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OXIDE POWDERS PRODUCTION FROM IRON CHLORIDE

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1. Introduction

At present, iron oxide powders, particularly hematite and magnetite, are widely used in industry. They are a promising material for the production of both sorbents and catalysts [1, 2]. The magnetic properties of the materials under discussion ensure their use for instrument fabrication and as magnetic information carriers (magnetic pigments) [3]. The anodic resistance and high electrical conductivity of magnetite makes it feasible for use as electrodes in various electrochemical processes and in cathodic protection systems, as well as in the creation of electrochemical storage for electrical energy.

A number of processes for the preparation of ultrafine powders of metal oxides with advanced properties (such as nano crystallinity, narrow particle size distribution, preparation of metastable phases, etc.) have been described in the scientific literature.

A large number of works are devoted to the study of the process of obtaining ultrafine powders of iron oxides by high-temperature hydrolysis of iron nitrates to produce a nanostructured α -Fe₂O₃ powder [4]. The results of thermal decomposition of α -FeOOH with formation of ultradispersed α -iron oxide are reported in [5].

The source of raw materials for the production of iron oxide powders can be iron-containing hydrochloric acid solutions obtained in hydrometallurgy of various metals. Usage of hydrochloric acid in the processing of iron-containing products every year increases, which is confirmed by a number of works [6]. Apart from technologies for processing of ore and non-metallic raw materials, chloride solutions are formed during metal etching, processing of computer cards, etc [7].

Hydrochloric acid started to replace sulfuric acid during the etching of steel from the 1960s. This became economi-

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ABSTRACT

This article presents the results of the high-temperature hydrolysis process of ${\rm FeCl_2}\times 4{\rm H_2O}$ and the study of iron oxide powder properties produced. It is observed that the process has a topochemical character and proceeds in a diffusion zone. Conditions sufficient for practically complete decomposition of ${\rm FeCl_2}\times 4{\rm H_2O}$ are a temperature in the range of 803-903 K and a duration of 40 minutes. The powder, obtained as a result of high-temperature hydrolysis of ${\rm FeCl_2}\times 4{\rm H_2O}$, is represented by an ultradisperse powder of iron oxides of 10-100 nm in size (average -20 nm), having a basically round shape. The results of microprobe, X-ray phase analyzes, magnetic susceptibility analysis, and Mössbauer spectroscopy have revealed that the product of high-temperature hydrolysis consists of a mixture of iron oxides consisting of magnetite and hematite.

cally justified when the method for regenerating hydrochloric acid from productive solutions was developed. Thus, for example, the ferrous chloride produced in pickling solutions can be decomposed into iron oxide and HCl after evaporation and crystallization in accordance with the following equation:

$$2\text{FeCl}_2 + 2\text{H}_2\text{O} + 1/2\text{O}_2 \rightarrow \text{Fe}_2\text{O}_3 + 4\text{HCl}$$
 (1)

Hydrochloric acid is regenerated, and commodity oxides of iron are obtained as a result of the reaction [8, 9]. To obtain iron oxides and regenerate hydrochloric acid, a two-stage technology has been studied, consisting in the oxidation of FeCl₂ to FeCl₃ by air oxygen according to the reaction:

$$12\text{FeCl}_2 + 3\text{O}_2 + 12\text{HCl} \rightarrow 12\text{FeCl}_3 + 6\text{H}_2\text{O}$$
 (2)

and subsequent high-temperature hydrolysis of the resulting trivalent chloride according to the reaction [10]:

$$4\text{FeCl}_3 + 6\text{H}_2\text{O} \rightarrow 2\text{Fe}_2\text{O}_3 + 12\text{HCl}$$
 (3)

The process of high-temperature hydrolysis of iron salts and the study of the properties of powders obtained by the researchers are of great interest. However, it should be noted that the literature on the process of high-temperature hydrolysis of divalent iron chloride and the properties of the powders obtained is insignificant.

The thermodynamics of processes of high-temperature hydrolysis of $FeCl_2 \times 4H_2O$ has been considered in detail in the published papers [8, 9, 11]. The thermodynamics of the main reactions of the one-stage high-temperature hydrolysis of iron chlorides is given in **tab. 1**.

