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This paper considers prospects for increasing the level of environmental safety of industrial enterprises as a result of the implementation of resource-saving technology of processing galvanic sludge using the ferritization method. The effectiveness of the use of electromagnetic pulse discharges for resource-saving activation of the ferritization process with the extraction of heavy metal ions from sludge (Fe, Ni, Cu, Zn) has been confirmed. The influence of key parameters of the process such as the pH value of the reaction mixture and the initial concentrations of metals in the solution on the quality of processing galvanic sludge by ferritization has been experimentally investigated. It was determined that with an increase in the pH value from 8.5 to 10.5 the residual concentrations of metal ions decrease to the values of 0.1+0.25 mg/dm<sup>3</sup> regardless of the total initial concentrations. It has been established that the technique of electromagnetic pulse activation ensures an adequate degree of extraction of metal ions of 99.9 %; it also has indisputable energy advantages compared to the thermal method: energy costs are reduced by more than 60 %. That indicates the suitability of purified water for reuse in galvanic production in terms of the requirements for the content of heavy metal ions in it. In addition, the structural studies of ferritization sediment samples have been carried out. The sediment is characterized by the maximum content of crystalline ferromagnetic phases of ferrite. It was established that an increase in the pH of the initial reaction mixture leads to an increase in the ferrite phase in sedimentation: at pH=10.5, phases were detected, which are characterized by a maximum ferrite content (exceeding 76 %). The proposed resource-saving ferritization process prevents environmental pollution, ensures efficient and rational utilization of raw materials and energy in the industry; it also makes it possible to obtain commodity products from industrial waste Keywords: ferritization, galvanic sludge, heavy metals,

ferrite sediment, electromagnetic pulse discharges

UDC 628.349+628.386 DOI: 10.15587/1729-4061.2021.239102

## DETERMINATION OF INFLUENCE OF pH ON REACTION MIXTURE OF FERRITATION PROCESS WITH ELECTROMAGNETIC PULSE ACTIVATION ON THE PROCESSING OF GALVANIC SLUDGE

Gennadii Kochetov Corresponding author Doctor of Technical Sciences, Professor Department of Chemistry\* E-mail: gkochetov@gmail.com Dmitry Samchenko PhD, Senior Researcher Scientific Research Department\* Tetiana Arhatenko PhD, Associate Professor Department of Water Supply and Drainage\* \*Kyiv National University of Construction and Architecture Povitroflotskyi ave., 31, Kyiv, Ukraine, 03037

Received date 07.07.2021 Accepted date 17.08.2021 Published date 25.08.2021 How to Cite: Kochetov, G., Samchenko, D., Arhatenko, T. (2021). Determining the effect of reaction mixture pH on ferritization process involving electromagnetic pulse activation on galvanic sludge processing. Eastern-European Journal of Enterprise Technologies, 4 (10 (112)), 24–30. doi: https://doi.org/10.15587/1729-4061.2021.239102

### 1. Introduction

The problem of man-made pollution is increasingly exacerbated every year and is becoming global. One of the serious environmental problems is the formation and accumulation of sludge as a result of the most common treatment of galvanic production wastewater by alkaline reagents. Galvanic sludge belongs to the II-III hazard class, it contains hydroxides of heavy metals (nickel, copper, zinc, iron, etc.) [1]. Every year in Ukraine, industrial enterprises accumulate about 1 million tons of these toxic wastes, which are practically non-neutralized and recycled. In general, in Eastern Europe, the volume of these wastes reaches 15 million tons [2], When stored in industrial areas within the city zone, such sludge is a source of high man-made environmental impact. This, in turn, requires effective management of these wastes due to potential threats to the migration of heavy metal ions to the environment [3]. In addition, the processing of galvanic sludge, followed by burying it at special landfills, is unprofitable. Therefore, there is a task to devise effective methods for disposing of waste containing valuable compounds of heavy metals as the final stage of waste management in galvanic production. The reuse of materials removed from sludge could allow an enterprise to reduce the fee for the storage of hazardous waste, receive additional economic benefits from the sale of new products, and reduce the load on the ecosystem [4]. Therefore, it is a relevant task to conduct studies aimed at removing toxic heavy metals from galvanic sludge enabling the minimization of formed sediment and the rational utilization of water, raw materials, and energy in the galvanic production system.

