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Research Article

Ferrite Composite Materials for Water Purification from Organic Dyes

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KEYWORDS

copper-zinc ferrite materials synthesis; co-precipitation
water purification
organic dyes
Sorption characteristics

ABSTRACT

Background: Research in the field of sorption materials obtaining based on secondary resources is applicable, as it saves raw materials. One of the actively developing areas of chemical technology is the synthesis of ferrite composite materials (FC) as sorbents. **Methods:** The concentrations of Cu(II) and Zn(II) ions were determined by atomic absorption spectrometry. The FCs compositions was determined by X-ray phase analysis. The morphological features of FCs were studied by means of electron-probe microanalysis on a scanning electron microscope. The sorption activity of FCs was investigated in the processes of sorption of methyl violet by spectrophotometry. **Results:** The possibility of copper-zinc ferrite materials obtaining from sulfate copper-zinc electrolyte by the co-precipitation method in four modifications was substantiated and experimentally confirmed. The possibility of synthesized FCs using for water purification from organic dyes, such as methyl violet, was proven experimentally. The features and effectiveness of the FCs application as sorption materials were evaluated. **Conclusions:** The main stages of copper-zinc ferrite materials producing were determined. The producrion scheme of FCs obtaining was proposed. It was shown that the presence of ferrite phases in the form of spinel with the general formula FexZnyCuzO4 and the developed surface of the FCs determine their high sorption activity. **Significance:** The practical use of industrial wastewater as a man-made raw material for the technically useful materials production has been further developed.

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1. Introduction

Current scientific and technical progress is connected with the constant acceleration of the rate at which industrial enterprises consume water. Chemical, construction, petrochemical and other industries take large volumes of fresh water. Wastewater from enterprises contains various pollutants, including petroleum products, dyes, surfactants, heavy metal ions, etc. The flow of untreated or insufficiently treated wastewater into water bodies causes economic damage and negatively affects the surrounding natural environment [1,2]. The development of new highly effective wastewater purification technologies is still relevant.

The variety of industrial wastewater treatment methods presented in the scientific literature shows the high efficiency of sorption methods of water purification [3-5]. Research in the field of sorption materials obtaining based on secondary resources is applicable, as it saves raw materials [6,7]. One of the actively developing areas of chemical technology is the synthesis of ferrite composite materials (FC) and the study of their properties as sorbents [3,8]. The ferrite method is a modification of the oxidizing reagent method of wastewater treatment from various pollutants using Fe-containing reagents. In comparison with traditional reagent methods of industrial wastewater purification [9], the possibilities of their treatment with the help of ferrites are significantly expanded. Magnetic nanocomposite sorbents showed high efficiency in the utilization of anionic surfactants and polyphosphates from wastewater [10]. The sorption action of ferrites in water treatment processes for the removal of organic dyes is considered, as well as their application at treatment facilities in the oil industry [11-14].

There is also an ecological aspect to the use of ferrites as sorbents. The process of purifying copper-containing wastewater from impurities with the subsequent formation of ferrite compounds was investigated [15,16]. The cleaning effect when using ferritization was more than 99.9 %.

Research in the field of creating sorption materials on the basis of secondary resources is considered relevant. During the utilization of waste, the resource intensity of production is reduced, cheap sorbents with unique properties are synthesized, and ecological problems of water treatment processes are solved.

The purpose of the research is to determine the main parameters of the process of obtaining copper-zinc ferrite composites and justification of their sorption characteristics.

The research objectives are to justify the stages and parameters of the synthesis of copper-zinc ferrite materials, to evaluate the features and effectiveness of their application.

2. Materials and Methods

The choice of model solutions is justified by the qualitative and quantitative composition of wastewater from enterprises using α-brass processing. The process of ferritization of sulfate copper-zinc electrolyte includes the main stages:

– mixing a model solution with a water-soluble Fe(II) salt;
– formation of metal hydroxides;
– oxidation of Fe(II) ions and formation of ferrites;
– separation of the obtained ferrites from the liquid phase and their washing;
– determination of the composition of the obtained ferrites.

The sequence of stages ensures the production of copper and zinc ferrites in a short time interval with complete precipitation of heavy metal ions from the copper-zinc electrolyte.

