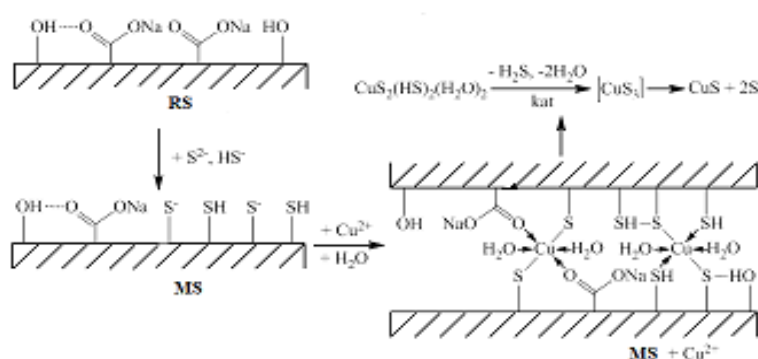# Evaluation of the Efficiency of the Reuse of Sorbents and their Modified Forms for the Removal of Copper Ions from Water

Olha S. Khudoiarova* [ID], Oleg A. Blazhko [ID], and Alina V. Blazhko[ID]

In recent years, spent kieselguhr and activated carbon are considered not as waste, but as secondary raw materials. The purpose of the work is to investigate the effectiveness of using a previously regenerated spent food industry sorbent modified with sulphide and hydrosulphide ions for adsorption removal of copper (II) ions from water. A comparative analysis of the degree of removal and adsorption of copper (II) ions by the regenerated sorbent (RS) and its modified form (MS) was carried out. Insignificant adsorption of Cu^{2+} on the surface of the regenerated sorbent (RS) is explained both by the nature of the adsorbate and the morphology of the adsorbent after its acid-alkaline activation. Sorption of Cu^{2+} ions is mainly carried out by carboxyl groups. Modification of the regenerated sorbent surface with more active sulphide and hydrosulphide ions leads to a significant increase in its selective adsorption in relation to copper (II) ions. It was established that topochemical reactions of formation of copper (II) sulphide CuS and elemental sulphur take place on the surface of the modified sorbent (MS). The possibility of topochemical transformations has been established by IR-spectral and XRD studies. The use of a sorbent modified with sulphide and hydrosulphide ions increases the removal of copper (II) cations from the studied solutions by 65.5 times. The obtained results allow us to recommend the use of a regenerated sorbent (RS) of the food industry, modified with sulphide and hydrosulphide ions, to remove copper (II) ions from water.

Graphical abstract

1. Introduction

Pollution of the environment by wastewater and solid industrial waste is a significant burden on the environment. Industrial enterprises of the chemical and engineering industries generate significant volumes of industrial

Vinnytsia Mykhailo Kotsiubynskyi State Pedagogical University, Ostrozhskogo Street, 32, 21100, Vinnytsia, Ukraine. *Corresponding author's e-mail: **helgakhudoyarova@gmail.com**

wastewater. Wastewater from galvanic industries is the main source of heavy metals entering the environment and irrational use of water as a technical resource The flow of such wastewater into natural reservoirs leads to negative phenomena, such as increasing concentrations in the reservoir water and entering water supply systems. As a result, water quality indicators deteriorate. This poses a threat to human health [1,2].

Toxic solutions are sometimes diluted with water to reduce the concentration of pollutants below the maximum permissible concentration (MPC). However, this procedure cannot always be carried out, so the problem of mandatory water purification arises. Various physical and chemical methods are used to implement such water purification, among which the most popular method is adsorption using natural and synthetic sorbents [3,4], industrial waste [5,6]. To increase the sorption capacity, modification of the sorption matrix is popular [7,8]. Nevertheless, even complete wastewater treatment does not guarantee environmental safety. Solid waste, which is formed a result of cleaning, is most often buried in special storage facilities or stored in landfills. The problem of accumulation of spent sorbents is partially solved by restoring their sorption properties for reuse in cleaning technological processes [9]. Also, spent sorbents can be used to obtain valuable technical products, for example, components of carbon-sulphur-containing plastic lubricants [10].

