

This paper reports the results of studying the application of a ferritization method for the integrated purification of used etching solutions. A feature of this work is the use of energy-saving activation of the process by alternating magnetic fields. Its advantages are shown in comparison with traditional thermal activation. The influence of magnetic induction amplitude and key technological parameters of ferritization on the quality of cleaning an etching solution has been studied. The qualitative and quantitative composition of sediments obtained after the ferritization of etching solutions was investigated.

Used etching solutions are large-tonnage waste of industrial enterprises. They contain harmful pollutants that have a detrimental effect on the environment. It is promising to treat these solutions in order to obtain valuable commodity products.

It is established that with an optimal value of magnetic induction amplitude of 0.1 Tl, the degree of extraction of iron ions from the solution reaches a value of 99.99 %. The best values for the main technological parameters of the process have been determined: the concentration of iron ions in the reaction mixture is 6.6 g/dm³; pH, 11.5; the duration of ferritization is 15 min. The residual concentration of iron ions in purified solutions does not exceed 0.3 mg/dm³. Thus, according to the norms of current standards, they can be reused in production. Comparative analysis indicates the advantages of electromagnetic activation of the reaction mixture. The phases of magnetite Fe₃O₄ and iron monohydrate δ – FeOOH were detected by the method of X-ray phase analysis in ferritization sediments. It is established that with an amplitude of 0.1 Tl, the sediment contains only magnetite. The study's results indicate the possibility of further use of sediments for the manufacture of important ferromagnetic substances.

The application of the improved ferritization process in production will achieve less energy consumption compared to well-known processing technologies

Keywords: etching solutions, waste processing, ferritization, electromagnetic activation, ferrite sediments, magnetite

DETERMINING THE RATIONAL PARAMETERS FOR PROCESSING SPENT ETCHING SOLUTIONS BY FERRITIZATION USING ALTERNATING MAGNETIC FIELDS

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1. Introduction

Most countries in the world pay considerable attention to reducing emissions of harmful substances into the environment. Industrial enterprises, and especially galvanic production, are one of the most dangerous sources of pollution as a result of the formation of significant amounts of toxic waste containing heavy metal compounds. Sources of contamination in galvanic production are not only washing wastewater but also used highly-concentrated solutions. By volume, these liquid wastes account for 10÷15 % of the total amount of wastewater, and, in terms of pollution by heavy metals, reach 70 % [1]. It should be noted that the volume of solutions after metal etching operations accounts for 85 % of the total volume of highly-concentrated wastewater in galvanic production [2].

Etching solutions are formed as a result of treating the surface of metal products with acids in order to clean them from sinter and rust [3]. Every year, hundreds of millions of tons of spent highly-concentrated etching solutions are formed at industrial enterprises in Eastern Europe, which are subject to disposal and processing [4]. Waste acid etching solution, depending on the composition and processing conditions of steel parts, contains up to 300 g/dm³ FeSO₄ [5]. As a result, with insufficiently treated galvanic wastewater, thousands of tons of toxic iron-containing compounds enter water bodies every year. Iron ions have high migratory mobility, the ability to accumulate in living organisms and cause various physiological disorders [6]. In addition, etching solutions belong to the class of danger 2–3; their neutralization and disposal requires significant costs [7].

Therefore, it is a relevant task to improve and devise effective methods for processing etching toxic solutions with the disposal of heavy metals. The development and implementation of new technologies at industrial enterprises will reduce the cost of storing hazardous waste, increase the efficiency of production through waste disposal, and reduce the burden on the ecosystem.

2. Literature review and problem statement

There are two main directions of processing waste solutions of etching of steel surfaces: the regeneration of these solutions and the extraction of iron ions in the form of solid-phase chemically resistant compounds [8]. If the above methods of processing are not provided for in production, these wastes are sent to specialized enterprises for their further disposal.

There is a well-known technique of regeneration of spent etching solutions, which is based on their cooling in an isohydric-type crystallizer [9]. Crystals of ferrum sulfate are separated while the regenerated solution is returned to the technological process. However, this regeneration technique [9] requires the use of multi-stage processes, high electricity consumption, capital investments, and careful monitoring of the purity of the resulting solution.

There is also a technique to process etching solutions by heat treatment at a temperature exceeding 900 °C using tetraborate and sodium silicate [10]. As a result of this treatment, chemically resistant products are formed that can be used for further disposal. The disadvantages of this method are significant energy consumption and the use of special technological equipment.