The obtained calculation results show that in the hightemperature hydrolysis of FeCl₂×4H₂O in the oxidizing atmosphere, the formation of magnetite (reaction 1), hematite

Table 1. Gibbs energy of iron chlorides hydrothermal decomposition

		Gibbs Energy ΔG° kJ / mol FeCl ₂ (FeCl ₃)					
Nº	Reaction		Temperature, K				
		473	573	673	773	873	
1	$3\text{FeCl}_2 \times 4\text{H}_2\text{O} + 0.5\text{O}_2 = \text{Fe}_3\text{O}_4 + 6\text{HCI} + 9\text{H}_2\text{O}$	-92,9	-206,3	-326,3	-454,3	-589,7	
2	2FeCl ₂ ×4H ₂ O + 0.5O ₂ = Fe ₂ O ₃ + 4HCl + 6H ₂ O	-180,8	-308,4	-433,4	-556,4	-678,0	
3	$5 \text{FeCl}_2 \times 4 \text{H}_2 \text{O} + \text{O}_2 = 2 \text{FeCl}_3 + \text{Fe}_3 \text{O}_4 + 18 \text{H}_2 \text{O} + 4 \text{HCl}$	-344,9	-637,3	-936,8	-1233,4	-1527,4	
4	$2\text{FeCl}_3 + 6\text{H}_2\text{O} = \text{Fe}_2\text{O}_3 + 3\text{H}_2\text{O} + 6\text{HCl}$	-87,0	-84,0	-96,0	-151,3	-271,6	
5	$5 \text{FeCl}_2 \times 4 \text{H}_2 \text{O} + \text{O}_2 = \text{Fe}_3 \text{O}_4 + \text{Fe}_2 \text{O}_3 + 10 \text{HCl} + 15 \text{H}_2 \text{O}$	-87,8	-165,7	-242,1	-317,3	-391,7	

(reaction 2), and FeCl $_3$ (reaction 3) is possible, which will be hydrolyzed to hematite (reaction 4). It follows that in the presence of oxygen the resultant reaction of high-temperature hydrolysis of FeCl $_2$ ×4H $_2$ O will be reaction 5, producing a mixture of powders of magnetite and hematite, hydrochloric acid and water vapor. Reaction 5 is taken as the basis for carrying out kinetic and technological studies of the process of high-temperature hydrolysis.

In connection with this, the authors of the work have set two goals:

- a) study of the mechanism, kinetics and conditions of the process of direct (single-stage) high-temperature hydrolysis of divalent ferric chloride (FeCl $_2$ ×4H $_2$ O) in an oxidizing atmosphere;
- b) study of the composition and properties of oxide iron powders obtained by high-temperature hydrolysis of $FeCl_2\times 4H_2O$.

2. Experimental Procedure

Technique of carrying out of experiments and used materials

Hydrothermal decomposition was studied on samples of crystalline hydrate $\text{FeCl}_2 \times 4\text{H}_2\text{O}$, which was obtained by reducing the solution of trivalent iron chloride with metallic scrap. After filtration, the resulting ferrous solution was evaporated and crystallized on a water bath at a temperature of 313 K. Throughout the process, in order to avoid oxidation of the solution, the crystallizer was washed with nitrogen. The obtained crystals were filtered from the mother liquor on a vacuum filter at T=313 K.

The resulting crystals of divalent iron chloride $FeCl_2 \times 4H_2O$, were sent to high-temperature hydrolysis. High-temperature hydrolysis was carried out in a plant, as shown in **fig. 1**. In the periodic regime of high-temperature hydrolysis, the dehydration of $FeCl_2 \times 4H_2O$ crystals can outstrip the interaction processes, so steam and air have been introduced into the reaction vessel to maintain a high partial vapor pressure in the system throughout the experiment.

A sample of iron chloride crystals was placed in an alundum boat in the reaction tube {3}. Both ends of the tube were closed with rubber stoppers with outlet holes for discharging steam and gases from the reaction space. A thermocouple with a quartz cover was inserted into the tube through one of the holes. Upon reaching the preselected temperature in the reaction zone, a boat with the sample was pushed under the hot junction of the thermocouple and steam from a steam generator was introduced {1}. The time of the experiment

was also recorded. In order to prevent condensation of the steam, the pipe that led from the evaporator to the reaction tube was heated.

The weight of the sample of iron chloride crystals was about 5 grams. During the experiment, the temperature in the reaction zone was maintained at a con-

stant within \pm 5°. Gases formed during the process were sent to a condenser, from which the acid solution was gathered at room temperature.