According to the principles of "green chemistry", production waste or secondary industrial raw materials should be subject to chemical processing, not incineration and storage in special landfills [11]. Thus, during the production of sugar syrup for soft drinks, a large amount of waste sorbents is formed, which includes activated carbon and kieselguhr. In recent years, spent kieselguhr and activated carbon are considered not as waste, but as secondary raw materials. One of the promising areas of research can be the use of spent sorbents for removing copper (II) ions from water. Activated carbon and its synthetic modifications, as is known, are characterized by more pronounced sorption, ion exchange and redox properties [12-14]. Also, a promising area of research is the use of spent kieselguhr as a sorbent for wastewater treatment. It is known that pure kieselguhr has weak adsorption properties [15]. Spent kieselguhr has greater adsorption activity [6]. This is due to the presence of additional adsorbing sites - organic substances bound to the kieselguhr mineral matrix. In the case of acid-alkaline activation, obviously, such centres are formed the most. This makes it possible to use spent kieselguhr to remove copper (II) ions from water [6].

The presence of even a small amount of certain surface groups or atoms can significantly affect the characteristics of activated carbon and kieselguhr and fundamentally change the parameters of processes involving them. In order to intensify the sorption removal of copper (II) ions from aqueous solutions, we propose to modify the surface of the sorbent studied in the work with sulphide and hydrosulphide ions.

Previously, studies [9,16] established the possibility of regeneration of spent sorbent after cleaning sugar syrup. The authors explained the restoration of the sorption capacity after regeneration by the passage of acid-base chemical reactions on the surface of the sorbent and the washing out of the products of their interaction with water. The technology of regeneration of spent sorbent proposed by the authors allows for 100% recovery of its sorption capacity with a yield of 98%. A large amount of technical water 995 L (~ 83%), which is used at the same time, is directed to repeated sorption purification with the formation of a closed technological cycle.

The use of regenerated sorbent for sorption treatment of sulphide-alkaline wastewater of chemical and petrochemical industries has been investigated in the study [17]. The degree of extraction of sulphide and hydrosulphide ions from water solutions depends on their concentration, adsorption time, and the mass of the used sorbent. The degree of removal of sulphide and hydrosulphide ions by the regenerated sorbent from aqueous solutions is about 96%. The analysis of the obtained data confirms the effective use of the studied sorbent in the proposed technology. The sorbent, the surface of which is modified with sulphide and hydrosulphide ions, can be used for cleaning industrial copper (II)-containing wastewater.

The purpose of the study is to investigate the effectiveness of using a regenerated food industry sorbent modified with sulphide and hydrosulphide ions to remove copper (II) ions from water.

2. Material and Methods

Used reagents: sodium sulphide nonahydrate ($Na_2S \cdot 9H_2O$, Ukraine), copper (II) sulphate pentahydrate ($CuSO_4 \cdot 5H_2O$, Ukraine), iodine (I_2, Chile), potassium iodide (KJ, India), sodium thiosulphate pentahydrate ($Na_2S_2O_3 \cdot 5H_2O$, Ukraine), sodium ethylenediaminetetraacetate dihydrate ($C_{10}H_{14}N_2O_8Na_2 \cdot 2H_2O$, Czech Republic), Murexid ($C_8H_8N_6O_6$, China), starch (($C_6H_{10}O_5)_n$, Ukraine), sodium hydroxide (NaOH, Ukraine), hydrochloric acid (HCl, 36-38%, Ukraine). Reagents were purchased from Sfera Sim LLC (Lviv, Ukraine) and were used without further purification.

As a sorbent, a mixture of Decolor A brand activated carbon and kieselguhr "Bekogur 200" and "Bekogur 3500" (manufactured by E. Begerow GmbH & Co (Germany)) in a ratio of 4:6, selected at the enterprise, was used for the production of carbonated drinks "Panda" (Vinnytsia, Ukraine).

