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Research article

Assessment of the possibility of using iron-magnesium production waste for wastewater treatment from heavy metals (Cd^{2+}, Zn^{2+}, Co^{2+}, Cu^{2+})

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Abstract. Relevant problems associated with treatment of industrial wastewater from heavy metal ions are considered. Due to industrial development, the amount of wastewater increases as well as the risks of heavy metals getting into surface and groundwater, accumulating in water bodies and becoming aggressive environmental pollutants, which affect the animal and human organisms. To assess the possibility of extracting metal ions (Cd^{2+}, Zn^{2+}, Co^{2+}, Cu^{2+}) from industrial wastewater and their further treatment, studies were carried out on redistribution of heavy metals in the "wastewater – waste" system using iron-magnesium production waste. Samples of the investigated waste weighing 0.1; 0.2; 0.5; 1; 1.5; 2 g were taken for wastewater volume of 50 ml per each subsample. Contact time varied from 5 to 180 min, waste fraction was 1 mm. The interaction process showed that the waste efficiently removes metal ions (Cd^{2+}, Zn^{2+}, Co^{2+}, Cu^{2+}) from industrial wastewater. The efficiency of removing a pollutant from the solution depends on the weight of the waste subsample, initial concentration of metal ions, and contact time.

***Keywords*:** extraction; heavy metals; wastewater; removal of ions from solution; waste

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Introduction. The urgency of solving the problems of preventing environmental pollution by toxic elements is growing with industrial development. According to the Ministry of Nature of the RF, in 2021, the number of cases of extremely high pollution levels (EHP) increased by 15 % and of high levels of surface freshwater pollution (HP) in the RF increased by 12 % compared to 2020. Total number of cases of HP and EHP reached a maximum for the period 2012-2021, deviation of the annual value of the indicator from the average for 10 years was 14 %*. Sources of pollutants getting into water bodies are wastewater generated as a result of discharges from industrial enterprises and waste disposal sites (WDS) [1-4].

For many years of industrial development in the Ural region, mining industry of the Sverdlovsk Region remained one of the main components of economy, the development of which is accompanied by large-scale environmental pollution and accumulation of man-induced waste. Of particular hazard are the WDS of extraction and beneficiation of non-ferrous metals, in which metals are in a sulphide form, with the generation of the process of sulphuric acid leaching: dumps of off-balance ores; beneficiation products; waste of metallurgical conversion [5].

* On the state and protection of environment in the Russian Federation in 2021. State report. Moscow: Ministry of Nature of Russia; MGU named after M.V.Lomonosov, 2022, p. 684.

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One of the examples of an area experiencing a significant technogenic impact is the group of Kabanskiye copper and sulphur pyrite deposits. The abandoned deposit lies on the eastern slope of the Middle Urals, about 15 km from the watershed ridge. Distance to the administrative centre of the Ural Federal District and the Sverdlovsk Region (Yekaterinburg) is 190 km. The approximate disturbed area is over 80 ha, of which 11 ha are water bodies.

The deposit was mined by the open pit method, leaving after completion of the work unreclaimed overburden dumps and two quarry excavations filled with acidic waters which are affected by underspoil waters with pH 2.33-3.10, content of zinc 50.12 mg/l, cobalt – 1.73 mg/l, cadmium – 0.20 mg/l, copper – 78.10 mg/l (Table 1).