The degree of decomposition was continuously determined by the amount of hydrochloric acid formed by titrating the solution in the acid collector. Oxidized iron powders obtained during hydrolysis were subjected to an integrated validation of their properties through both XRD and microscopy.

3. Results and discussion

a) Study of the mechanism, kinetics and conditions of the high-temperature hydrolysis of divalent ferric chloride (FeCl₂×4H₂O) in an oxidizing atmosphere

The degree of decomposition of $FeCl_2 \times 4H_2O$ crystals was continuously determined by the amount of hydrochloric acid formed (**fig. 2**). An increase of $FeCl_2 \times 4H_2O$ hydrolysis temperature above 803 K has no noticeable effect on the de-



Fig. 1. Apparatus for hydrothermal decomposition of ferric chloride:

1 — steam generator; 2 — glass faucet; 3 — furnace;

4 — quartz reaction tube; 5 — acid bath or gatherer;

6 — a refrigerator; 7 — burette with NaOH solution

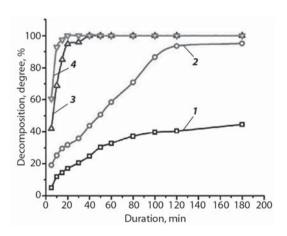


Fig. 2. The dependence between temperature and degree of the decomposition of divalent ferric chloride: 1-603 K; 2-703 K; 3-803 K; 4-903 K

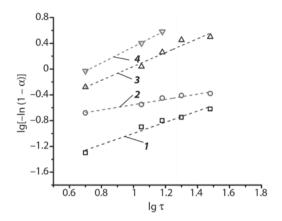


Fig. 3. An analysis of the experimental data hydrothermal decomposition of FeCl₂×4H₂O, according to the equation $\alpha = 1 - e^{-kvn}$: 1 - 603 K; 2 - 703 K; 3 - 803 K; 4 - 903 K

gree of decomposition. An almost complete decomposition of $FeCl_2 \times 4H_2O$ occurs in 40 minutes at temperatures from 803 to 903 K. Lowering the hydrolysis temperature increases the decomposition time to 180 minutes at 703 K, and at an even lower temperature of 603 K, the decomposition time is 180 minutes and the decomposition rate is only 44.46% (fig. 3).

The results were analyzed by a topokinetic Erofeev – Kolmogorov equation: $\alpha = 1 - e^{-k\tau n}$ [12]. The coordinates of $\log[-\ln(1-\alpha)]$ vs. $\log \tau$ the obtained results satisfactorily fit in a straight line, which indicates the topochemical nature of the reaction and indicates that the nucleation of the new phase and its growth proceeds on the surface of the crystals of the iron chlorides.

By the Sakovich formula, $K = nk^{1/n}$, the reaction rate constants are determined (**fig. 4**). The dependence of the rate constant of the reaction on the temperature obeys the Arrhenius equation-in the coordinates $\lg K^{-1} / T$ the results fit satisfactorily on a straight line (see fig. 4). The apparent activation energy of the high-temperature hydrolysis FeCl₂×4H₂O is E = 9.732 kJ/mol. The temperature coefficients of the reaction rate constants $\gamma = K_{t+10}/K_t$, calculated from the equation $\lg \gamma = 2E/T^2$, are: $\gamma_{603-703} = 2,13$; $\gamma_{703-803} = 2,74$; $\gamma_{803-903} = 1,35$.

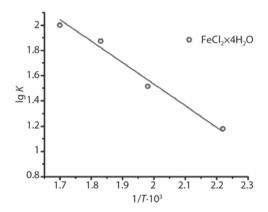


Fig. 4. Dependence of lg K reaction of high-temperature hydrolysis of $FeCl_2 \times 4H_2O$ on the reciprocal temperature

The values of the apparent activation energies and the temperature coefficients of the reaction rates show that at all studied temperatures the rate of the decomposition process is limited by the rate of supply of the steam-air mixture to the reaction zone-the process proceeds in the diffusion zone.

Technological research

According to kinetic studies, almost complete decomposition of $FeCl_2 \times 4H_2O$ is achieved at a temperature of 803-903 K in 40 min. (figure 2). Technological studies have been carried out in the same manner as the kinetic ones, the process has been carried out at a temperature of 903 K and at a duration of 40 min., the weight of the sample of $FeCl_2 \times 4H_2O$ crystals is 20 g.