To remove copper (II) ions from water, model solutions of copper sulfate were used, which corresponded to the copper content in the washing waters of the electrochemical copper plating process after the use of acidic electrolytes with pH = 1.5-2.5.

Procedure of regeneration of spent sorbent

Regeneration of the sorbent was carried out according to the method described in works [9,15]. First, the sorbent was washed with water, and then 1.25% NaOH (or sequentially 1% NaOH and 4% HCl) was added. Rational parameters of regeneration are: mass ratio of sorbent : H_2O = 1:4; regeneration time 45–60 min; process temperature 50–60° C; the intensity of mixing of the reaction mass is 200 rpm. After filtering, washing with water and drying at 105 °C, a regenerated sorbent (RS) was obtained.

Procedure for preparing model solutions

The sorbent was modified with a solution with a concentration of sulphide and hydrosulphide ions of 1.40 mol/L and 0.21 mol/L, respectively, which was prepared by dissolving a portion of $Na_2S \cdot 9H_2O$ in distilled water.

A model copper-containing solution with a Cu^{2+} concentration of 300.0 mg/L was prepared by dissolving a portion of $CuSO_4 \cdot 5H_2O$ in distilled water. Solutions with the

concentration of copper (II) cations of 1.5, 5.0, 30.0, 50.0, and 150 (mg/L) were obtained by diluting the initial solution (300.0 mg/L) with distilled water in appropriate proportions. The residual concentration of sulphide and hydrosulphide ions in the filtrate after the sorbent modification was determined by the method of iodometric titration [18].

The residual concentration of copper (II) ions in the filtrate after their removal was determined by the complexometric titration method, the degree of ion removal and the amount of adsorption were calculated [19].

The degree of removal E, % of copper (II) ions was determined by by Eq. (1).

$$E = \frac{C_0 - C_e}{C_0} \cdot 100 \qquad (1)$$

where, C_0 − initial concentration of copper (II) ions in the solution, mg/L; C_e − residual concentration of copper (II) ions in the solution, mg/L.

The amount of adsorption A, mg/g of copper (II) ions was calculated by Eq. (2).

$$A = \frac{C_0 - C_e}{m} \cdot V \qquad (2)$$

where, C_0 − initial concentration of Cu^{2+} in the solution, mg/L; C_e − concentration of Cu^{2+} after reaching adsorption equilibrium, mg/L; m − sorbent mass, g; V − the volume of the solution in which adsorption is carried out, L.

General method of removal of copper (II) ions.

The mixture was prepared by adding 1 g of RS to 100 mL of a model solution of copper (II) ions. The resulting mixture was intensively stirred (350 rpm) for 30−45 minutes at a temperature of 20−25°C and left for the final completion of the process for one day at the same temperature. The mixture was filtered under vacuum and the residual concentration of Cu^{2+} ions was determined in the filtrate. Based on the obtained experimental data, the degree of removal E (%) of copper (II) ions from the solution was calculated.

The adsorption kinetics was studied on a solution with a concentration of Cu^{2+} ions of 150 mg/L. Research was conducted for different doses of RS (solid : liquid ratio of 2 g/100 mL; 5 g/100 mL; 10 g/100 mL; 15 g/100 mL and 20 g/100 mL). The mixture was stirred and left for 0.5; 1.0; 1.5; 2.0; 4.0; 8.0; 24.0 hours. The residual concentration of Cu^{2+} ions was determined in the filtrates, the degree of ion removal was calculated using Eq.1, and the amount of adsorption was calculated using Eq.2. As the main kinetic dependence, an integral kinetic curve was obtained, that is, the change in adsorption over time: $A=f(\tau)$, where A is adsorption at the time τ.