Table 1

Elemental composition of the studied materials (gross content)

Chemical element	Iron-magnesium production waste, mg/kg	MPC/APC, mg/kg*	Chemical element	Underspoil waters "Kaban 1", mg/l	MPC, mg/l**
Cu	41.25±1.14	3/132	Cu^{2+}	78.10±2.94	0.001
Mg	175,000.00±3,498.32	–	Mg^{2+}	322.50±5.98	40
Fe	52,000.00±1,984.29	–	Fe^{3+}	147.71±3.94	0.1
Zn	77.70±1.84	23/220	Zn^{2+}	50.12±1.14	0.01
Cd	< 1	/2	Cd^{2+}	0.20±0.01	0.005
Co	119.75± 1.41	5/	Co^{2+}	1.73±0.01	0.01
Ni	3,083.00±48.13	4/80	Ni^{2+}	0.23±0.01	0.01
Ca	43,211.00±581.45	–	Ca^{2+}	33.20±0.99	180
K	30,140.00±787.40	–	K^{+}	5.26±0.96	50

* SanPiN 1.2.3685-21 "Hygienic standards and requirements for ensuring safety and (or) harmlessness of environmental factors for humans".

** Order of the Ministry of Agriculture of Russia dated 13.12.2016 N 552 (as amended on 10.03.2020) "On approval of water quality standards for water bodies of commercial fishery importance, including standards of maximum permissible concentrations of harmful substances in waters of water bodies of commercial fishery importance".

Zinc plays an important role in behaviour of various biochemical reactions, metabolism of proteins and nucleic acids, and is a cofactor of a large group of enzymes. Daily toxicity threshold for humans is about 600 mg [3]. Cadmium is toxic [3], irritating to the respiratory tract, and its long-term exposure can cause a severe dysfunction of kidneys, reproductive system, liver, brain, and central nervous system [4, 6]. The physiological role of cadmium is not well understood [3]. Copper and cobalt in high concentrations are also toxic and lead to adverse health effects [3, 7].

Neutralization of high concentrations of heavy metals can be achieved using various physical, chemical, and biological processes [8]. Common methods for removing metal ions from wastewater solution are precipitation, coagulation, ion exchange, and adsorption. Methods associated with precipitation and coagulation lead to formation of a large amount of sludge [9]. Ion exchange is expensive and requires preliminary wastewater treatment since the matrices of ion exchangers can become clogged during operation with various substances in wastewater [9]. Therefore, for ecological rehabilitation of technogenically polluted ecosystems, it is necessary to evaluate the efficiency of using inexpensive materials for wastewater treatment from heavy metals [10-12]. The materials that are widespread in the area of the source of industrial wastewater are of economic interest. Thus, the studies of the processes that determine the accumulation and migration of heavy metals in technogenically polluted ecosystems (especially in areas where industrial enterprises operate), the implementation of methods for detoxifying water resources based on the use of materials which contain production waste, are an intensely developing area of research [13, 14].

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Many researchers [15, 16] studied the efficiency of using industrial waste to remove metals from wastewater solutions. The article [15] substantiates a possibility of using a magnesium-silicate reagent based on serpentinite magnesite, an overburden rock at the Khalilovskoye magnesite deposit, for an efficient multistage wastewater treatment. Thermally activated serpentine minerals can be used to neutralize and treat technogenic solutions, which contributes to precipitation of iron, aluminium, copper, and nickel. For copper, zinc, and nickel, the process of adsorption on the surface of reagent and coprecipitation is recorded.

In the study [16] the alkaline leach residual wire sludge (AWRS) was modified by heat treatment to produce an inexpensive and highly efficient material for removing Cu^{2+} and Ni^{2+} from wastewater. The results showed that the AWRS calcined at 700 °C demonstrated the maximum ability to remove Cu^{2+} and Ni^{2+}. Taking into account a low cost and a high efficiency of the AWRS, the sorbent developed by the authors offers good prospects for removing Cu^{2+} and Ni^{2+} from industrial wastewater solution.

Methods. As part of the research, the experiments were carried out on studying the selective removal of Cd^{2+}, Zn^{2+}, Cu^{2+}, Co^{2+} from a model solution using the material that represents unused resources in the form of iron-magnesium production waste, which forms in large quantities and is a pasty substance of red-brown colour (Table 1). Based on the results of experiments, the influence of the mass of waste subsample, initial concentration of metal ions and contact time for removal of metal ions from the solution of industrial wastewater by iron-magnesium production waste were analysed. Wastewater sampled in the summer of 2021 as part of the field studies within the boundaries of the depleted Kabanskoye deposit was chosen as a model solution (Table 1).