The conditions for conducting technological research ensure the complete decomposition of $FeCl_2 \times 4H_2O$ crystals. The resulting powder of iron oxides was subjected to various physico-chemical analyses.

b) The study of iron oxide powder properties produced by high-temperature hydrolysis of $FeCl_2 \times 4H_2O$

Results of electron-probe analysis and transmission electron microscopy

The size and morphological features of the oxidized iron powder have been determined using a JEOL JEM-2100F transmission electron microscope (**fig. 5**). The element composition of the samples with depth resolution has been determined by the method of electron-probe X-ray microanalysis (**tab. 2**). The spectrum has been registered with an energy-dispersive spectrometer IncaX-act.

The obtained material is represented by ultradisperse powders of iron oxides of 10–100 nm fineness (average 20 nm), having a basically round shape. The atomic ratio of Fe/O in magnetite is 0.75, and in hematite it is 0.66, the atomic ratio in the obtained powder is 0.7, which suggests that the powder consists of a mixture of magnetite and hematite (see tab. 2).

X-ray diffraction results

An X-ray diffractometer DRON-3 with $Cu_{K\alpha}$ radiation, and a β -filter has been used in this work. The conditions for shooting diffraction patterns are U= 35 kV; I= 20 mA; shooting θ -2 θ ; detector at 2 deg/min. X-ray phase analysis on a

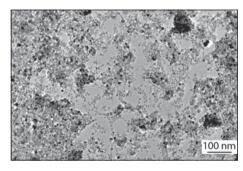


Fig. 5. A snapshot of magnetite powder obtained via hydrolysis T = 903 K

Table 2. Results of elemental analysis of powders at the temperature of high-temperature hydrolysis 903 K					
O, g	O, g∙at	Fe, g	Fe, g·at	Fe/O	
28,92	1,81	71,08	1,27	0,70	

semi-quantitative basis has been performed on the diffraction patterns of powder samples using the method of equal weights and artificial mixtures. Quantitative ratios of crystalline phases have also been determined. An interpretation of the diffractograms has been carried out using the ICDD file data: powder diffractometry database PDF2 (powder diffraction file). The hematite phase was PDF # 898104, while the magnetite phase was PDF # 870246. (fig. 6).

The results of X-ray phase analysis confirm the presence of hematite and magnetite in the resulting powders (tab. 3). Results of semi-quantitative X-ray phase analysis show that the ratio of magnetite: hematite in the powders obtained has been 62.3: 37.7 (tab. 4). The obtained composition of powders corresponds to the composition of products according to reaction 5 (see tab. 1).

Mössbauer analysis

To determine the composition of the powders and their type of crystal lattice, Mössbauer analysis has been performed (**fig. 7**, **tab. 5**). The results of the analysis show that a high-temperature hydrolysis of $FeCl_2 \times 4H_2O$ produces a polycomponent composition with a composition consisting of tetrahedral and octahedral subbarrays of three and divalent iron and alpha hematite. The obtained results correspond to reaction products in thermodynamic calculations, and correspond to the results of X-ray phase analyzes.

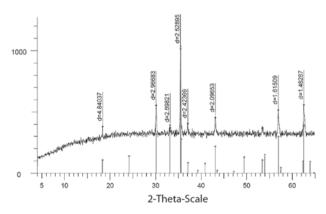


Fig. 6. Radiographs of oxidized iron powders obtained during hydrolysis T = 903 K

Table 3. Interplanar distances and phase composition $FeCl_2$ T = 903 K			
d, Å	1, %	Mineral	
4.84037	36.6	-	
2.96683	53.9	-	
2.69821	35.7	Hematite	
2.52895	100.0	Magnetite	
2.42369	39.1	-	
2.09653	43.9	-	
1.61509	50.2	-	
1.48287	54.4	_	

Table 4. Results of a semi-quantitative XRF product of high-temperature hydrolysis				
Phase	Chemical formula	Content,%		
Magnetite	Fe ₃ O ₄	62,3		
Hematite	Fe ₂ O ₃	37,7		

Table 5. Decoding to Mossbauer spectroscopy

ls, mm/s	Qs, mm/s	H, kE	S, %	Feedback
0,28	-0,01	491	25	Magnetite tetrahedral subbarray with Fe3+
0,67	-0,05	462	27	Magnetite octahedral subbarray with Fe ³⁺ and Fe ²⁺
0,66	0,09	452	13	Octahedral subbarray with Fe ³⁺ , Fe ²⁺ and with small number of elements standing in the periodic table to the left of Fe (e.g. Cr, Mn)
0,37	-0,21	517	35	α-Fe ₂ O ₃

