Effective use of slag as a product of the CFBC technology to purify water environment of copper ions

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Research Article

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Abstract

The world of today is faced with many problems related to global pollution of the aquatic environment, including groundwater, rivers, lakes, bays, seas and oceans with substances harmful to ecosystems and living organisms. Water plays a major role in maintaining life, hence its quality is crucial in the entire food chain. One of the greatest threats to the aquatic environment is industrial wastewater containing harmful heavy metals and other toxic substances. For this reason, it is necessary to search for new, cheap and clean technologies for removing metals from wastewater. In this study, slag was used, which is a waste obtained in the modern and clean circulating fluidized bed combustion technology (CFBC), considered to be the most effective method of incineration of municipal sewage sludge. The physical and chemical properties of the slag material were characterized, such as grain composition, bulk density, SEM-EDS analysis, thermogravimetric measurements, BET adsorption and analysis of SEM and TEM images. Then, adsorption properties of slag in relation to Cu(II) ions in aqueous solutions were investigated. The research results showed high adsorption process efficiency and adsorption capacity. On the basis of the obtained results, it was found that the slag waste generated in the CFBC technology may be a cheap and highly effective adsorbent to be used in adsorption processes to remove heavy metals from wastewater.

Introduction

Intensive economic development, industrialization, emerging new technologies or excessive consumptionism significantly contribute to degradation of the natural environment. Therefore, challenge for modern world is to stop the expansion of pollution, search for clean technologies and, at the same time, effectively treat industrial wastewater to remove toxic substances such as heavy metals. Industrial wastewater containing heavy metals and other harmful substances most often ends up in water bodies, including rivers, lakes, seas and oceans, thus increasing the amount of pollution (Kalak et al. 2019; Renu et al. 2017). Their source of origin can be various industries such as refineries, petrochemicals, chemical industry (fertilizer production), pulp and paper industry and others (). According to the World Health Organization (WHO), copper, cadmium, chromium, lead, mercury and Nickel are the most toxic metals (WHO 2006). Heavy metal contamination and its bioaccumulation in human tissues and organs can cause many serious health problems, such as cancer and brain damage, tumors of other organs, and diseases of respiratory, circulatory or nervous systems (Briffa et al. 2020).

Copper is one of heavy metals with a wide industrial use and, at the same time, the most common pollutant of the aquatic environment. It was found in wastewater and first listed by the United States Environmental Protection Agency (USEPA) in 1978 (Mukhopadhyay 2008). Copper is obtained and processed in many industries, including tanneries, mining, pyrometallurgical processes, surface treatments, etc., Copper is soluble in aqueous solutions in the form of divalent Cu(II) metal, which appears to be more poisonous than the metal itself. The ionic form is able to be absorbed by living organisms, it can accumulate and cause many harmful health effects (Taylor et al. 2020; Kalak and Cierpiszewski 2018). Considering the dangers associated with the presence of this metal in the environment, the USEPA agency recommends a maximum contamination level (MCL) of 1.3 mg/L (Taylor et al. 2022).

Current global trend is proper waste management in line with circular economy and sustainable development. According to this idea, it is necessary to eliminate the negative impact on the environment by limiting waste storage and as well its reuse, or by reducing the mass and neutralizing it in incineration processes. Neutralization of sewage sludge by incineration is an alternative method due to limitations in the use of sewage sludge due to the excessive content of heavy metals. The method using heat with energy recovery is seen as the most effective method of neutralizing sewage sludge due to its energy and environmental benefits (Malerius and Werther 2003). The most important benefits include the following: significant reduction of waste mass, energy use of sewage sludge waste treatment, low susceptibility to changes in the composition of waste, system stability, minimization of odors, the possibility of using by-products fly ash and slag as a filter material or as an addition to building materials, including concrete, asphalt, bricks, concrete blocks and hollow bricks. The limitations include formation of by-products, quantity and quality of which depend on the chemical composition of sewage sludge, as well as combustion conditions and flue gas cleaning technology, high operating costs and high costs of building an incineration plant (Fytili and Zabaniotou 2008). Among the many combustion methods, the circulating fluidized bed combustion (CFBC) technology is the most technologically advanced solution. In principle, only the advantages of this technology can be mentioned, for example: compatibility with a wide range of fuels (gas, oil, high and low grade coal, biomass, sewage sludge, plastic waste, used tires), low pollution, high combustion efficiency and energy efficiency, possibility of simultaneous combustion of dehydrated and dried and fermented sludge and ease of maintenance of installation (Yuansheng and Mengshu 2021).