Experiments on redistribution of metals in the "*wastewater – waste*" *system.* The samples of iron-magnesium production waste were averaged by quartering and crushed to a fraction of 1 mm using test sieves and a porcelain mortar and pestle. The samples were dried in a drying oven (ShS-80-01-SPU, OOO "PriborUfa", Russia). The samples were packed in synthetic fabric (perforation 0.025 mm).

Samples of industrial wastewater were taken into five-litre polyethylene bottles washed with distilled water. Samples were filtered using "blue ribbon" filters and stored in a refrigerator for 24 h. Preparation of wastewater samples for elemental analysis was carried out according to the NPDES method (Waste Water) [17].

Waste ashing was accomplished in MARS 5 Digestion Microwave System (CEM Corporation, USA) according to the EPA 3052 procedure [17]. 9 ml of HNO_3 and 3 ml of HF were added to the subsample of 0.5 g, the mixture was stirred, allowed to stand for 15 min, and the vessels were closed. EasyPrep vessels recommended by the manufacturer [17] were used. Temperature rising time to 180 °C was 6 min, temperature maintenance time was 10 min, and power was 1.800 W [17]. At the outlet, when diluted to 50 ml, a transparent sample was obtained without colour and particles.

Metal ion concentrations in wastewater, mineralized samples and obtained filtrates were determined by atomic absorption spectroscopy (AAS) in an air-acetylene flame (Varian AA 240 FS, Varian Australia Pty Ltd, Australia). Wavelengths used, nm: Cd 228.8; Zn 213.9; Co 240.7; Mg 202.6; Cu 324.7; Fe 248.3; Ni 232.0. Detection limits of elements in solution, μg/l: Cd 1.5; Zn 1.6; Cu 1.2; Mg 0.3; Fe 7.3; Ni 5.8; K 0.8; Ca 0.4.

Subsamples (0.1; 0.2; 0.5; 1; 1.5; 2 g) were weighed and placed in conical test tubes of the "falcon" type. Industrial wastewater of the volume of 50 ml was added to them. Then, the samples were stirred (99 rpm) for 120 min using an ELMI RM-1L rotary mixer (ELMI LTD, Latvia). The resulting solutions were filtered using "blue ribbon" filters. Industrial wastewater (50 ml) was added to 0.2 g subsamples, and the samples were stirred for 5 to 180 min. The resulting solutions were filtered using "blue ribbon" filters.

To construct isotherms and determine the effect of initial concentration of metal ions Cd^{2+}, Zn^{2+}, Cu^{2+}, Co^{2+} in solution on the efficiency of the wastewater treatment process, solutions were prepared with concentrations: 10; 30; 50; 100; 300; 500; 1,000 mg/l. Solutions were prepared from reagents of

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qualifications "extra pure" and SSS (state standard sample, initial concentration from 1 to 10 g/l). The interaction occurred at contact time of 120 min with subsample 0.2 g, 50 ml of solution, at room temperature and pH 2.3-2.5, regulated with NaOH and HNO_3. Hanna HI 99121 (Hanna Instruments, Germany) was used to determine pH and temperature. pH of the aqueous extract was determined at a weight ratio of the subsample and distilled water between them 1:5 (GOST 26423-85).

Static exchange capacity of waste q_e (mg/g) and the degree of pollutant extraction from solutions were calculated using the following equations:

$$\text{SEC} = \frac{\left(C_{\text{init}} - C_{\text{equil}}\right)V}{g};$$

$$E = \frac{C_{\text{init}} - C_{\text{equil}}}{C_{\text{init}}} \cdot 100,$$

where C_{init} is the initial concentration of copper ions in the solution, mg/l; C_{equil} – the equilibrium (residual) concentration of copper ions in the filtrate, which sets in in water after mixing of water and substrate, mg/l; g is the weight of a dry subsample of the substrate, g; V – volume of model solution added to waste, l.