Concentrations of copper and zinc ions were determined spectrophotometrically on the "Saturn" atomic absorption spectrophotometer at a wavelength of $\lambda = 328.1$ nm.

The identification of precipitated compounds was carried out by X-ray phase analysis on a powder diffractometer "Siemens D–500". The initial search for phases was performed by PDF-1 card index [17], after which X-ray diffraction calculations were performed according to the Rietveld method using the FullProf program [18].

Morphological features of FCs were studied by means of electron-probe microanalysis (ERMA) on a JSM-6390 LV scanning electron microscope with an INCA X-ray microanalysis system.

The sorption activity of FCs was investigated in the processes of sorption of the organic dye methyl violet (MV) as sorbate by spectrophotometry. The MV color is stable at pH ≥ 7.

The quantitative characteristics of the sorption process are: E − the efficiency of the FCs sorption process and a − the sorption capacity of FCs were calculated according to formulas (1) and (2)

$$E = \frac{(C_1 - C_2) \cdot 100\%}{C_1}, \ \% \tag{1}$$

$$a = \frac{(C_1 - C_2) \cdot V}{m}, \text{mg/g}, \tag{2}$$

where: C_1 is the initial concentration of the sorbate, mg/l; C_2 is the concentration of the sorbate after adsorption, mg/l; V is the solution volume, l; m is the mass of the ferrite composite FC, g.

3. Results and Discussion

3.1. The synthesis of FCs were received by the method of co-precipitation of copper, zinc and ferrous ions introduced in the form of $FeSO_4 \cdot 7H_2O$ salt. The ratio of initial molar concentrations is $\sum Me^{2+} : Fe^{2+} = 1 : 2.5$. The process was ongoing at stirring and heating. After $FeSO_4$ dissolving, a 20–25 % solution of NaOH was added to the mixture to pH 10. Mixed hydroxides of heavy metals were formed in an alkaline medium at 60–65 °C

$$xCu^{2+} + yZn^{2+} + zFe^{2+} + nOH^- \rightarrow Cu_xZn_yFe_z(OH)_n\downarrow. \tag{3}$$

Co-precipitation modifications differed in the type and method of oxidizer supply

– in the form of gaseous oxygen (with the production of composite FC-O materials) [19]

$$Cu_xZn_yFe_z(OH)_n + (2–0.25n)O_2 \rightarrow Cu_xZn_yFe_zO_4 + 0.5nH_2O. \tag{4}$$

– 35% hydrogen peroxide solution (FC-HP) [20]

$$Cu_xZn_yFe_z(OH)_n + 2H_2O_2 \rightarrow Cu_xZn_yFe_zO_4 + nH_2O + (4–n)H^+. \tag{5}$$

– solid salt of potassium peroxodisulfate $K_2S_2O_8$ (FC-PDS) [21]

$$Cu_xZn_yFe_z(OH)_n + S_2O_8^{2-} \rightarrow Cu_xZn_yFe_zO_4 + 2SO_4^{2-} + (8–n)H^+ + (n–4)H_2O. \tag{6}$$

In the fourth modification of synthesis [22], the solid salt $Fe_2(SO_4)_3$ was added to the solution of copper and zinc salts in the ratio $\sum(Cu^{2+} + Zn^{2+}) : Fe^{3+} = 1 : 1$. In an alkaline medium, with the injection of oxygen and the introduction of sodium sulfite γ-Fe_2O_3 was formed, which serves as the basis for the formation of Cu-Zn-ferrite (FC-FS)

$$2Fe^{3+} + 1{,}5O_2 + 6\bar{e} = \gamma\text{-}Fe_2O_3. \tag{7}$$

$$SO_3^{2-} + 2OH^- - 2\bar{e} = SO_4^{2-} + 2H_2O. \tag{8}$$

The reducing reagent Na_2SO_3 was introduced in the ratio $Fe^{3+} : SO_3^{2-} = 1 : 0.5$. After synthesis Cu-Zn ferrites were washed with water to remove impurities and dried.