## 2. Literature review and problem statement

Current techniques for extracting valuable components from hydroxide sludge of galvanic production are economically and environmentally inefficient. They require the use of multistage processes using a significant number of chemical reagents, electricity, and require significant capital investments. For these reasons, there have been publications recently about a new direction in solving the task of processing gal-

vanic sludge. It implies its chemical stabilization, as a result of which harmless or low-toxic compounds of heavy metals of IV hazard class are formed. They can be stored in open areas without the threat of environmental pollution. Such compounds include ferrites, which, in addition to high chemical stability, have valuable magnetic properties and are capable of eco-protection of living organisms from electromagnetic radiation [5]. Thus, to address the above issues, the development of technology for processing galvanic sludge using energy-saving ferritization is important and relevant. This method is aimed at removing toxic heavy metals from sludge with the minimization of formed sediment and the rational utilization of water, raw materials, and energy in the galvanic production system.

The main directions in solving the issue related to sludge are associated with using it as an additive in the production of various building materials: concrete [6], expanded clay [7], ceramic articles [8], pigments, catalysts [9], etc. These techniques are environmentally safer than taking the sludge to non-specialized landfills or to unauthorized storage locations. However, no thorough analysis was carried out regarding the sanitary and chemical safety of both products with additives of galvanic sludge and the technology of their production, which is important for industrial implementation [10]. One of the known ways to effectively process and neutralize pasty galvanic sludge is to extract toxic and valuable heavy metals from it when transferring them to the liquid phase [11]. To this end, it is advisable to dissolve galvanic sludge in sulfuric acid, followed by the extraction of heavy metal ions from it [12]. The disadvantages of this technique include the use of acid, which requires increased safety when handling it at facilities for the processing of galvanic sludge.

Promising is the resource-saving dissolution of galvanic sludge in the liquid acid waste of industrial production, followed by the production of persistent ferromagnetic sediments as a result of the process of hydrophase ferritization [13]. The process of ferritization occurs in an alkaline medium in the presence of  $Fe^{2+}$  ions in the solution and bubbling with an oxidizing agent (usually oxygenated air). At the same time, as a result of the oxidation of the ions of the bivalent ferrum with oxygen, heavy metal ferrite is formed. The results of a study on the purification of waste technological solutions by this method are reported in [14]. The disadvantage of existing technologies based on hydrophase ferritization is the execution of the process at temperatures above 75 °C, which requires significant energy costs. The course of the ferritization process is significantly influenced by the ratio of the concentration of ferrum ions to the total concentration of other metal ions removed from the solution [15], the pH value [16], the temperature [17], and the duration of the phase formation process [18]. It should be noted that works [15–18] considered the treatment of wastewater from copper lines, nickeling, and galvanizing with ferritization but there is no information about the possibility of processing hydroxide electroplating sludge using this technology. In addition, a large amount of reagent, ferrum sulfate [19], and a significant volume of tap water are used for dilution of highly concentrated electrolytes [20] to purify wastewater by ferritization. This disadvantage is absent in the technology of processing galvanic sludge with the involvement of ferrum-containing liquid industrial waste. Paper [21] shows that an alternative to hydrophase thermal ferritization is an activation of the process by electromagnetic pulse discharges at a process temperature of about 20 °C. Moreover, this method can be used both for the purification of highly concentrated wastewater and the processing of galvanic sludge.

However, unresolved issues remain in those studies, related to the possibility of processing hydroxide galvanic sludge with the use of a ferritization method. In addition, the effect of the pH of the reaction mixture on the degree of extraction of heavy metal ions and the structural characteristics of ferritization sediment has not been sufficiently studied. All this gives reason to assert that it is advisable to conduct research on the cost-effective processing of sludge by ferritization, which ensures the disposal of toxic heavy metals with the formation of environmentally safe and valuable ferrite compounds.