General method of modification of regenerated sorbent

To 1 g of RS through a drop funnel, a sulphide-alkaline solution was added in separate portions (~30 mL), in which the concentration of sulphide and hydrosulphide ions was 1.40 and 0.21 mol/L with pH= 12.6-13.2. The reaction mass was mixed with a high-speed stirrer for 5−15 min. Then a sample was taken and a qualitative analysis of the reaction mass was carried out for the presence of sulphide and hydrosulphide ions in it. Then another portion of sulphide-alkaline solution was added. Complete sorption of sulphide and hydrosulphide ions by the sorbent surface was

considered complete if sulphide and hydrosulphide ions were present in the solution. The modified sorbent (MS) was filtered on a Schott filter and dried.

Removal of copper (II) from water with a modified sorbent was carried out according to the following method. 1 g of MS was placed in the reactor. Then, the first portion (~30 mL) of a model solution with a copper (II) ion content of 150.0 mg/L was added from the dropping funnel. The reaction mass was mixed with a high-speed stirrer for 5−15 min. After that, a sample was taken and a qualitative analysis of the reaction mass was carried out for the presence of unbound copper (II) ions in it. Then another portion of water was added with a similar sequence of technological operations. Complete binding of copper (II) ions was considered complete in the presence of copper (II) ions in the reaction mass. During the reaction, a small amount of hydrogen sulphide was released. The reaction mass was kept for 24 hours, filtered, thoroughly washed with water and dried.

Instruments

Weighing substances were taken on an analytical balance AS 220.R2 (RADWWAG, Poland).

A VELP AREC high-speed magnetic stirrer (VELP Scientifica, Italy) was used for mixing.

The sorbent surface was studied by physic-chemical methods. The surface study of sorbent samples using the IR spectroscopic method was conducted at the Vinnytsia Scientific Research Expert and Forensic Centre of the Ministry of Internal Affairs of Ukraine. The modified surface of the sorbents was studied using the diffuse reflection method on a Nicolet iN 10FX IR Fourier spectrometer from Thermo Fisher Scientific in the range 4000−525 cm^{-1} with a maximum resolution of 4 cm^{-1}. A sample of the test substance weighing 0.0010 g was placed on the surface of the SmartiTX−Diamond attachment. The spectra were recorded in the total disturbed internal reflection mode. The obtained spectra were deciphered using the IR spectra libraries of the "Omnic Picta 1.5.126" program (USA).

The surface study of sorbent samples by X-ray phase analysis was performed at the Ukrainian State University of Chemical Technology (Dnipro) (Dron-2 diffractometer in monochromatized Co-K$_\alpha$ radiation (λ = 1.7902 Å). The data of the X-ray analysis were identified by comparing the interplanar distances (d, Å) and the relative intensities (I_{otn} - I/I_0) of the experimental curve with the data of the PCPDFWIN electronic file system.

3. Results and Discussion

Sorption of copper (II) ions on the matrix surface of the sorbent takes place in a strongly acidic environment at pH= 1.5−2.5. Adsorption removal of copper (II) ions using RS showed low efficiency (Tab. 1).

Table 1. Adsorption of copper (II) ions on the RS.

Cu^{2+} concentration, mg/L		Degree of removal E, %	Adsorption capacity A, mg/g
Initial concentration C_0	Equilibrium concentration C_e		
1.5	1.45	4.21	0.006
10	9.44	5.72	0.059
30	26.90	10.41	0.311
50	44.21	11.59	0.581
150	115.06	23.28	3.496
300	206.09	31.31	9.391