There is a known electro dialysis method for processing spent etching solutions with the production of metallic iron and sulfuric acid solution [11]. But the technical and economic indicators of this method were unsatisfactory for use in the industry. The method of regeneration of etching solutions with double electrolysis with the formation of mercury amalgam [12] has not been widespread in production due to its significant energy costs.

One of the methods of processing etching solutions involves the use of ion exchange sorption [13]. However, this process requires the use of a double number of reagents for the regeneration of ion exchange resins compared to the mass of the components removed in this process. In addition, the sorption process is significantly affected by the presence of organic impurities in spent solutions.

Among the numerous methods of processing etching solutions, the most common is the reagent method [14]. Iron ions are deposited in the form of low-soluble sediment when adding NaOH, Na₂CO₃, or cheaper Ca(OH)₂. However, the use of Ca(OH)₂ in the cleaning process contributes to the formation of a large amount of sludge, which is not subject to further disposal. In addition, when storing sludge in open landfills, due to the dissolution of hydroxides under the influence of precipitation, iron ions leach into the soil and reservoirs.

More promising is the method of processing etching solutions with the production of oxide compounds of iron – magnetite Fe₃O₄, hematite α – Fe₂O₃, maghemite γ – Fe₂O₃. These oxide compounds have a crystalline structure and are quickly sedimented during processing, and their volume and humidity are much lower than that of ferrum hydrox-

ides [14]. One of the promising methods of such processing of etching solutions is the method of hydro-phase ferritization [15]. The essence of this method is to create conditions in spent sulfuric acid solutions that contribute to the rapid formation of dispersed chemically resistant substances with magnetic properties [16]. The course of the process of hydro-phase ferritization depends on the following technological parameters: the concentration of iron ions, the pH value, the temperature, process duration, and oxidant flow rate. The disadvantages of hydro-phase ferritization are high energy consumption since it is carried out at temperatures above 75 °C [17]. Paper [18] shows that an alternative to thermal hydro-phase ferritization is the activation of the process by alternating magnetic fields (AMF activation) at room temperatures. The studies were conducted at a high amplitude of magnetic induction (0.3 Tl), which leads to significant cost of electrical energy to activate the reaction mixture during ferritization. This suggests that it is advisable to conduct research aimed at reducing the amplitude of magnetic induction with ensuring the high-quality cleaning of solutions from heavy metal ions. Such activation of the reaction mixture will significantly improve the energy efficiency of the ferritization process.

3. The aim and objectives of the study

The aim of this work is to establish the best technological parameters of ferritization processing of sulfuric acid etching solutions using electromagnetic activation. This will make it possible to improve the energy efficiency of the process, enhance the quality of cleaning spent solutions and obtain sediments capable of disposal.

To accomplish the aim, the following tasks have been set:

- to experimentally determine the effect of the amplitude of magnetic induction in the process of ferritization using the AMF activation on the degree of extraction of iron ions from a spent etching solution;
- to establish the dependence of the main technological characteristics of the ferritization process on the efficiency of extraction of iron ions and compare the obtained values with different techniques for activating the reaction mixture;
- to investigate the phase composition of the sediments of ferritization of spent etching solutions and the possibility of their further disposal.

4. The study materials and methods

The object of this study was the spent sulfuric acid solution from etching steel parts obtained at one of the leading industrial enterprises in Ukraine. The main characteristics of this waste from galvanic production are given in Table 1.

Table 1

Composition of spent sulfuric acid etching solutions

FeSO ₄ , g/dm ³	H ₂ SO ₄ , g/dm ³	pH	Suspended substances, g/dm ³	Color
126.5	21.0	1.41	0.35	brown

The ferritization process was investigated at the initial concentrations of iron ions that varied within 6.6÷46.6 g/dm³. To achieve certain values of concentrations, the etching solution

was diluted with technical water. The process of ferritization of solutions was studied in the pH interval of 8.5÷11.5. We adjusted pH with a 25 % solution of sodium hydroxide. The duration of the ferritization process varied from 5 to 30 minutes. The partial oxidation of Fe²⁺ to Fe³⁺ was carried out by the aeration of the reaction mixture with air oxygen at a speed of 0.15 m³/h.