## 3. The aim and objectives of the study

The purpose of this work is to determine the effect of the pH of solutions on the quality of two-stage ferritization processing of galvanic sludge using electromagnetic pulse activation of the reaction mixture. This would make it possible to establish the optimal values for one of the key technological parameters of the process.

To accomplish the aim, the following tasks have been set: – to investigate the conditions for dissolving galvanic sludge in the liquid wastes from galvanic production – sulfuric acid etching solutions;

 to experimentally determine the degree of extraction of heavy metal ions from a solution of galvanic sludge by ferritization at different initial pH values of the liquid phase;

 to investigate the structural properties of ferritized sediments, which are obtained when processing galvanic sludge.

#### 4. The study materials and methods

The object of research was sludge from galvanic production. Based on generalized information on the processing of galvanic waste, a scientific hypothesis was put forward regarding the possibility of resource-saving and environmentally safe processing of toxic galvanic waste using a ferritization method.

The starting raw material was a typical waste of the reagent cleaning of spent electrolyte in galvanic production – pasty sludge, which has pH=9.81 and a moisture content of 93 %. To dissolve the sludge, we used the waste sulfuric acid etching solution for steel with pH=1.45. To comply with the most acceptable conditions for ferritization, the etching solution was diluted with technical water until the ratio of ferrum ion concentrations to the total concentration of other heavy metals ions was 4/1. We adjusted the pH value using a 25 % sodium hydroxide solution. Partial oxidation of Fe<sup>2+</sup> in Fe<sup>3+</sup> was carried out by aeration of the reaction mixture with oxygen air at a speed of 0.15 m<sup>3</sup>/h. Samples of spent electrolytes were selected at one of the leading aviation enterprises in Ukraine. The main characteristics of that galvanic waste are given in work [22].

Ferritization was carried out under laboratory conditions in the reactor with a working volume of 1 dm<sup>3</sup> both with conventional thermal activation [23] at a temperature of 75 °C and with electromagnetic pulse activation of the reaction mixture at 18 °C [22]. Rational mode characteristics of pulse discharge generation, which are set in [22], were used.

We determined the effect of pH value on the residual concentration of ions of Fe, Ni, Cu, Zn in the ranges of pH values of 8.5...10.5. The initial concentration of heavy metal ions changed from 5.34 to 20.01 g/dm<sup>3</sup>. During the

experiment, other technological parameters of ferritization remained stable: the ratio of concentrations of metal ions  $[Fe^{2+}]/\Sigma([Ni^{2+}]+[Cu^{2+}]+[Zn^{2+}])=4/1$ , the process time was 25 min. The residual concentrations of heavy metal ions (ferrum, nickel, copper, and zinc) after processing sludge were determined at the atomic-absorption spectrophotometer AA-6800 (Shimadzu, Japan). The pH of the reaction mixture was controlled by the pH-meter pH-150 (Belarus).

Structural analysis of dried ferrite sediment was performed by powder X-ray diffraction in a step-by-step mode with Cu-K $\alpha$  radiation at the Ultima IV diffractometer (Rigaku, Japan). The video registration took place in the angle interval 2 $\theta$  6...70° in increments of scanning of 0.05° and an exposure time at the point of 2 s. A scanning electron microscope-analyzer REMMA-101A (SELMI, Ukraine) was used to study the microstructure of sediment samples.

The variance and error limits, based on the results of four measurements at each experimental point at a confidence probability of 0.95, were determined by a procedure from [24].