The obtained adsorption isotherm of Cu^{2+} ions on the RS belongs to the III-th type according to the classification of S. Brunauer [20], with a characteristic curve. This bend is caused by a weaker adsorbate-adsorbent interaction compared to the adsorbate-adsorbate interaction (Fig. 1).
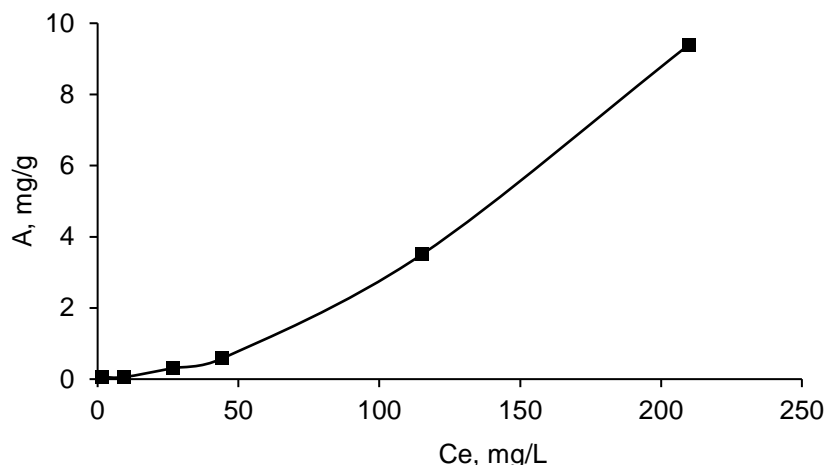

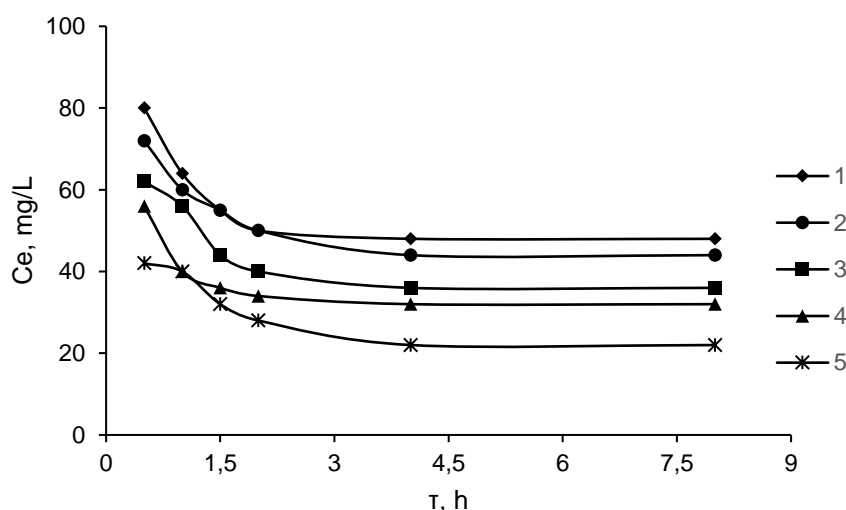**Fig. 1.** Adsorption isotherm of copper (II) ions on the RS.

Fig. 2. Kinetics curves of Cu^{2+} ions adsorption for different doses of RS (solid : liquid ratio of: 1 - 2 g : 100 mL; 2 - 5 g : 100 mL; 3 - 10 g : 100 mL; 4 - 15 g :100 mL and 5 - 20 g : 100 mL), C_0 (Cu^{2+}) = 150 mg/L.

Based on the results of kinetic studies, the time to reach equilibrium in the system "RS - copper (II) ions" was determined. The dependence of the residual concentration of Cu^{2+} ions in the model solutions on the concentration of the RS and the interaction time has been established (Fig. 2). The concentration of Cu^{2+} ions for all studied ratios decreases during the first 3 *h* of adsorption stabilizes at the 4 *h* mark and does not change further.

The change in the adsorption of Cu(II) ions over time for different doses of RS is shown in Fig. 3.The highest degree of removal of Cu^{2+} ions was recorded at the ratio "RS : purified water" of 20 g: 100 mL - 85.3%. However, the greatest adsorption is observed at the ratio of 2 g : 100 mL – 5.1 mg/g (Fig. 3, curve No. 1), which is almost 8 times more than for the ratio of 20 g : 100 mL (Fig. 3, curve No. 5). At the same time, the degree of removal is 68%, which is 17.3% less than at the ratio of 20 g : 100 mL. At other ratios, intermediate values of the degree of removal and adsorption are observed (curves No. 2-4).