The results of calculating the indicators of extraction degree of pollutants from the solution are given in Table 2.

Table 2

Results of chemical analysis of the obtained filtrates (50 ml, contact time 120 min)

Subsample, g	Content of chemical elements in filtrate, mg/l					pH
	Cd^{2+}	Zn^{2+}	Co^{2+}	Mg^{2+}	Cu^{2+}	
0.1	0.20±0.006	42.11±1.34	1.15±0.10	429.80±8.91	36.83±1.29	4.06±0.01
0.2	0.019±0.005	10.24±0.94	0.90±0.09	642.60±13.04	5.98±0.81	4.52±0.01
0.5	0.001±0.0007	0.12±0.011	< 0.005	849.60±14.01	0.09±0.005	5.87±0.01
1	< 0.0015	0.10±0.009	< 0.005	1,176.20±22.01	0.08±0.04	6.76±0.01
1.5	0.005.0.002	0.09±0.003	< 0.005	1,539.27±19.23	0.08±0.003	7.37±0.01
2	0.008±0.002	0.10±0.001	< 0.005	2,153.00±34.41	0.08±0.004	8.01±0.01

Discussion of results. To substantiate the possibility of using iron-magnesium production waste as a material for removing metals from wastewater solution, the following factors influencing the interactions were studied: initial concentration; contact time; waste dosage; pH of aqueous medium [18-20].

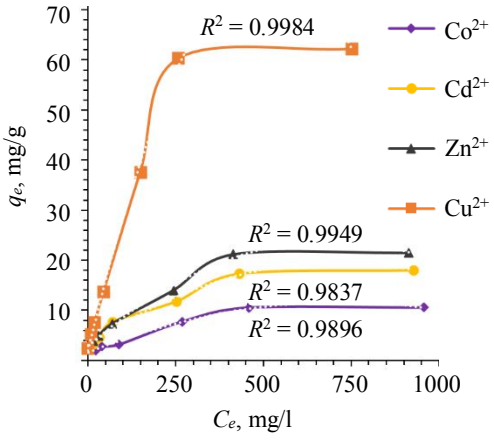


Fig.1. Effect of initial metal concentration in solution on wastewater cleaning process with iron-magnesium production waste

According to the results of elemental analysis, iron-magnesium production waste contains significant concentrations of the studied ions, mg/kg: Cu 41.25; Mg 175,000.00; Zn 77.70; Co 119.75. Industrial wastewater has an acidic medium (pH 2.33) and a high salinity. Content of the studied metal ions, mg/l: Cu 78.10; Zn 50.12; Co 1.73; Cd 0.20 (see Table 1).

Initial concentration of metal ions C_e (mg/l) is an important factor that controls the transfer of metal ions from aqueous solution to waste [21]. Its effect was studied in the range of 10-1,000 mg/l for Cd^{2+}, Zn^{2+}, Co^{2+}, Cu^{2+} at pH 2.3-2.5 (Fig.1). A model solution prepared from the GSS and reagents was used as a simulation of wastewater pollution.

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The number of extracted metal ions q_e increases due to the growth of initial concentration of metal ions from 10 to 500 mg/l. Further growth of metal concentration in the solution to 1,000 mg/l does not increase the extraction efficiency (Fig.1). Thus, the results showing a linear dependence at the initial stage can be explained by the presence of available active centres, i.e., areas on the surface of iron-magnesium production waste that can interact with the studied metal ions or compete for a certain number in their initial concentration. Further, the number of active centres decreases, therefore, an increase in the concentration of metal ions in the solution over 1,000 mg/l will lead to saturation of waste areas and will not increase the treatment efficiency [9, 18, 19]. This necessitates research to assess the possibility of selective removal of the studied elements in terms of forming a cascade of biological ponds, taking into account the material composition of the bed and the composition of the filtration dam sensitive to each chemical element.