The process of ferritization processing of spent etching solutions was investigated, both with the traditional thermal activation of the reaction mixture at a temperature of 75 °C [19] and using the AMF activation of the reaction mixture at room temperature. The scheme of the installation with the working volume of the reactor of 1 dm³ is shown in Fig. 1. To create AMF, a controlled rectangular pulse generator was designed, based on the ATmega328p microcontroller board (France). The output voltage of pulses at 8 digital outputs of the controller board was 5 V, the maximum current was 20 mA.

We changed the amplitude of magnetic induction in the working area of the installation by the UTP3305C power supply system (Ukraine) through the RS-232 interface in the ranges of values 0.02...0.1 Tl. Other parameters of the AMF generation experiment were constant: pulse frequency, 1 Hz; a period between the pulses, 100 ms; pulse duration, 1000 ms.

The residual concentrations of iron ions after processing of spent etching solutions were determined at the DR3900 spectrophotometer (USA). The pH value of the reaction mixture in the process of ferritization was controlled at the pH-meter PL- 700AL (Poland).

Dehydrated at the CM-5 centrifuge (Ukraine) with a separation factor of 3600 for 2 minutes, the ferrite sediment was dried at a temperature of 105 °C during 24 hours in the electric drying chamber SNOL 67/350 (Ukraine). The phase analysis of the powder of dried ferrite sediments was carried out by X-ray diffraction under a step-by-step mode with Cu-K α radiation at the Ultima IV diffractometer (Japan). Shooting was carried out in the interval of angles 2 θ 6...65° in scanning increments of 0.05° and an exposure time at point 2 s.

The evaluation of variance and boundaries of errors in the experiments to determine the residual concentrations of iron ions after the ferritization process was carried out according to the procedure given in [20] at a confidence probability of 0.95.

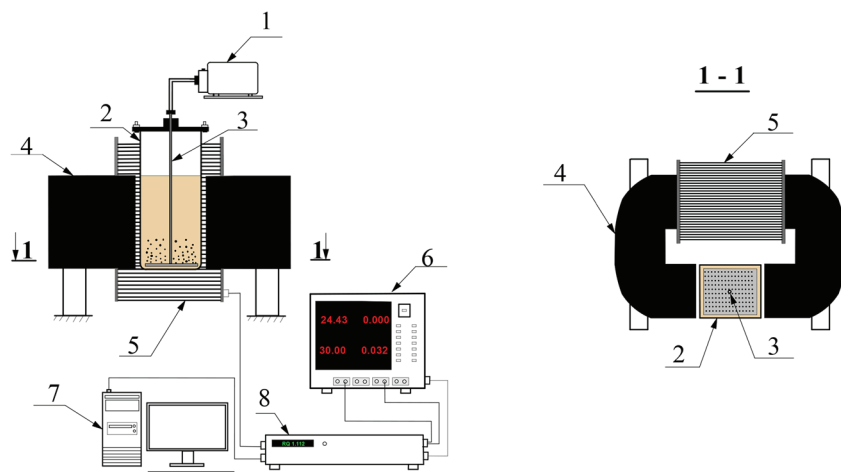


Fig. 1. Laboratory installation for processing etching solutions by ferritization: a - general view; b - cross-section 1 - 1; 1 - compressor; 2 - container with reaction mixture; 3 - aerator; 4 - steel core; 5 - electromagnetic coil; 6 - power supply; 7 - computer; 8 - electronic unit housing

5. Results of studying the energy-saving processing of spent sulfuric acid solutions from etching steel surfaces

5.1. Determining the effect of magnetic induction amplitude when activating the reaction mixture on the degree of extraction of iron ions

The focus of this stage of research was one of the most important parameters for the generation of pulse discharges in the process of AMF activation of the reaction mixture – the amplitude of magnetic induction.

The results of studying the effect of magnetic induction amplitude when activating the reaction mixture on the effectiveness of extraction of iron ions by ferritization are given in Table 2. The pH value of the starting reaction mixture in these experiments was 11.5; the ferritization process lasted for 15 minutes.