The aim of this research study was to examine the possibility of Cu(II) ions adsorption on slag (CFBC-S) that was generated as a result of sewage sludge incineration using the circulating fluidized bed combustion technology (CFBC). Physicochemical properties of the by-product as well as the impact of adsorbent dosage, contact time, initial pH, initial concentration on the adsorption efficiency and adsorption capacity were investigated. Furthermore, adsorption kinetics and isotherms were studied.

Materials And Methods

Slag (CFBC-S) preparation, characterization and Cu(II) adsorption

Slag (CFBC-S) was obtained as a result of sewage sludge incineration using the circulating fluidized bed combustion technology (CFBC) in one of the sewage treatment plants in Poland. The CFBC-S samples were taken from a fluidized bed reservoir and prepared according to standards PN-EN 15002:2006 and PN-EN 14899:2006. Next, they were dried at 105°C to constant weight. All chemicals were analytically pure and distilled water was used in the research.

CFBC-S samples were sieved and those with a diameter less than 0.212 mm were used in experiments. Their physicochemical properties were determined as follows: granulation analysis, particle size distribution, bulk density, the elemental composition using SEM-EDS analysis, specific surface area and pore diameter (BET), pore volume (BJH), SEM and TEM analysis, FT-IR. The research methods as well as procedure of Cu(II) adsorption processes were described and attached as supplementary material (SM Methods).

Results And Discussion

Characterization of the adsorbent

Granulation analysis was performed and the results are as follows: 0–0.212 mm – 13.1%; 0.212–0.5 mm – 73.8%; 0.500–1.0 mm – 11.2%; 1.0–1.7 mm – 0.51%; > 1.7 mm – 1.39%. Based on these studies, it was found that the CFBC-S particles are heterogeneous and their diameter influences the course of adsorption. According to literature reports, smaller slag particles are characterized by a larger specific surface area and the number of active centers, which translates into higher efficiency of sorption processes of metal ions (Kostura et al. 2017). These data influenced the decision to use the smallest fractions with a diameter less than 0.212 mm.

The analysis of particle size distribution showed one peak at the particle size of 955.4 nm (Fig. SM1, supplementary material). The analysis was difficult to carry out because some of slag particles did not suspend in the aqueous solution, and larger ones fell to the bottom of the suspension. Hence, it was only possible to analyze suspended samples.

Bulk density measurements were carried out by loosely filling slag into a cylinder and by compaction on a vibrating table. The results were estimated at 0.82 and 1.34 g/cm^3, respectively. The results of the analysis may be useful from the point of view of industrial application in building and construction materials.

Elemental analysis using the SEM-EDS method was performed and the results are shown in Ta ble 1 and Fig. SM2 (supplementary material). As it is seen, slag mainly includes following elements: Ca, O, P, Si, Al, Mg, Fe, C. The method is based on a spot measurement on the sample surface. Slag is a complex mixture, hence the quantitative and qualitative composition may slightly differ in different places of the agglomerates due to the location of the measuring point.