Study of the influence of interaction characteristics on the removal of metals from industrial wastewater solution. The time of equilibrium onset is an important parameter when conducting research on wastewater treatment [22, 23]. The maximum possible absorption of Cd^{2+}, Zn^{2+}, Co^{2+}, Cu^{2+} ions by iron-magnesium production waste depending on contact time was studied to determine the time of equilibrium onset. The effect of contact time on wastewater treatment was investigated using a constant concentration of the solution (Table 3) with different time intervals in the range of 5-180 min at room temperature. Elemental composition of the filtrates obtained as a result of interaction of iron-magnesium production waste with wastewater is given in Table 3.

Table 3

Results of chemical analysis of the obtained filtrates (50 ml, subsample 0.2 g)

Contact time, min	Content of chemical elements in filtrate, mg/l							pH
	Cd^{2+}	Zn^{2+}	Co^{2+}	Mg^{2+}	Cu^{2+}	Fe^{3+}	Ni^{2+}	
5	0.19±0.001	43.01±1.09	1.06±0.003	530.88±14.03	43.19±1.32	1.55±0.030	0.23±0.011	4.05±0.012
10	0.18±0.001	39.48±0.98	1.03±0.003	539.84±13.12	19.18±1.01	0.03±0.003	0.22±0.010	4.10±0.014
15	0.17±0.001	38.48±0.67	1.03±0.003	558.40±12.05	17.85±0.78	0.09±0.002	0.21±0.009	4.33±0.011
30	0.18±0.001	37.52±0.70	1.03±0.003	604.24±15.09	16.85±0.81	< 0.0073	0.13±0.014	4.78±0.015
60	0.04±0.007	10.62±0.79	1.02±0.004	1,076.88±21.87	6.74±0.77	< 0.0073	0.05±0.001	4.89±0.011
120	0.03±0.007	9.61±0.91	1.03±0.001	979.04±4.94	5.25±0.76	< 0.0073	< 0.0058	4.99±0.014
180	0.03±0.007	9.05±0.68	1.03±0.001	980.00±5.19	5.20±0.86	< 0.0073	< 0.0058	5.01±0.012

In order to assess the possibility of secondary pollution as a result of interaction of iron-magnesium production waste with a model solution of industrial wastewater, the obtained filtrates were also analysed for Fe^{3+} and Ni^{2+} (Table 3).

There is a dynamics in the decrease of concentrations of the studied elements depending on contact time and a possible absence of secondary pollution, provided that iron-magnesium production waste is used. The values of iron and nickel ions in the obtained filtrates do not exceed the initial ones. Figure 2 shows the effect of contact time on the removal degree of Cd^{2+}, Zn^{2+}, Co^{2+}, Cu^{2+} ions from filtrates using iron-magnesium production waste.