Table 2

Efficiency of extraction of iron ions from spent etching solutions by ferritization using AMF activation

Experiment series No.	Amplitude of magnetic induction, Tl	Iron ion concentration, mg/dm ³		
		Before purification	After purification	Extraction degree (α), %
1	0.02	16600	1.38	99.9916
2	0.04		1.32	99.9920
3	0.06		1.27	99.9924
4	0.08		1.22	99.9926
5	0.1		0.78	99.9953

Our analysis of Table 2 indicates that the residual concentrations of iron after ferritization are in the range of 0.78÷1.38 mg/dm³. It should be noted that an increase in the value of the amplitude of magnetic induction from 0.02 to 0.1 Tl leads to a decrease in the residual concentration of iron ions in the purified solution.

It was established that after ferritization, solutions were obtained that meet the requirements for water to be discharged to centralized drainage systems in terms of the maximum permissible concentrations of iron ions – 3 mg/dm³.

Given this, at the next stage of research, it is advisable to solve the task of achieving the quality of cleaning the solution for its reuse in production in accordance with the requirements of the current standards for iron ions – 0.3 mg/dm³. To do this, it was necessary to work out the technological parameters of the ferritization process to achieve the set requirements.

5.2. Determining the main technological parameters of the ferritization process

Studies have been conducted on the influence of key parameters of the ferritization process – the pH value, the duration of the ferritization process, and the initial concentration of iron ions on the degree of extraction of iron ions. Our data on the ferritization process using AMF were compared with those corresponding to traditional thermal activation. The results of ferritization experiments at different pH values of the reaction mixture are shown in Fig. 2, a. In this series of exper-

iments, the following remained constant: the concentration of iron ions in the reaction mixture, 16.6 g/dm³; the duration of the process, 15 minutes. Based on studying the process of ferritization using the AMF activation, the amplitude of the magnetic field of 0.1 Tl was selected.

The resulting values of the residual concentrations of iron ions in the purified solution (Fig. 2, a) indicate that the degree of purification of solutions increases with an increase in the pH value. At a pH value of 11.5, the lowest concentration of iron ions in a purified solution of 0.78 mg/dm³ is achieved. This quality of the treated solution meets the requirements for wastewater, to be discharged to the centralized drainage system.

The results of our experiments on the effectiveness of extraction of iron ions at different durations of the ferritization

process are shown in Fig. 2, b. The pH value of the reaction mixture in this series of experiments was 11.5. The results of the research that are shown in Fig. 2, b demonstrate that the residual content of iron ions decreases with an increase in the duration of the processing process of the solution and reaches a value of 0.57 mg/dm³ after 30 minutes of ferritization.

It was also important to study the dependence of the effectiveness of extracting iron ions on their original concentration in the reaction mixture (Fig. 2, c). During these experiments, the pH value of 11.5 and the process time of 15 minutes remained constant. The concentration of iron ions in the reaction mixture varied between 6.6÷46.6 g/dm³.

The dependence curves (Fig. 2, a–c) were fitted with the R² probability of approximation, which varies in the range from 0.996 to 0.999.