Table 1
Elemental composition of CFBC-S (SEM-EDS analysis)

Elements	**C**	**O**	**Na**	**Mg**	**Al**	**Si**	**P**	**S**	**K**	**Ca**	**Ti**	**Mn**	**Fe**
CFBC-S [%], weight	1.33	43.8	0.74	2.51	3.54	3.74	14.2	0.46	0.19	27.1	0.44	—	1.89
CFBC-S [%], atomic	2.5	61.6	0.72	2.33	2.95	2.99	10.3	0.32	0.11	15.2	0.21	—	0.76
Oxides	CO_2		Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	K_2O	CaO	TiO_2	MnO	Fe_2O_3
CFBC-S [%]	4.89		1.0	4.17	6.68	7.99	32.48	1.14	0.23	37.98	0.73	-	2.71

The BET and BJH analysis revealed the following results: specific surface area 1.87 m^2/g, pore volume (V_p) 0.0096 cm^3/g and mean pore diameter (A_{pd}) 21.2 nm. The shape of the obtained adsorption isotherms resembles the type III isotherm, which is related to cooperative adsorption (Figs SM3 – SM6, supplementary material). This means that previously adsorbed particles can lead to increased sorption of the remaining particles. The interaction of copper ions with the slag adsorbent has a positive effect on the adsorption of remaining copper ions when the ions have already been adsorbed at least once. The result is a bulging isotherm towards the pressure axis.

SEM images of CFBC-S particles are presented in Fig. 1. The slag particles are irregular in shape, compact, spongy and have a porous surface. The irregular shape depends on temperature and duration of a combustion process. More crystalline and spherical particles result from a longer process (Liu et al. 2014). Figure 2 presents TEM images of the samples showing flocs of irregular shape and different sizes. The particles are of different shades, the darker zones correspond to the thicker material. Similar observations were found in the literature (Li et al. 2011; Assi et al. 2020).

FT-IR studies of CFBC-S before and after Cu(II) ions adsorption were performed and the spectra are shown in Figure SM7 (supplementary material). To this purpose, sorption frequencies of functional groups, functional bonds and types of vibrations were identified. As it is seen, there is an increase in the intensity of peaks after Cu(II) adsorption observed. The following peaks have been observed: 593, 416, 406, 390 cm^{-1} (bending vibrations Si-O-Si), 677, 611 cm^{-1} (stretching vibrations Al–O), 713 cm^{-1} (symmetric stretching of Si–O–Si and Al–O–Si). 874 cm^{-1} (symmetric stretching of Al–O–M, vibration of carbonates (calcite)), 113 cm^{-1} (asymmetric stretching vibrations of silica Si–O–Si and Al–O–Si), 1408 cm^{-1} (valence vibration of carbonate ions), 3252 cm^{-1} (stretching vibrations O–H), 3643 cm^{-1} (asymmetric and symmetric stretching vibrations O–H (probably amorphous silicates or hydrated aluminum silicates)). The wide band at around 3600 – 3200 cm^{-1} appeared after, but the weak sharp peak at 3643 cm^{-1} disappeared. The peaks at 594, 611, 677, 713, 874, 1113, 1408 cm⁻1 became more intensive due to a probable complexation reaction and formation of surface complexes with Cu(II) ions or bonds with the metal ions (Cu–O) (Kavaliauskas et al. 2015; Ueda et al. 2000; Iliashevsky et al. 2016).

Adsorption studies

Effect of initial pH

The impact of initial pH on the adsorption efficiency was analyzed and the results are shown in Fig. 3 and (Fig. SM8, supplementary material). The following conditions were applied in the experimental procedure: initial pH range of 1.8–5.6, initial concentration of Cu(II) ions 100 mg/L, adsorbent dosage 1–5 g/L, contact time 60 min, temperature 23°C, agitation speed 200 rpm. As it is seen, high adsorption efficiency was reported at initial pH 1.8 for the slag doses of 1 g/L (86.6%), 3 g/L (90.7%), 5 g/L (98.8%). An increase in initial pH resulted in a gradual decrease in adsorption efficiency. The experimental adsorption capacity also reported a stable decrease in the range of 19.5–14.7 mg/g. In considering the influence of the initial pH, the surface charge of the slag and the degree of speciation should be taken into account. The tested adsorbent is alkaline in nature, therefore it may increase the pH in an aqueous solution during adsorption. The presence of such anions in the adsorbing material as SiO_3^{2-}, CO_3^{2-}, PO_4^{3-}, OH^-, SO_4^{2-} may contribute to the precipitation of copper ions from the solution at higher pH. The pH parameter influences electrostatic charge of the metal oxides present in the adsorbent material. Hence, it is highly probable that the adsorption of Cu(II) ions may be associated with ion exchange and/or complexation by bonding with oxygen groups. The probable ion exchange mechanism between H^+ and Cu^{2+} ions can be proposed by the equations 1–3.