5. The results of studying the energy-saving processing of galvanic sludge by ferritization

## 5. 1. Investigating the dissolution of galvanic sludge in sulfuric acid etching solutions

At the first stage of research into the processing of galvanic sludge, it was dissolved in a sulfuric acid etching solution at constant stirring. To achieve concentrations acceptable for the ferritization process, an etching solution containing  $40\div100$  g/dm<sup>3</sup> of bivalent iron ions was diluted with tap water. To achieve the required total concentrations of heavy metals of  $5\div20$  g/dm<sup>3</sup> in the reaction mixture for the ferritization process [22], the etching solution was diluted by  $3\div10$  times, respectively. The results of measuring the acidity of the environment of diluted etching solution showed that

the pH values were within 2÷2.5, respectively. The results of our study demonstrated that the complete dissolution of the pasty sludge occurred within 5 minutes (Fig. 1).



Fig. 1. Galvanic sludge after treatment with a diluted etching solution

The liquid phase, which was formed as a result of the dissolution of the galvanic sludge in the diluted etching solution, had a pH value of 4.5÷5. This indicates a weakly acidic environment of the resulting solution for the ferritization process.

## 5. 2. Determining the effect of pH value on the degree of extraction of heavy metal ions

The second stage of the process was to carry out the process of ferritization of the liquid phase with the extraction of heavy metal ions from the solution and the formation of chemically resistant ferrite sediment. The focus of this stage of research was one of the most important technological parameters of the ferritization process – the pH value of the reaction mixture. The results of experiments to determine the effect of pH in thermal and electromagnetic pulse activation of the ferritization process on the effectiveness of extraction of heavy metal ions from the reaction mixture are shown in Fig. 2–5.

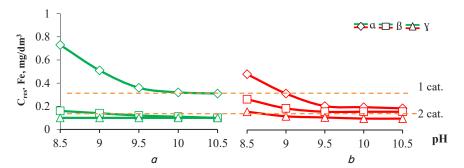
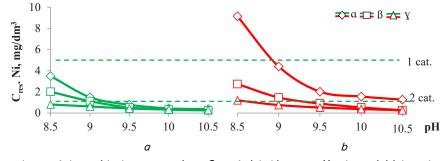
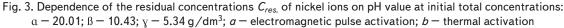


Fig. 2. Dependence of the residual concentrations  $C_{res.}$  of ferrum ions on pH value at initial total concentrations: a - 20.01;  $\beta$  - 10.43;  $\gamma$  - 5.34 g/dm<sup>3</sup>; a - electromagnetic pulse activation; b - thermal activation





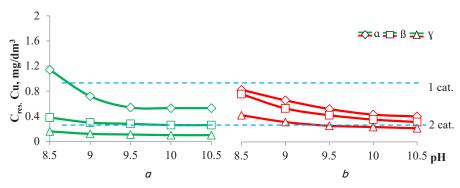


Fig. 4. Dependence of the residual concentrations  $C_{res.}$  of copper ions on pH value at initial total concentrations: a - 20.01;  $\beta$  - 10.43;  $\gamma$  - 5.34 g/dm<sup>3</sup>; a - electromagnetic pulse activation; b - thermal activation

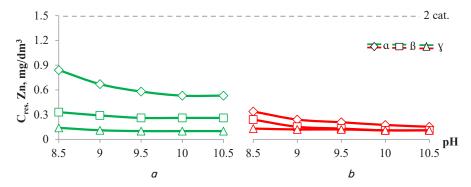


Fig. 5. Dependence of the residual concentrations  $C_{res.}$  of zinc ions on pH value at initial total concentrations: a - 20.01;  $\beta$  - 10.43;  $\gamma$  - 5.34 g/dm<sup>3</sup>; a - electromagnetic pulse activation; b - thermal activation

The resulting values for the concentrations of heavy metals with a relative error of measurements, which does not exceed 4 %, show that the residual content of  $Fe^{tot}$ ,  $Ni^{2+}$ ,  $Cu^{2+}$ ,  $Zn^{2+}$  ions, regardless of the ferritization activation technique and the total initial concentrations, decreases with an increase in the pH value. The residual concentrations of ferrum, nickel, copper, zinc ions in the solution after ferritization are within 0.1+0.73; 0.25+8.89; 0.1+1,14; 0.1+0.84, respectively.