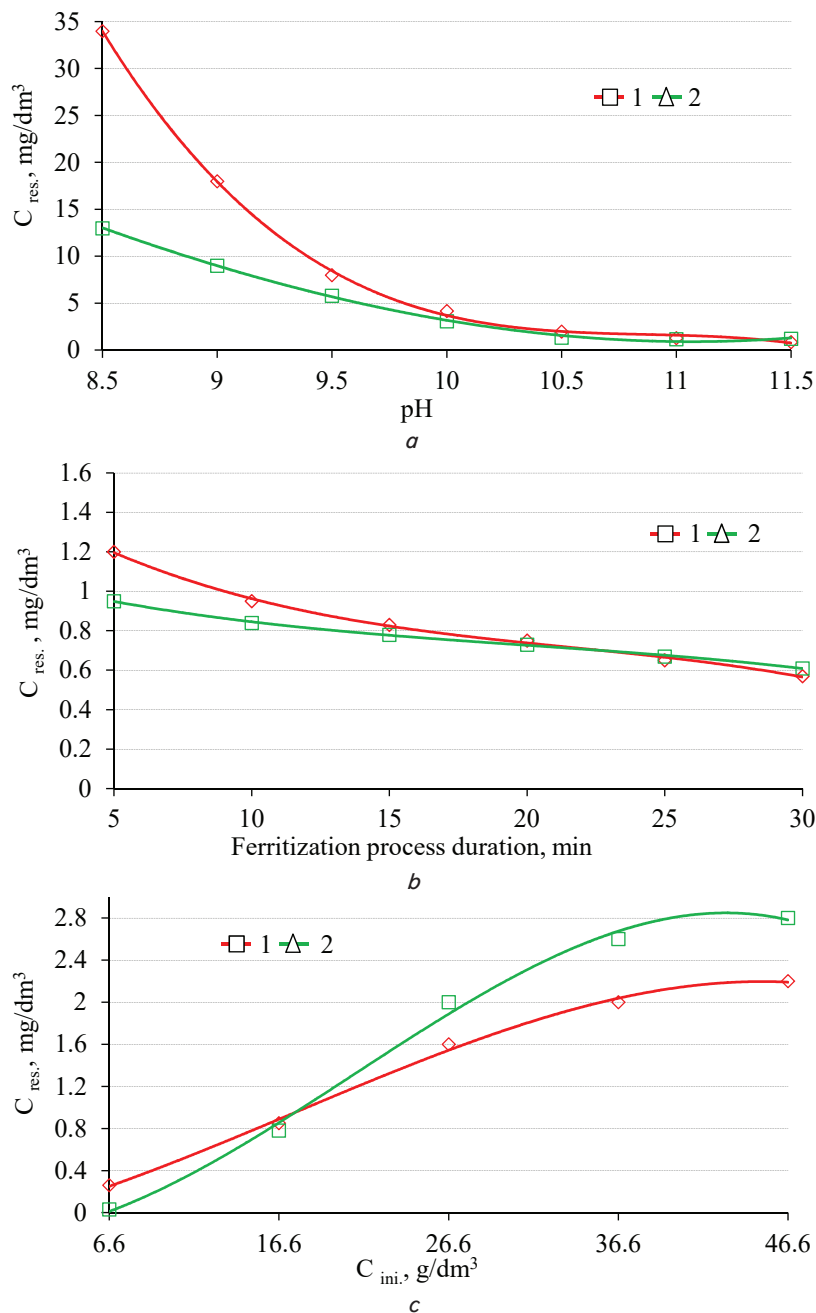


Fig. 2. Dependence of residual concentrations C_{res} of iron ions in the purified solution on: a – the pH value; b – the duration of the ferritization process; c – the initial concentration of iron ions C_{ini} in the reaction mixture; activation: 1 – thermal; 2 – by alternating magnetic fields

Table 3

Quantitative phase composition of ferrite sediments

Sediment sample No.	The method of activating the reaction mixture during ferritization	Amplitude of magnetic induction, Tl	Phase content, %	
			Fe ₃ O ₄	δ – FeOOH
1	Thermal	–	100.0	–
2	AMF	0.1	100.0	–
3		0.06	89.9	10.1
4		0.02	81.0	19.0

5.3. Investigating the phase composition of ferrite sediments

In the process of ferritization, with different techniques of its activation, a dispersed suspension is formed in the reaction mixture, which subsequently crystallizes with the formation of mostly dense ferrite structures. The qualitative and quantitative composition of sediments of ferritization of the reaction mixture with magnetic induction amplitude of 0.02–0.1 Tl was investigated. The quality of cleaning the solution was compared with the appropriate one for the ferritization with thermal activation. As one can see from Fig. 3, the sample of the sediment, which is obtained at the amplitude of magnetic induction of 0.1 Tl has a higher degree of crystallinity of the structure.

In the studied samples, the ferromagnetic phases of magnetite Fe₃O₄ and the monohydrate of iron δ – FeOOH with lattice parameters 8.36 and 2.95 Å, respectively, were identified.

The results of the quantitative phase composition of sediment samples (Table 3) showed that an increase in the amplitude of magnetic induction from 0.02 to 0.1 Tl leads to an increase in the volume of Fe₃O₄ from 81 to 100 %. This, in turn, reduces the volume of phase δ – FeOOH.