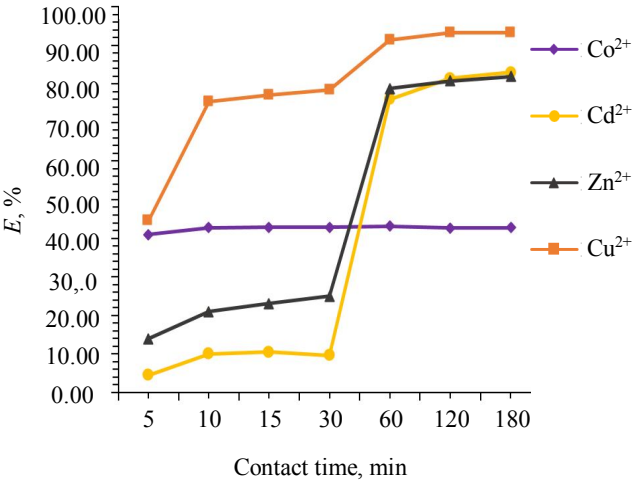


Fig.2. Effect of contact time on degree of metal ions extraction from solution

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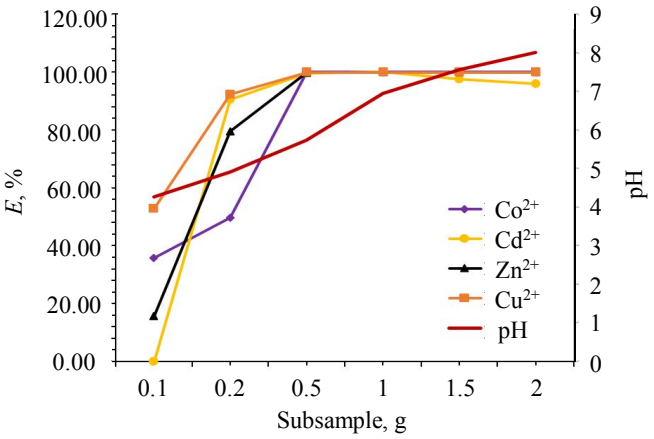


Fig.3. Influence of waste dosage and filtrate pH on the degree of ions extraction from solution

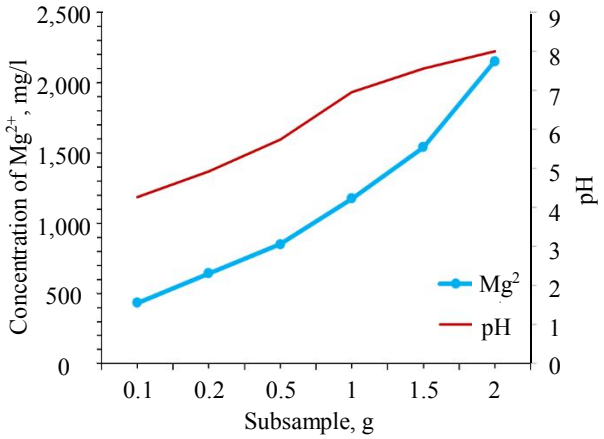


Fig.4. Change of filtrate pH relative to Mg^{2+} concentration in solution

Removal of the studied elements from solutions increases after 30 min of interaction with waste and reaches equilibrium at 120 min. An exception is Co^{2+} – the extraction degree for this element varies from 40.89 to 42.72 % and practically does not change with respect to a given time period. The efficiency of initial removal for copper, zinc and cadmium can be associated with the occurrence of a large number of free areas available on the waste surface. As the contact time increases, the number of active centres decreases, and further increase does not lead to significant changes in the treatment process [18, 19] (see Table 2).

pH of the medium [24, 25] affects both the chemical composition of the solution and the efficiency of the treatment process [26, 27]. An equally important parameter for metal extraction from solution is also the dosage of the material under study [28-30]. The effect of the waste dose on the treatment process should be optimized [31-33], since the mass of the waste affects the ability of the material to extract pollutants from the solution at a given initial waste concentration in operating conditions [34-36]. The process of extracting ions from solution was studied by varying the mass of waste from 0.1 to 2 g in 50 ml of solution with a constant concentration (see Table 1), constant stirring rate (99 rpm) for 120 min at room temperature. The effect of waste subsample on the removal of heavy metal ions from wastewater is shown in Fig.3.

Percentage of metal ions removal from the solution increases with a simultaneous increase in the dosage of waste (Fig.3). The minimum values of *E* indicator were recorded at waste dosage of 0.1 g and are, %: for Cd^{2+} 0; Zn^{2+} 15.78; Cu^{2+} 52.78; Co^{2+} 35.83; when the dosage is increased to 0.5 g, this indicator increases, %: Cd^{2+} 99.5; Zn^{2+} 99.74; Cu^{2+} 99.88; Co^{2+} 100 and reaches the equilibrium. Thus, in the graphs (Fig.2, 3) there is a tendency of increase in percentage of heavy metal ions removed from wastewater. Figure 4 shows the effect of pH on the concentration of magnesium ions in the filtrate depending on the weight of subsample of the investigated material.

pH increases depending on the weight of subsample of the investigated material: the larger the subsample, the higher the pH of the filtrates (see Table 2). This is due to the fact that iron-magnesium production waste has an alkaline pH of 8.7, since it contains a large amount of magnesium (Fig.4), thus diluting the acidic underspoil water with initial pH 2.33.