The quality of the purified solution (Fig. 2–5) in most experiments meets the requirements for the water of categories 1 and 2 for galvanic production, regarding the maximum permissible concentrations (MPC) of ferrum, nickel, copper, and zinc ions. Excessive MPC indicators for the water of category 1 is observed with an electromagnetic pulse technique of activation at an initial concentration of  $20.0 \text{ g/dm}^3$ , namely: for Fe<sup>tot</sup> ions at all pH values; for Cu<sup>2+</sup> ions within pH=8.50+8.65. At the thermal activation with an initial concentration of  $20.01\,g/dm^3\!\!:$  for  $Fe^{\text{tot}}$  ions – within  $pH=8.5\div9.0$ ; for Ni<sup>2+</sup> ions – within  $pH=8.5\div8.9$ . In addition, the use of various techniques of ferritization activation makes it possible to achieve an increase in the efficiency of extraction of the specified metal ions to a level that meets the water requirements for galvanic production of category 2. Such effects are observed with electromagnetic pulse activation at an initial concentration of up to  $10.43 \text{ g/dm}^3$  for all considered metal ions within  $pH=9.5\div10.5$ . At the thermal activation, such requirements are met only at an initial concentration of up to  $5.34 \text{ g/dm}^3$  within pH=9.0+10.5.

# 5.3. Investigating the structural properties of ferrite sediment

In the process of ferritization, at various ways of its activation, a black dispersed suspension is formed in the reaction mixture, which subsequently crystallizes with the formation, in particular, of dense ferrite structures. The structure of sediment samples obtained at the initial total concentration of heavy metal ions of  $10.43 \text{ g/dm}^3$  and pH values of  $8.5 \pm 10.5$  was studied. The content of crystalline phases in these samples was also examined.

Structural studies of ferritization sediment indicate its high crystallinity (Fig. 6) except for the sample obtained with electromagnetic pulse activation of the process and pH=8.5.

Identification of phases of the examined samples showed that they contained ferrite of metals whose composition is  $Ni_{0,53}Cu_{0,3}Zn_{0,17}Fe_2O_4$ . The detected ferrite phases demonstrate ferromagnetic properties. Such ferrites of heavy metals, unlike hydroxides, do not dissolve in diluted solutions of strong mineral acids and alkaline at normal temperatures. This is due to the special structure of their crystal lattice of the spinel-type [15]. In addition, there are peaks that belong to the phase of oxyhydroxide ferrum–nickel, namely nickel limonite Fe(Ni)O(OH) with the parameter of the crystal lattice a=2.96 Å, as well as peaks of the sodium sulfate phase  $Na_2SO_4$  with a=5.84 Å.

The results of the quantitative phase analysis of sediment samples (Fig. 7) showed that an increase in the pH of the initial solution leads to an increase in the ferrite phase in sedimentation: with a thermal activation technique, by 24.02 %, electromagnetic pulse – by 36.7 %. This, in turn, helps reduce the phases of nickel limonite Fe(Ni)O(OH) by 19.06 and 27.17 %, respectively, with thermal and electromagnetic pulse activation, as well as sodium sulfate Na<sub>2</sub>SO<sub>4</sub>, by 5.36 %, and 9.53 %, respectively. It should be noted that we have identified in the samples, at different activation techniques and pH=10.5, the same phases, and their quantitative content is almost the same, the difference does not exceed 1.0 %.