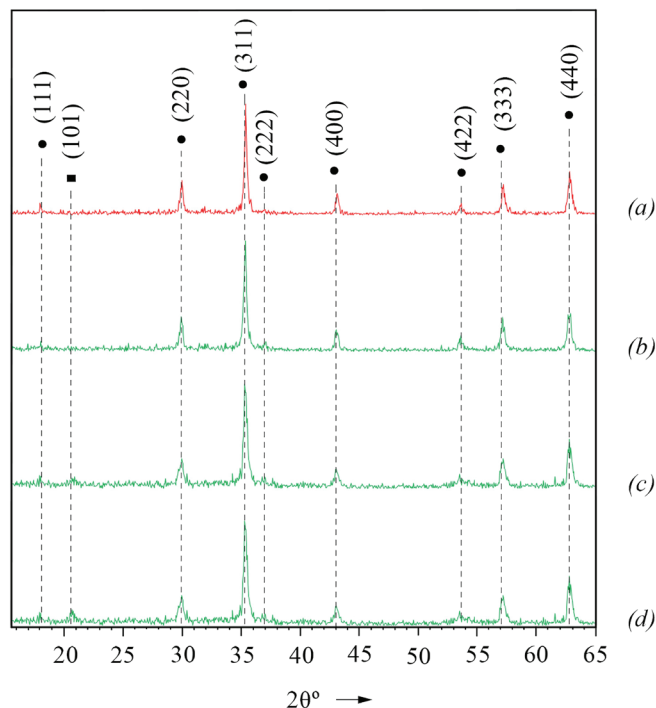


Fig. 3. Diffractograms of ferritization sediments, which are obtained by:

a – thermal; *b–d* – activation via alternating magnetic fields; magnetic induction amplitude: *b* – 0.1; *c* – 0.06; *d* – 0.02 Tl; ● – Fe₃O₄; ■ – δ – FeOOH

It should be noted that the qualitative and quantitative composition of the sediment obtained at the amplitude of magnetic induction of 0.1 Tl is identical to the sample from the thermal activation of ferritization.

6. Discussion of results of processing spent etching solutions by ferritization method

The improved technology of ferritization processing of spent etching solutions has been investigated. The analysis of our experiments indicates the prospects of energy-saving activation of the process of ferritization by AMF. As a result of the experiments on choosing the optimal values for the amplitude of magnetic induction in the working zone of the ferritization plant, it was found that the residual concentrations of iron ions decrease with an increase in the value of the amplitude of magnetic induction (Table 2). This is likely due to the fact that with an increase in the amplitude of magnetic induction, the activation energy of the ferritization process decreases. With a magnetic induction amplitude of 0.1 Tl, the degree of extraction of iron ions is 99.99 %. The identified patterns of processing of spent solutions of AMF activation suggest that a further increase in the amplitude of magnetic induction does not significantly affect the degree of extraction of iron ions.

The influence of the most important technological parameters of the ferritization process has been determined – the pH value, the duration of the ferritization process, and the initial concentration of iron ions on the quality of cleaning spent etching solutions. A comparative analysis of the use of various activation techniques of the reaction mixture in the process of ferritization was carried out. As a result of our studies (Fig. 2, *a*), it was found that an increase in pH from 8.5 to 11.5 of the reaction mixture significantly affects the degree of extraction of iron ions for both ferritization processes. The residual concentrations of these ions have close values with both techniques of activating the reaction mixture: 0.85 and 0.78 mg/dm³ for thermal and AMF, respectively. This is due to the fact that with the growth of pH, the predominant role is played not by the sorption of ions but the crystallization of dispersed compounds of heavy metals on the surface of ferromagnetic particles [21]. The best condition for ferritization with respect to the pH of the reaction mixture should be a value of 11.5 since further increase in pH does not affect the quality of water purification but only leads to the consumption of an excessive volume of reagents.

The influence of the duration of the ferritization process on the degree of extraction of iron ions has also been determined. When carrying out the process for 15 minutes at the pH of the reaction mixture of 11.5, the residual concentrations of these ions reach values of less than 0.85 mg/dm³ with both activation techniques. With an increase in the duration of the process, the quality of purified water almost does not change (Fig. 2, *b*). Thus, it can be assumed that mainly within 15 minutes of the ferritization process with the aeration of the reaction mixture with air oxygen, intermediate compounds are destroyed and transferred to chemically resistant ferrite phases.