$$XOH + H_3O^+ \leftrightarrow XOH_2^+ + H_2O$$

1

$$XOH + OH^- \leftrightarrow XO^- + H_2O$$

2

$$2\left(XO^-\right) + Cu^{2+} \leftrightarrow (XO)_2Cu$$

3

where: X may be Fe, Si, Al or another element. It should be pointed out that the proposed mechanism was not confirmed by additional experiments in this research (Kalak and Cierpiszewski 2019).

Effect of sorbent dosage

The impact of CFBC-S dosage on the adsorption of Cu(II) has been studied and the results are presented in Fig. 4. The experiments were conducted under following conditions: initial pH 1.9, initial concentration 100 mg/L, contact time 60 min, temperature 23°C, agitation speed 200 rpm. The results showed a rapid increase in adsorption efficiency up to 98% with the use of slag dosage 0.25–5 g/L. The dose of 5 g/L is considered optimal under these experimental conditions. The addition of higher doses of CFBC-S did not cause any significant changes and the process efficiency remained at the same level. Moreover, analysis of adsorption capacity revealed that: firstly, it increased up to 2.58 mg/g (dosage 3 g/L) and secondly, it gradually decreased to 0.4 mg/g at a dose of 25 g/L (Fig. SM9, supplementary material). At higher slag doses, the active centers available for further adsorption were not fully utilized, hence a decrease in sorption capacity was observed (Liu et al. 2014). Consequently, the optimal experimental adsorption capacity can be estimated in the range between 2.0 and 2.6 mg/g.

Effect of initial concentration of Cu(II) ions

The influence of the initial concentration of Cu(II) ions was investigated and the results are shown in Fig. 5. Based on the previous research results, the following experimental conditions were used: initial concentration of Cu(II) (2.5–100 mg/L), adsorbent dosage 2–5 g/L, initial pH 1.9, contact time 60 min, agitation speed 200 rpm, T = 23°C. The adsorption curves show an upward trend in all cases of slag doses. Higher adsorption efficiencies were obtained at the initial concentration of 100 mg/L (98.2% – 5 g/L of slag, 91.8% – 4 g/L, 89.0% – 3 g/L, 87.9% – 2 g/L). An increase in experimental adsorption capacity was also observed (Fig. SM10, supplementary material). This is due to the fact that free active sites were still available, and their total available amount influenced the adsorption efficiency.

Effect of contact time

The research results on the influence of contact time on adsorption efficiency and adsorption capacity are shown in Figs. 6 and 11 and Figure SM11 (supplementary material). The study of the contact time in adsorption is important from the point of view of potential industrial use. Determining optimal contact time can help to design processes efficiently and reduce costs. The following experimental conditions were used in these studies: initial concentration of Cu(II) ions 100 mg/L, initial pH 1.9, slag dosage 1–5 g/L, temperature 23°C, agitation speed 200 rpm. As shown in Fig. 6, the maximum level of adsorption efficiency was achieved in the range of 20–30 minutes of the process and no changes were observed until 60 minutes, hence there was no need to continue the experiment for a longer time. An increase in adsorption efficiency in the first stage may be a consequence of the availability of a large number of free active sites on slag surface and a high concentration of Cu(II) ions at the interface. The gradual occupation of active centers by metal ions during mixing contributed to equilibrium in the system.