Adsorption removal of copper (II) ions from solutions showed low efficiency (*E* = 23.3%). The obtained data indicate insignificant adsorption of Cu^{2+} on the RS sorbent, which is explained both by the nature of the adsorbate and the morphology of the adsorbent after its acid-alkaline activation. It is obvious that the RS sorbent we studied exhibits cation exchange properties. The degree of affinity of Cu^{2+} ions to this sorbent is high, but there are few functional groups that ensure this affinity. In the region of low *pH* values, the degree of sorption is small, since the carboxyl and phenolic groups of the sorbent are protonated, at rather low concentrations of copper (II), hydrogen ions compete for free sorption centres.To increase the efficiency of removal of copper (II) ions, the surface of the RS was previously modified with sulphide and hydrosulphide ions. The degree of adsorption of sulphide and hydrosulphide ions on the surface of the regenerated sorbent at the modification stage was 95.7%. The adsorption capacity of the sorbent was 163.2 mg/g. X-ray diffraction studies confirm the presence on the surface of the modified sorbent of a number of peaks characteristic of Na_2S [18] (Fig. 4).
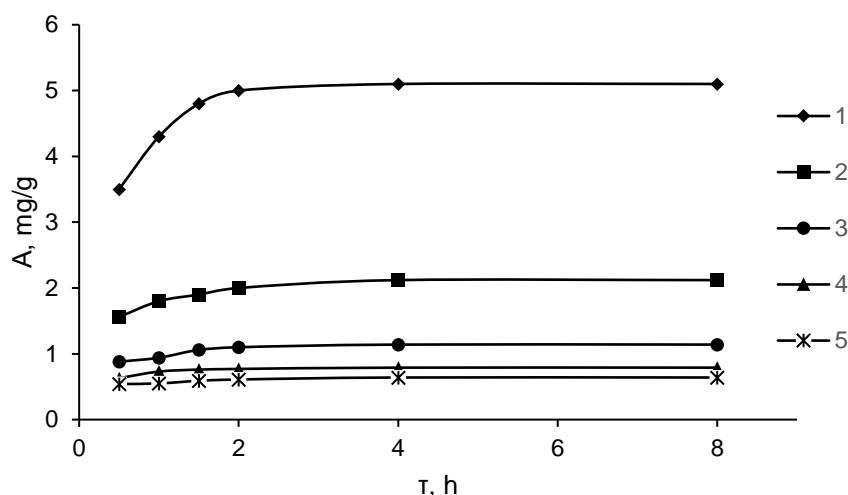
Fig. 3. Change in adsorption of Cu(II) ions over time for different doses of RS (solid : liquid ratio of: 1 - 2 g : 100 mL; 2 - 5 g : 100 mL; 3 - 10 g : 100 mL; 4 - 15 g :100 mL and 5 - 20 g : 100 mL).
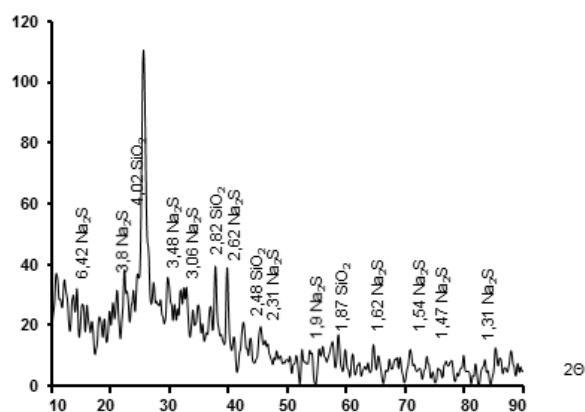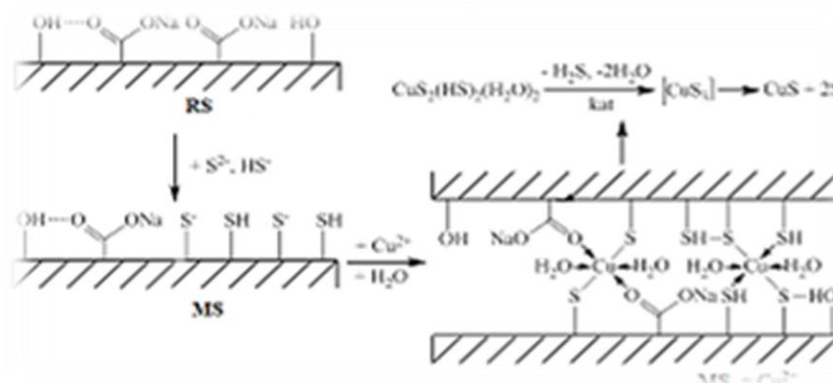