The process of FCs obtaining during the purification of spent sulfate copper-zinc solutions is presented in Figure 1. After the brass processing, the spent copper-zinc sulfate electrolyte from tank 1 is sent to the first stage of purification in reactor 2 for heating and mixing with the $FeSO_4 \cdot 7H_2O$ salt, which comes through dispenser 3 from tank 4. After the dissolution of $FeSO_4$ to carry out the stage of precipitation of heavy metal hydroxides, NaOH solution is supplied to reactor 2 through dispenser 5 from container 6. Mixing in reactor 2 is carried out at a temperature of 60–70 ºC, maintained by thermostat 7. Oxidation of Fe(II) ions is carried out with an oxidizing reagent, which is fed into reactor 2 from tank 8 through dispenser 9. At the same time, the temperature increases to 85–100 °C and is maintained by temperature regulator 7. To control and adjust the pH of the mixture, samples are taken from reactor 2 using sampler 10.

The mixture enters the settler-distributor 11, in which solid sediment is separated. The filtrate flow containing sodium hydroxide is pumped through pump 12 into tank 13, where the concentration of the resulting NaOH solution is adjusted to the values required by the technological regulations and returned to the technological process in tank 6. The sediment is sent to tank 14 for the purpose of washing away soluble impurities. After sedimentation and decantation of the precipitate, the filtrate flow is pumped by pump 15 into tank 16. The ferrite precipitate is sent to control the purity of the product with the help of X-ray phase analysis 17.

3.2. Mineral composition of FCs. According to the results of X-ray phase analysis, the samples of FCs contain ferrite phases in the form of spinel with the general formula $Fe_xZn_yCu_zO_4$, as well as impurities Na_2SO_4, $NaHSO_4$, $KNaSO_4$, $K_3Na(SO_4)_2$, CuO, Cu_2O, Fe_2O_3. The CuO phase is characteristic for FC-O (9 %), FC-HP (4 %) and FC-PDS (7 %) samples. The presence of maghemite Fe_2O_3, recorded in samples FC-HP (10.5 %), FC-PDS (36 %) and FC-FS (37 %), indicates some instability of the ferrite phase, since before washing with water there was no Fe_2O_3. The multicomponent nature of FCs and the presence of insoluble CuO, ZnO, and Fe_2O_3 oxides are not critical in terms of their use by FC as sorbents, since these oxide phases.

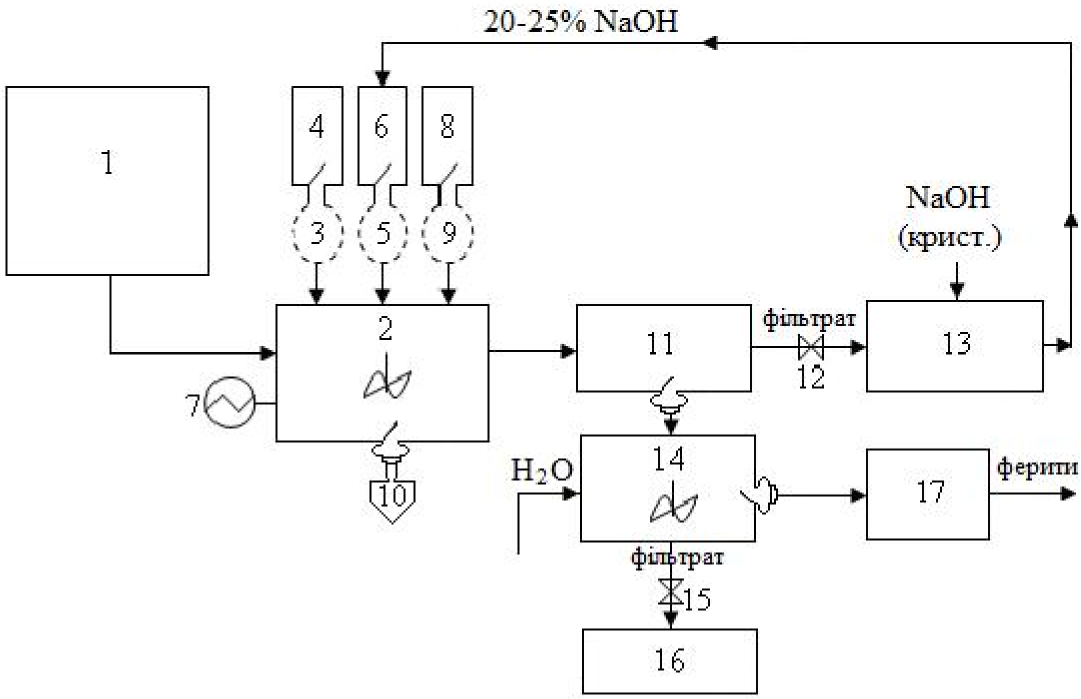
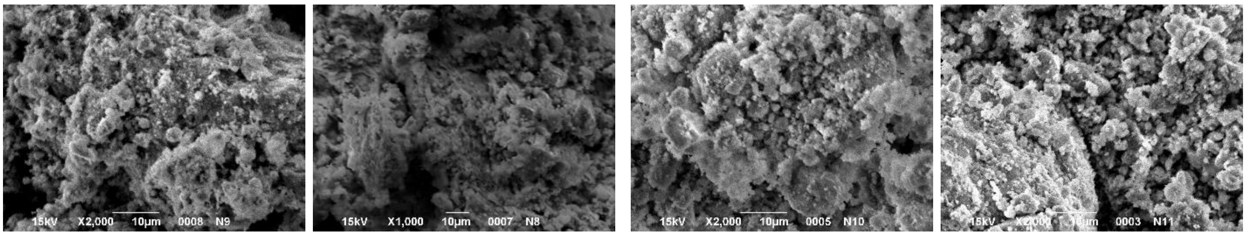