Kinetic models

The kinetics of Cu(II) adsorption on CFBC-S was analyzed with pseudo-first order (PFO) and pseudo-second order (PSO) models. The calculation results (reaction rate constant k, equilibrium adsorption capacity q_e and correlation coefficients R^2) are presented in Table 2, and the plots are shown in Figures SM12 and SM13 (supplementary material). The performed calculations showed that higher coefficients R^2 were recorded in the case of PSO model, which is related to greater correlation between the experimental q_e and the calculated q_t values. Therefore, it can be concluded that the kinetics of Cu(II) adsorption on CFBC-S is better described by the PSO model. Chemisorption and electrostatic attraction on the adsorbent surface could take place during these processes.

Table 2
Kinetic parameters of pseudo-first order and pseudo-second order models

Adsorbent	Adsorbent dosage [g/L]	PFO kinetic model			PSO kinetic model		
		k_{ad} [min^{-1}]	q_e [mg/g]	R^2	k [g/mg min]	q_e [mg/g]	R^2
CFBC-S	3	0.134	9.449	0.971	0.008	24.110	0.999
	4	0.130	10.223	0.934	0.010	22.332	0.999
	5	0.118	7.229	0.958	0.012	19.711	0.999

Isotherm models

Langmuir and Freundlich isotherms were used to analyze the studied adsorption process. The calculations of isotherm parameters are shown in Table 3 and the isotherms are included in Figures SM14 and SM15 (supplementary material). Based on the Langmuir equation, the maximum adsorption capacities were calculated and are as follows: 53.04 mg/g (CFBC-S dosage 2 g/L), 56.05 mg/g (3 g/L), 60.92 mg/g (4 g/L) and 70.34 mg/g (5 g/L). According to the calculated correlation coefficients R^2, adsorption reactions carried out in these studies are more closer to the Freundlich model than to the Langmuir model.

Table 3
Parameters of Langmuir and Freundlich isotherm models

Adsorbent	Adsorbent dosage [g/L]	Langmuir isotherm			Freundlich isotherm		
		Calculated q_m [mg/g]	K_L [L/mg]	R^2	K_f [mg/g] [L/mg] (1/n)	n	R^2
CFBC-S	2	53.04	0.049	0.824	1.315	0.986	0.933
	3	56.05	0.037	0.888	1.213	0.938	0.938
	4	60.92	0.031	0.910	1.171	0.906	0.940
	5	70.34	0.019	0.929	1.049	0.964	0.950

Conclusions

In this study, slag (CFBC-S) obtained as a result of incineration of municipal sewage sludge with the use of the circulating fluidized bed combustion (CFBC) technology was used. In the first stage, selected physicochemical properties of the adsorbent were characterized using various methods. In the second stage, copper ion adsorption experiments were carried out and the influence of such factors on the process efficiency as initial pH, adsorbent dosage, initial concentration of Cu(II) ions and contact time were examined. As a result of the research, high adsorption efficiency of 98.8% was reported under the following conditions: adsorbent dose 5 g/L, initial concentration of Cu(II) ions 100 mg/L, initial pH 1.9, T = 23°C, contact time 60 min. Many experiments have shown that it is possible to achieve high process efficiency at the level of at least 90%. In the third step, the adsorption kinetics and isotherms were analyzed. It turned out that the investigated sorption processes are more closer to the pseudo-second-order kinetic model and to the Freundlich model.

Summarizing, it can be stated that slag obtained as a result of CFBC technology can be successfully used to remove copper from aqueous solutions. Slag is industrial waste that can be reused as a product, which is in line with the promoted zero waste policy. This adsorbent can be proposed for potential use in wastewater treatment plants for the treatment of industrial and municipal effluents.