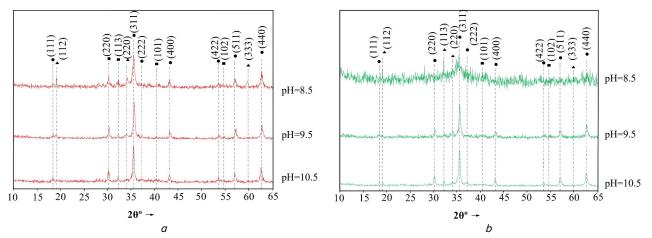


Fig. 6. Diffractograms of ferritization sediment, obtained at different pH of the reaction mixture: a - thermal; b - electromagnetic pulse activation:  $\bullet -$  Fe<sub>2</sub>(Fe,Ni,Cu,Zn)O<sub>4</sub>;  $\blacktriangle -$  (FeNi)O(OH);  $\blacksquare -$  Na<sub>2</sub>SO<sub>4</sub>

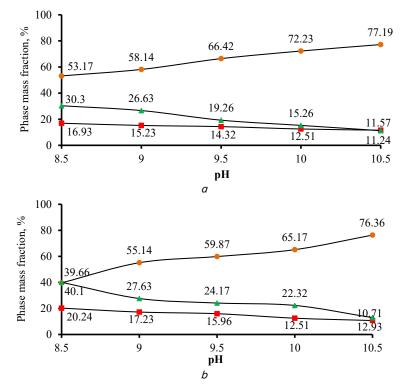


Fig. 7. Quantitative phase composition of ferrite sediment at different pH values: *a* − thermal; *b* − electromagnetic pulse activation; • − Ni<sub>0.53</sub>Cu<sub>0.3</sub>Zn<sub>0.17</sub>Fe<sub>2</sub>O<sub>4</sub>; ▲ − (FeNi)O(OH); ■ − Na<sub>2</sub>SO<sub>4</sub>

Data from the structural analysis of sediment samples correlate well with the results from the scan electron microscopy of ferrite sediment samples [22], which were obtained with different activation techniques of the ferritization process and pH 10. These samples contain irregular spherical crystals in the porous space, and, therefore, have a better sorption ability for both heavy metal ions and organic matter. In addition, the resulting substances have micropores that are formed in the process of obtaining ferrite [15].

## 6. Discussion of results of galvanic sludge processing

The study results indicate the prospects of the two-stage processing of pasty galvanic sludge. The first stage of the process implies dissolving the sludge in the etching solution, the second - the use of energy-saving hydrophase ferritization. It has been experimentally established (Fig. 2-5) that the residual concentrations of heavy metal ions removed from dissolved sludge, regardless of the activation technique of ferritization and initial concentrations, decrease with an increase in the pH value from 8.5 to 9.5. In the pH area of 9.5÷10.5, the residual concentrations of metals are almost non-variable. Obviously, this is due to the fact that with the growth of pH, the predominant role is not the sorption of ions but the crystallization of dispersed compounds of heavy metals at the surface of ferromagnetic particles [25]. At the same time, the effectiveness of the extraction of ions is significantly influenced by the structure and size of these particles. The study results (Fig. 2-5) have established that the ferritization process provides a high degree of extraction of heavy metal ions into environmentally safe insoluble

compounds. It reaches 99.995+99.997 %; 99.589+99.977 %; 99.905+99.968 %; 99.871+99.942 % for Fe<sup>tot</sup>, Ni<sup>2+</sup>, Cu<sup>2+</sup>, Zn<sup>2+</sup> ions, respectively. Under the best conditions of the process (pH=10.5, the output total concentration of metals is 5.23 g/dm<sup>3</sup>), the residual concentrations of heavy metal ions are reduced to the following values: Fe<sup>tot</sup> – 0.1; Ni<sup>2+</sup> – 0.25; Cu<sup>2+</sup> – 0.1; Zn<sup>2+</sup> – 0.1 mg/dm<sup>3</sup>. The quality of the purified solution meets the requirements for water used in galvanic production in terms of the MPC of these heavy metal ions.