Figure 1. Scheme of the method of obtaining FCs during the purification of copper-zinc sulfate solutions: 1 is the spent sulfate copper-zinc electrolyte; 2 is a tank for mixing with a $FeSO_4 \cdot 7H_2O$ salt; 3, 5, 9 are dispensers; 4 is a tank with a $FeSO_4 \cdot 7H_2O$ salt; 6 is a container with NaOH; 7 is a thermostat; 8 is a tank with oxidizer; 10 is a sampler; 11 is a settler-distributor; 12, 15 are pumpa; 13 is a tank for adjusting the concentration of the NaOH solution; 14 is a tank for washing the received ferrite; 16 is a tank with washed ferrite exhibit sorption properties.

Microscopic studies of FC-O, FC-HP, FC-PDS, FC-FS showed that the samples have a developed surface with a large number of pores of different shapes and sizes (Figure 2).



a b c d

Figure 2. Microphotographs of the surface of FCs particles (magnification 1000–2000):
a – FC-O, b – FC-HP, c – FC-PDS, d – FC-FS

Complex texture the FCs surface can contribute to the layer-by-layer penetration and retention of adsorbed metal ions, molecules of organic compounds, petroleum products, and other hydrocarbon products. This means that, in terms of choosing an effective sorbent according to the characteristics of the surface layer, all studied FCs are good sorbents.

3.3. Sorption properties of obtained copper-zinc FCs were verified in experiments on decolorization of organic dye solutions of methyl violet MV (C_0 = 10 mg/l) (Figure 3) . Analysis of the graphical data (Figure 3) shows that the sorption capacity of FCs in relation to MV changes with variations in *n*. With different mass ratio *n* = "FC : MV" sorption efficiency of *E* varies. It is possible to consider the purification of the aqueous phase from MV as effective in the ratio "FC : MV" for FC-HP, FC-O, FC-PDS at *n* = 500–1000, for FC-FS – at *n* = 3000–5000. At this *n*