Declarations

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Figures

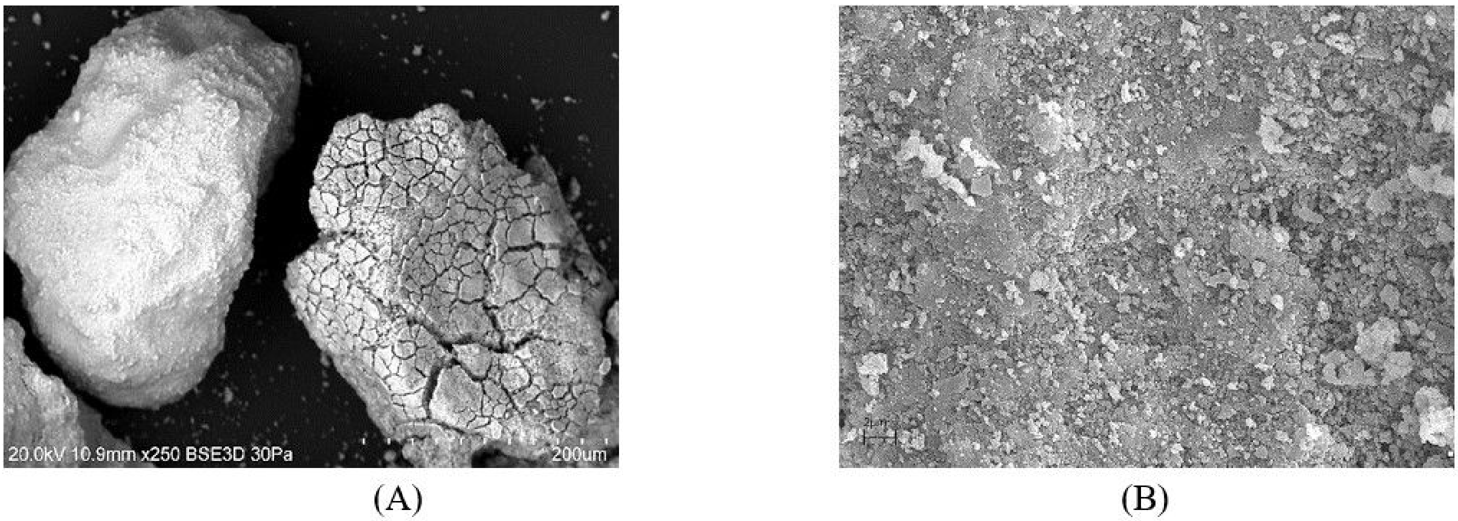

Figure 1

SEM images of CFBC-S (magn.: x200, scale bar: 200 μm (A); magn.: x20000, scale bar: 2 μm (B))