Fig. 4. X-ray diffraction pattern of Na_2S on the surface of the RS.

Modification of the surface with more active sulphide and hydrosulphide ions leads to a significant increase in its adsorption relative to copper (II) ions. Adsorption begins with the formation of a sorption complex between the sorbate (Cu^{2+}) and the primary activated centre of the sorbent. Physic-chemical modification of the matrix surface takes place through the formation of the $CuS_2(HS)_2(H_2O)_2$ coordination centre [10,17,18] (Scheme 1).

The type of interaction and the products formed depend on the chemical structure of the sorbent surface, *pH* of the environment and some other factors [21]. The possible catalytic effect of activated carbon on the passage of topochemical transformations on the surface of the sorbent was taken into account. This will cause a change in the valence-coordination node. At the same time, polysulphides CuS_x (x = 2,3) will be formed, with subsequent disproportionation to copper (II) sulphide and elemental sulphur [22].

Scheme 1. Physic-chemical modification of the RS surface and adsorption of Cu^{2+} ions on MS.

X-ray studies have established the ability to undergo topochemical transformations. X-ray data confirm the amorphous state of activated carbon and the presence in the left part of the diffractograms of the main peak of SiO_2 crystallite, located at 26° and corresponding to the original and regenerated mixture of sorbents [9]. X-ray studies confirm the presence of copper (II) sulphide and elemental sulphur on the surface of the modified sorbent after removal of Cu (II) ions from water (Fig. 5).

The possibility of undergoing topochemical transformations was established by IR-spectral studies. In the studied samples, characteristic stretching vibrations of organic functional groups were observed: −OH, −C(=O)OH, −C(=O)O− and stretching vibrations of chemical bonds Si−O, C−S, Cu−S (Tab. 2). Associated, blurred stretching vibrations of the −OH group were observed in the spectra of all studied samples in the interval 3410−3110 cm^{-1}.
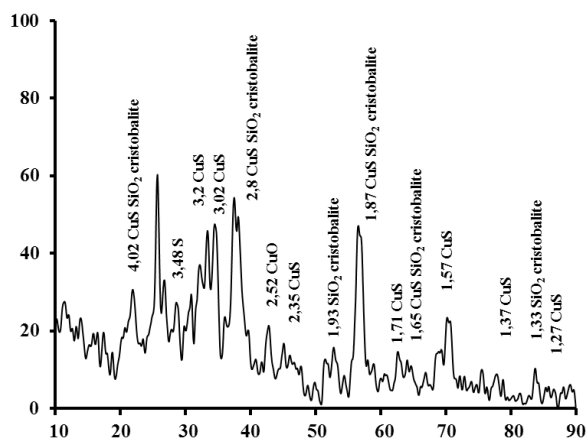
Fig. 5. X-ray diffraction pattern of CuS and S compounds on the surface of the MS after removal of Cu (II) ions.

Table 2. Characteristic fluctuations of functional groups of IR-Fourier diffuse reflectance spectra.