Our analysis of structural studies showed (Fig. 6) that sediment samples obtained by thermal and electromagnetic pulse technique of activation contain mainly ferrite phases that have magnetic properties. It should be noted that in addition to the formation of ferrite phases, intermediate solid-phase products of the ferritization reaction, nickel limonite Fe(Ni)O(OH), remain in the sediment. The identified intermediate phase Fe(Ni)O(OH) is less stable compared to metal ferrite but also has ferromagnetic properties [22]. The radiograms also revealed phases of sodium sulfate Na<sub>2</sub>SO<sub>4</sub>, which is in sediment in insignificant quantities. Its presence is explained by the fact that the samples of ferrite sediment obtained were not subjected to preliminary washing with distilled water. The study results also demonstrate that an increase in the pH of the initial reaction mixture leads to an increase in the ferrite phase of Ni<sub>0.53</sub>Cu<sub>0.3</sub> Zn<sub>0.17</sub> Fe<sub>2</sub>O<sub>4</sub> in sedimentation and a decrease in the phases of nickel limonite Fe(Ni)O(OH) and sodium sulfate Na<sub>2</sub>SO<sub>4</sub>. In addition, the quantitative phase composition of ferrite sediment, presented in Fig. 7, indicates that the samples obtained during thermal and electromagnetic pulse activation and pH=10.5are characterized by the maximum content of the ferrite phase of the metal >76 %. Thus, the influence of one of the main technological parameters of the process of ferritization processing of galvanic sludge, the pH of the reaction mixture, on the degree of extraction of heavy metal ions, and the quality of sediment ferritization suitable for further disposal has been shown.

It should be noted that an important advantage of the electromagnetic pulse technique of activating the ferritization process is its energy efficiency that makes it possible to reduce electricity consumption by more than 42 % during the processing of the reaction mixture, compared to thermal activation [22]. At the same time, the electromagnetic pulse technique of activation does not impair the degree of extraction of heavy metal ions from the reaction mixture and leads to the formation of sediment with a high crystalline structure.

It should be noted that the universal technique of processing galvanic sludge proposed in this work has certain limitations imposed by the different qualitative and quantitative compositions of sludge during a certain galvanic production. That insignificantly affects the degree of extraction of heavy metals at the pH values defined in this study but can affect the quality of the formed ferrite sediment for their further useful use.

In the future, it is advisable to study the influence of other technological parameters of the ferritization process using electromagnetic pulse activation on the quality of processing of galvanic sludge.

#### 7. Conclusions

1. The result of the experimental studies is the devised technique of two-stage processing of sludge formed as a result of neutralization of galvanic production wastewater, its transfer to the liquid phase, and subsequent hydrophase ferritization. The possibility of dissolving pasty galvanic sludge in galvanic production waste has been established. Depending on the total concentrations of heavy metals in the reaction mixture in the range of  $5\div20$  g/dm<sup>3</sup>, sulfuric acid etching solution should be diluted by 3 and 10 times to transfer the sludge to the liquid phase, followed by its ferritization. The pH of the resulting solution was within 4.5+5.

2. We have established the effect of the pH value of the reaction mixture on the degree of extraction of heavy metal ions in the process of ferritization processing of galvanic sludge. It was found that with an increase in the pH from 8.5 to 10.5, the residual concentrations of heavy metal ions are reduced to values of  $0.1+0.25 \text{ mg/dm}^3$ ; the degree of their extraction from the reaction mixture is 99.9 %. The water that is thus purified is suitable for reuse in galvanic production in terms of the maximum permissible concentrations of heavy metal ions.

3. Structural features of the obtained sediment have been investigated, in which the following phases were identified: ferrite metals of composition  $Ni_{0.53}Cu_{0.3}Zn_{0.17}$  Fe<sub>2</sub>O<sub>4</sub>, ferrum-nickel oxyhydroxide (FeNi)O(OH), and sodium sulfate  $Na_2SO_4$ . It was established that an increase in the pH of the original reaction mixture leads to an increase in ferrite phases in sedimentation. It was found that at pH=10.5, phases were found in the samples, which are characterized by the maximum content of phases of ferrite of heavy metals – exceeding 76 %.

#### Acknowledgments

We are grateful for the financial support of this research project at the Kyiv National University of Construction and Architecture 5 DB-2021 for the implementation of research and development carried out with support from the Ministry of Education and Science of Ukraine.

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