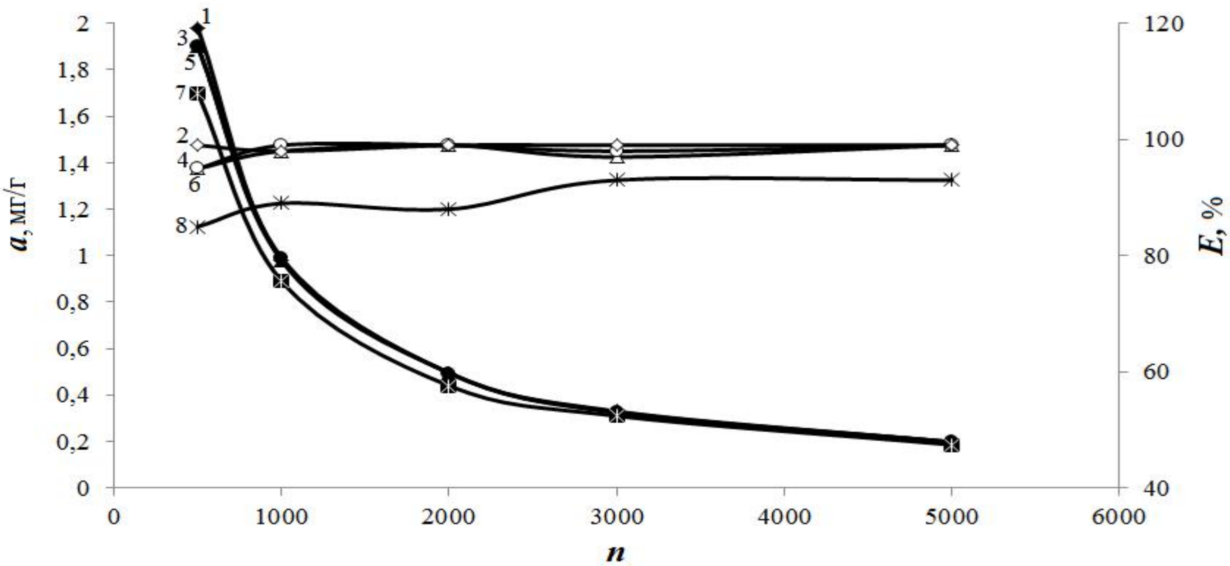


Figure 3. Dependence of the sorption capacity *a* (curves 1, 3, 5, 7) and the efficiency of the sorption process *E* (curves 2, 4, 6, 8) by FCs of the MV dye in the presence of FC-HP (curves 1, 2), FC-O (curves 3, 4), FC-PDS (curves 5, 6) and FC-FS (curves 7, 8) depending on *n* for a time interval of 96 h value the high efficiency of the sorption process is achieved: *E* = 96–100 % (Figure 3, curves 2, 4, 6, 8); simultaneously the maximum sorption capacity of FC: *a* = 1.91–1.98 mg/g (curves 1, 3, 5, Figure 3), *a* = 1.72 mg/g (curve 7, Figure 3). The sorbent can be used 7 times with such parameters of the sorption process.

4. Conclusions

The possibility of obtaining FCs from sulfated copper-zinc electrolyte by co-precipitation method was confirmed. Four modifications of this method are given, which are carried out with a certain initial ratio of components.

The main stages of producing copper-zinc ferrite materials were defined: mixing of spent copper-zinc sulfate solution with a ferrous salt; formation of metal hydroxides; oxidation of Fe(II) ions and formation of ferrites;

separation of the obtained ferrites from the eluate and their washing; determination of the composition of the obtained ferrites.

Presence of ferrite phases in the form of spinel with the general formula of $Fe_xZn_yCu_zO_4$ and a complex developed texture of FCs surfaces can characterize their sorption properties in terms of choosing an effective sorbent.

Sorption properties of synthesized copper-zinc FCs during the decolorization of methyl violet solutions were proven. The high efficiency of the sorption process (96–100 %) was shown as well as the achievement of the maximum sorption capacity of FCs at the mass ratio of *n* = "FC : MV" = 500–1000 for FC-HP, FC-O-1, FC-PDS and with *n* = 3000–5000 for FC-FS.

Further research will be aimed at substantiating the practical application of FCs in the purification of water from other organic dyes in a wide range of their concentrations in solution.

Author's Contributions

V. Datsenko's individual contribution includes conducting synthesis of ferrite composite materials, justification of ferrite production scheme as well as writing the Introduction, Results and Discussion and Conclusions sections.
E. Khobotova's individual contribution includes conducting research on the mineral composition of ferrite composite materials, conducting research on their sorption properties as well as writing the Results and Discussion and Conclusions sections.

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Data Availability

The data supporting the findings of this published article are available from the authors [HFK] upon request.

Conflict of interest

The authors declare no conflict of interest.

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