NPs is in good agreement with the result of microscopic analysis, the S value is 18.3 m^2/g, which corresponds to the average size of Fe_3O_4 NPs $D = 65$ nm. Thus, the selected time-temperature regime for the reduction of the hydroxide compound α-FeOOH makes it possible to obtain nanosized crystalline magnetite powder with high purity at an increased rate of the process.

Conclusion

The kinetics of synthesizing process of Fe_3O_4 NP by hydrogen reduction from α-FeOOH under isothermal conditions at temperatures from 310 to 375 °C were studied. It was found that the reduction process of α-FeOOH NP begins to accelerate strongly in the temperature range from 340 to 375 °C. The reduction rate constant at 375 °C is about 2 times that of the reduction at 310 °C.

The value of the activation energy of synthesizing process of Fe_3O_4 NP was ~ 36 kJ/mol, which indicates a mixed mode of limiting the kinetics of the process. In this case, the rate of the general process is limited both by the rate of its own chemical reaction and by the rate of diffusion of reagents and reduction products through the layers of powders, respectively, an expedient way to accelerate the process is to increase the temperature or eliminate diffusion layers in the bulk of the powder sample by intensive mixing.

It has been established that the time-temperature regime of reduction of α-FeOOH with exposure at 340 °C for 3 h makes it possible to obtain a nanosized crystalline magnetite powder with high purity at an increased rate of the process. The obtained Fe_3O_4 NPs generally have a polygonal or irregular shape, the size of which ranges from 30–80 nm with an average value of 65 nm, and each of them is connected to several neighboring particles by isthmuses.

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ПОЛУЧЕНИЕ НАНОПОРОШКА МАГНЕТИТА ВОДОРОДНЫМ ВОССТАНОВЛЕНИЕМ ГИДРОКСИДНОГО СОЕДИНЕНИЯ α-FeOOH В ИЗОТЕРМИЧЕСКИХ УСЛОВИЯХ

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В данной работе проведено изучение процесса получения нанопорошка магнетита Fe_3O_4 водородным восстановлением гидроксидного соединения α-FeOOH в изотермических условиях. Нанопорошок α-FeOOH заранее получали методом химического осаждения из водных растворов нитрата железа $Fe(NO_3)_3$ (10 мас. %) и щелочи NaOH (10 мас. %) при комнатной температуре, pH = 11, при условии непрерывного перемешивания. Процесс водородного восстановления порошка α-FeOOH в изотермических условиях проводили в трубчатой печи в интервале температур от 310 до 375 °С. Исследование кристаллической структуры и фазового состава образцов порошков выполняли методом рентгенофазового анализа. Удельную поверхность S образцов измеряли методом БЭТ по низкотемпературной адсорбции азота. Средний размер частиц D рассчитали по данным измерения величины удельной поверхности. Размерные характеристики и морфологию частиц порошков изучали методом сканирующей и просвечивающей электронной микроскопии. Расчет кинетических параметров процесса получения нанопорошка Fe_3O_4 в изотермических условиях проводили с помощью модели Грея – Веддингтона и уравнения Аррениуса. Установлено, что процесс восстановления нанопорошка α-FeOOH начинает сильно ускоряться в интервале температур от 340 до 375 °С. Константа скорости восстановления при 375 °С примерно в 2 раза больше, чем в случае восстановления при 310 °С. Величина энергии активации процесса получения нанопорошка Fe_3O_4 в изотермических условиях составила ~ 36 кДж/моль, что говорит о смешанном режиме лимитирования кинетики процесса. Показано, что временно-температурный режим восстановления гидроксидного соединения α-FeOOH с выдержкой при 340 °С в течение 3 ч позволяет получить наноразмерный порошок магнетита Fe_3O_4 с высокой чистотой при повышенной скорости процесса. Полученные наночастицы Fe_3O_4 обладают кристаллической структурой и в основном имеют многоугольную или неправильную форму, их размер составляет 30–80 нм со средним значением 65 нм, при этом каждая из них соединена с несколькими соседними частицами перешейками.

Ключевые слова: кинетика, нанопорошок магнетита, водородное восстановление, изотермические условия, степень превращения, константа скорости, энергия активации.

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