Sample	Fluctuations of functional groups in IR-Fourier spectra, cm^{-1}				
	v (OH)	v (−C(=O)O)	v (Si−O)	v (C−S)	v (Cu−S)
RS	3410 3196	1510	1045	−	−
MS	3390	1550	1045	744	−
MS + Cu^{2+}	3110	1550	1045	770	590

There are no strong stretching vibrations of the carboxyl group in the spectra −C(=O)OH in the interval 1725−1700 cm^{-1}. Instead, fluctuations in the average intensity of the carboxylate anion −C(=O)O− were observed in the range 1550−1510 cm^{-1}. This fact logically corresponds to the activated surface of the regenerated sorbent after its treatment with NaOH solution. In the IR spectra, there is a very strong and constant absorption at 1045 cm^{-1}. This corresponds to the fluctuations of the Si−O bond and indicates the inertness of the Bekogur brand regenerated sorbent during topochemical transformations. In many works, it is noted that activated carbon acts as an active catalyst of topochemical reactions on a solid surface [23,24]. The last two samples in the IR spectra have weakly intense stretching vibrations of the bonds *v* (C−S) and *v* (Cu−S), respectively, at 770 − 744 cm^{-1} and 590 cm^{-1}.

The results of the study showed that the amount of adsorption of copper (II) ions from model solutions with a concentration of 150 mg/L on the regenerated sorbent is about 3.5 mg/g. The use of this method is impractical without additional surface modification of the regenerated sorbent matrix.

The use of a sorbent modified with sulphide and hydrosulphide ions made it possible to increase the adsorption of copper (II) ions to 229.3 mg/g. This fact is explained by the occurrence of chemical reactions on the surface of the modified sorbent.

In the case of removal of copper (II) cations by a regenerated sorbent, their sorption is mainly carried out by carboxyl groups, but there are few of these groups. When using a modified sorbent - due to sulphide-hydrosulphide sorbed fragments. Modification of the surface of the RS with sulphur-containing ions (S^{2-}, HS^{-}) made it possible to increase the removal of copper (II) cations by 65.5 times. Therefore, for more complete sorption removal of Cu (II) ions, it is recommended to modify sorption materials, which include activated carbon and kieselguhr, with sulphide and hydrosulphide ions.

The obtained sorbent with sulphur and copper (II) sorbed on its surface can be used as a component of special purpose plastic lubricants [10,25]. There is a high probability of formation of CuS$_2$ in the friction process [17].

The obtained results can be successfully used in practice, both for the purification of water from copper (II) ions, and for the production of a component of special purpose plastic lubricants.

The obtained research results allow the effective use of modified forms of regenerated sorbents of the food industry to remove copper (II) ions from water. The research results can also be used in complex water purification from Cu^{2+}, S^2, HS^{-} ions by the sorption method.

4. Conclusions

The effectiveness and prospects of using food industry waste sorbents as secondary industrial raw materials for removing copper (II) ions from water were studied. The feasibility of using RS based on activated carbon and kieselguhr to remove copper (II) ions from water has been established. The studied technique is based on the sorption of Cu^{2+} cations by a regenerated sorbent modified with sulphide and hydrosulphide ions. Sorption of copper (II) cations is carried out by interaction with sulphide and hydrosulphide fragments on the surface of the sorbent. Physicochemical modification of the surface of the matrix occurs through the formation of the coordination centre CuS$_2$(HS)$_2$(H$_2$O)$_2$ and is completed by topochemical reactions with the formation of copper (II) sulphide and elemental sulphur. The ability to undergo topochemical transformations was established by X-ray and IR-spectral studies. The use of MS increases the removal of copper (II) cations from the studied solutions by 65.5 times.

Author Contributions

Olha S. Khudoiarova − Conceptualization, Data Curation, Methodology Writing − Original Draft, Writing − Review & Editing. Oleg A. Blazhko - Formal Analysis, Validation. Alina V. Blazhko - Data Curation, Writing − Review & Editing.

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