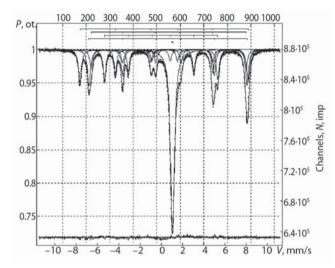


Fig. 7. The Mössbauer spectra of a sample of hightemperature hydrolysis of $FeCl_2 \times 4H_2O$ at a temperature of 903 K

Magnetic susceptibility

The magnetic analysis is performed on a SatisGeoKM-7 kappa-meter, the magnetic susceptibility of a sample obtained by high-temperature hydrolysis of $FeCl_2 \times 4H_2O$ is $1250-1358 \cdot 10^{-6}$ CI/g.

4. Conclusions

- the process of high-temperature hydrolysis of $FeCl_2 \times 4H_2O$ in a stationary layer in the presence of oxygen has a topokinetic character, the rate of the decomposition process is limited by diffusion factors.
- practically complete decomposition of FeCl₂×4H₂O is achieved at a temperature of 803–903 K.
- obtained as a result of high-temperature hydrolysis of $FeCl_2 \times 4H_2O$ at a temperature of 903 K, the product is represented by an ultradisperse powder of iron oxides of a size less than 100 nm (average 20 nm), consisting of a mixture of magnetite and hematite in a percentage ratio of 62.3% to 37.7%, which confirms that the reaction proceeds according to equation:

$$5 \text{FeCl}_2 \times 4 \text{H}_2 \text{O} + \text{O}_2 = \text{Fe}_3 \text{O}_4 + \text{Fe}_2 \text{O}_3 + 10 \text{HCl} + 15 \text{H}_2 \text{O}.$$

— Thus, as a result of high-temperature hydrolysis of the crystal hydrates $FeCl_2 \times 4H_2O$ in the presence of oxygen in the gaseous phase, stable ultrafine powders consisting of Fe_3O_4 and Fe_2O_3 with predominance of Fe_3O_4 are formed.

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SIMPLE ANALYTICAL DEPENDENCE OF ELASTIC MODULUS ON HIGH TEMPERATURES FOR SOME STEELS AND ALLOYS

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Modules of elasticity are the most important physical quantities, included in the various engineering calculations at the determining of the strength and stability of machine parts and constructions and the natural frequencies of the moving parts of machines and mechanisms. The knowledge of them is necessary for the metallurgical technological calculations. The accuracy of the determining of the values of the elastic modules and their changes due to the influence of various factors is of great interest, since the requirements for the reliability of the metallurgical and engineering products, saving in materials and reducing the weight of constructions have increased. For the investigating of the stress state of the solidifying ingots (without which it is impossible to choose the optimal technological regimes of their production) and the designing of metal equipments, the information about the values of the elastic modules and the rheological behavior of metals and alloys at high temperatures is necessary. The investigations of the influence of various factors, including temperature, on the elastic modules of metals are extremely important. The mechanical properties of metals and alloys (for example, strength, plasticity, toughness, and others) depend on both microstructure (number of phases, their dispersion, distribution, mutual arrangement) and substructure (defects of crystal structure, their density, mobility, interaction with each other and with the atoms of impurities) and are in a certain way connected with the interatomic interactions in metals. Therefore, the correlations between the mechanical properties and the elastic modules of materials are not accidental. In this paper, based on the analysis of experimental data about the dependence of the elastic modulus of elasticity (young modulus) for various steels and alloys, the analytical dependence of the elastic modulus on temperature for the heat-resistant steels and alloys, used in the production of steel sheets and steel pipes of large diameter for main pipelines, is obtained. The results of investigation can be widely used in the metallurgical and machine-building plants.

1. Elastic properties of steels

To describe the elastic properties of the isotropic and polycrystalline (quasi-isotropic) bodies [1-37], we use the normal elastic modulus (young modulus) E, the shear modulus G, the all-round compression modulus (the volume compression modulus) K, the compressibility $\alpha = 1/K$ and the Poisson ratio u, which are related to each other by known relations of the ideal elasticity theory:

$$E = \frac{18KG}{6K + 2G}, \quad \mu = \frac{3K - 2G}{6K + 2G}.$$

The reverse dependencies have the form
$$K = \frac{E}{3(1-2\mu)}$$
, $G = \frac{E}{2(1+\mu)}$.

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