As a result of our experiments, it was found that the initial concentration of 6.6 g/dm^3 of iron ions in the solution is reduced to values of 0.26 and 0.03 mg/dm^3 with thermal and AMF activation of ferritization, respectively (Fig. 2, c). It should be noted that the residual content of iron ions in the purified solution with AMP activation is much lower compared to thermal. This can be explained by the fact that at high initial concentrations of iron ions ($\geq 20 \text{ g/dm}^3$), the use of AMP with the proposed characteristics is not enough to effectively initiate a reaction with the formation of ferrites with a dense crystalline structure. Therefore, in the process of such ferritization, the formation of intermediate, less chemically resistant solid-phase compounds largely occurs. It should be noted that at the initial concentration of metal ions of 6.6 g/dm^3 , the quality of the purified solution, which has a content of iron ions, meets the requirements for water regarding its use in galvanic production.

The results of the structural studies of ferritization sediments are well consistent with the research data on the main technological parameters of ferritization and allow for a more complete understanding of the processes that occur during ferritization. Analysis of these studies suggests (Fig. 3, Table 3) that the samples of sediments obtained during the AMF activation of the reaction mixture contain a phase of magnetite Fe_3O_4 , which has ferromagnetic properties [22]. It should be noted that in addition to magnetite, the sediment, which is obtained by activating the mixture with the amplitude of magnetic induction in the range from 0.02 to 0.06 Tl , contains an intermediate solid phase product of the ferritization reaction – iron monohydrate $\delta - \text{FeOOH}$. This phase is less stable than Fe_3O_4 but also has magnetic properties [23]. The results of our studies also demonstrate that an increase in the amplitude of magnetic induction to 0.1 Tl leads to the formation of a crystalline structure of the sediment, which contains only one phase – magnetite Fe_3O_4 . The resulting magnetite powder can be used for the manufacture of catalysts for various chemical processes [24], the production of cast metal products [25], flexible thermal conductive systems [26], fire extinguishing systems [27], building materials [28], binders [29], and others.

A feature of the proposed method of processing the etching solution is the AMF activation of the ferritization process. At the same time, the energy efficiency of the process of treating spent solutions is significantly increased since electricity consumption decreases by more than 40% compared to thermal activation of the process [18]. The results of this work strongly suggest that AMF activation does not impair the degree of extraction of iron ions from the reaction mixture compared to traditional thermal activation. In addition, such energy-saving activation leads to the formation of mainly magnetite sediment, which is easily disposed of.

Our study of AMF activation was limited to the amplitude of magnetic induction. Given this, in the future, we consider it expedient to explore the impact of other regime characteristics of the generation of magnetic fields in the

process of ferritization to improve the quality of processing of etching solutions.

7. Conclusions

1. As a result of our experimental studies, the prospects of processing spent sulfuric acid etching solutions by ferritization with AMF activation of the reaction mixture have been shown. It is established that when using a magnetic induction amplitude from 0.02 to 0.1 Tl , the residual concentrations of iron ions are in the range of $1.38-0.78 \text{ mg/dm}^3$, which corresponds to the degree of purification of solutions of 99.99% . The use of an optimal magnetic induction amplitude value of 0.1 Tl will make it possible to achieve lower energy costs compared to known technologies when implementing an improved ferritization process in production.

2. We have established the dependences between the main technological parameters of energy-saving ferritization of spent etching solutions (the pH value of the reaction mixture, the concentration of iron ions in it, the duration of the process) and the degree of extraction of heavy metal ions. It is found that the best quality of cleaning spent solutions is achieved with the use of AMF activation at the following values of technological parameters: pH, 11.5 ; duration of the ferritization process, 15 min ; initial concentration of iron ions, 6.6 g/dm^3 . In this case, the residual concentration of iron ions in purified solutions does not exceed 0.3 mg/dm^3 , which allows them to be reused in galvanic production in accordance with the norms of current standards for iron ions. A comparative analysis of the use of various methods of activation of the reaction mixture has been performed. It is established that the above technological parameters could achieve the appropriate quality of the purified solution when using thermal activation as well.

3. Based on the data of X-ray phase analysis, the qualitative and quantitative composition of sediments obtained as a result of ferritization of spent etching solutions at different values of magnetic induction amplitude is determined. In these sediments, we identified the phases of magnetite Fe_3O_4 and monohydrate of iron $\delta - \text{FeOOH}$. It is established that an increase in the amplitude of magnetic induction during the activation of the reaction mixture by AMF leads to an increase in the Fe_3O_4 phase in the sediments. With an amplitude of 0.1 Tl , the sediment of ferritization is characterized by a maximum content of magnetite. The results of our study indicate the prospects for the use of ferritization sediments for the manufacture of valuable commodity products.

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