The results of elemental analysis of iron-magnesium production waste after interaction with model solution of industrial wastewater are given in Table 4. The data show the number of extracted metal ions under study by production waste from the solution on a dry weight basis. According to the obtained results, the number of detected metal ions in waste increases with time; the maximum values are recorded at contact time of 120 min, then a decrease in the values of metal ions in waste is observed (Table 4). The data show that in the composition of iron-magnesium production waste samples after interaction with industrial wastewater, with an increase in contact time from 5 to 120 min,

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Cd, Zn, Fe and Cu are found in higher concentrations than in initial samples. A significant decrease in Mg in the waste composition is also recorded.

Table 4

Results of elemental analysis of waste after interaction with wastewater (gross content)

Contact time, min	Content of chemical elements in waste, mg/kg					
	Cd	Zn	Co	Mg	Cu	Fe
5	1.20±0.03	120.54±1.23	100.00±1.75	10,021.32±1,948.24	351.21±25.04	61,819.25±2,948.47
10	1.25±0.03	127.50±1.41	131.75±2.37	16,822.50±2,821.52	407.75±21.48	73,386.34±3,645.75
15	2.21±0.09	615.54±29.47	135.11±2.04	28,983.21±3,948.63	885.37±36.94	64,528.73±3,562.74
30	3.00±0.14	844.04±41.22	140.25±3.17	32,430.64±4,958.01	3,354.01±138.04	68,945.50±3,465.39
60	3.75±0.02	1,035.09±28.98	169.24±4.08	27,562.21±4,756.06	4,137.50±241.10	69,746.25±2,547.05
120	3.78±0.16	1,041.66±21.84	154.32±5.19	24,842.30±2,484.05	3,775.32±194.90	61,207.93±2,567.93
180	3.54±0.17	663.99±17.38	96.22±2.28	17,594.54±1,875.75	1,499.55±53.10	62,641.50±2,857.05

Conclusion. Studies on redistribution of heavy metals in the "wastewater – waste" system using iron-magnesium production waste are given. The choice is due to the waste availability, since this is production waste forming in large amounts and is potentially cheap material for wastewater treatment. The enterprise, at which waste forms in the production process is not far from the facility (the depleted Kabanskoye deposit), which has a negative impact on the environment, this helps cutting the expenses on environmental protection measures and reducing the amount of waste to be disposed of at the WDS.

Treatment process showed that the waste efficiently removes metal ions (Cd^{2+}, Zn^{2+}, Co^{2+}, Cu^{2+}) from industrial wastewater. The removal efficiency depends on the weight of the waste subsample, contact time, initial concentration of metal ions in the solution and pH, and increases with growing dosage. The most efficient indicators of wastewater treatment from ions of the studied metals using iron-magnesium production waste are: contact time 120-180 min; dosage 4-10 g/l; initial concentration of metal ions (Cd^{2+}, Zn^{2+}, Co^{2+}, Cu^{2+}) in the solution not more than 500 mg/l.

The data obtained indicate a possibility of using iron-magnesium production waste to reduce the metallic aggressiveness of wastewater and are of great interest in the development of measures on wastewater treatment, in terms of forming a cascade of biological ponds, taking into account the material composition of the bed and the filtration dam sensitive to each chemical element. Thus, further research to find a possibility of involving industrial waste in the economic turnover for environmental purposes offers good prospects.

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