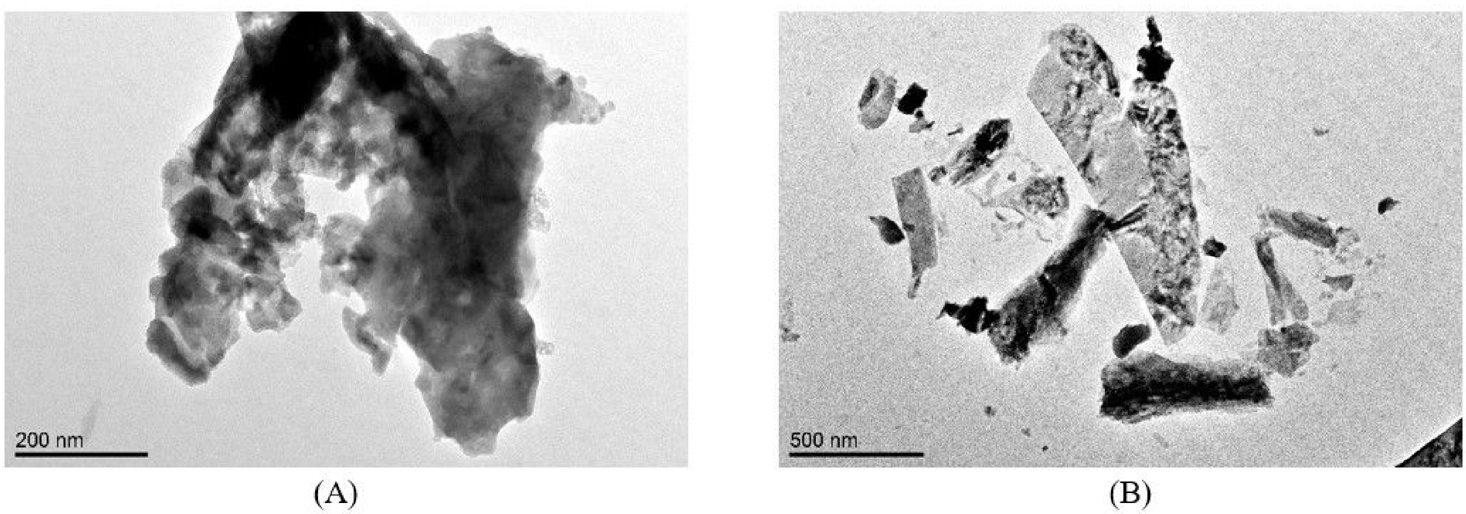

Figure 2

TEM images of CFBC-S (scale bar: 200 nm (A); 500 nm (B))

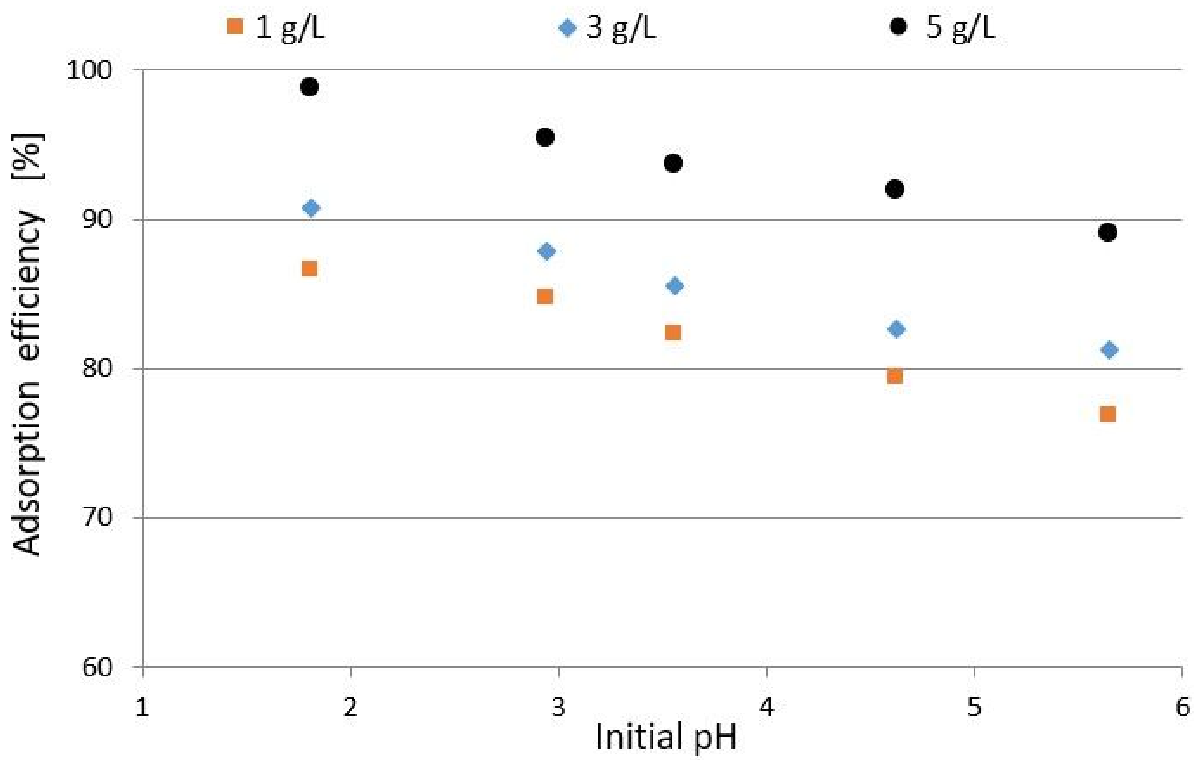

Figure 3

The impact of initial pH on adsorption efficiency of Cu(II)

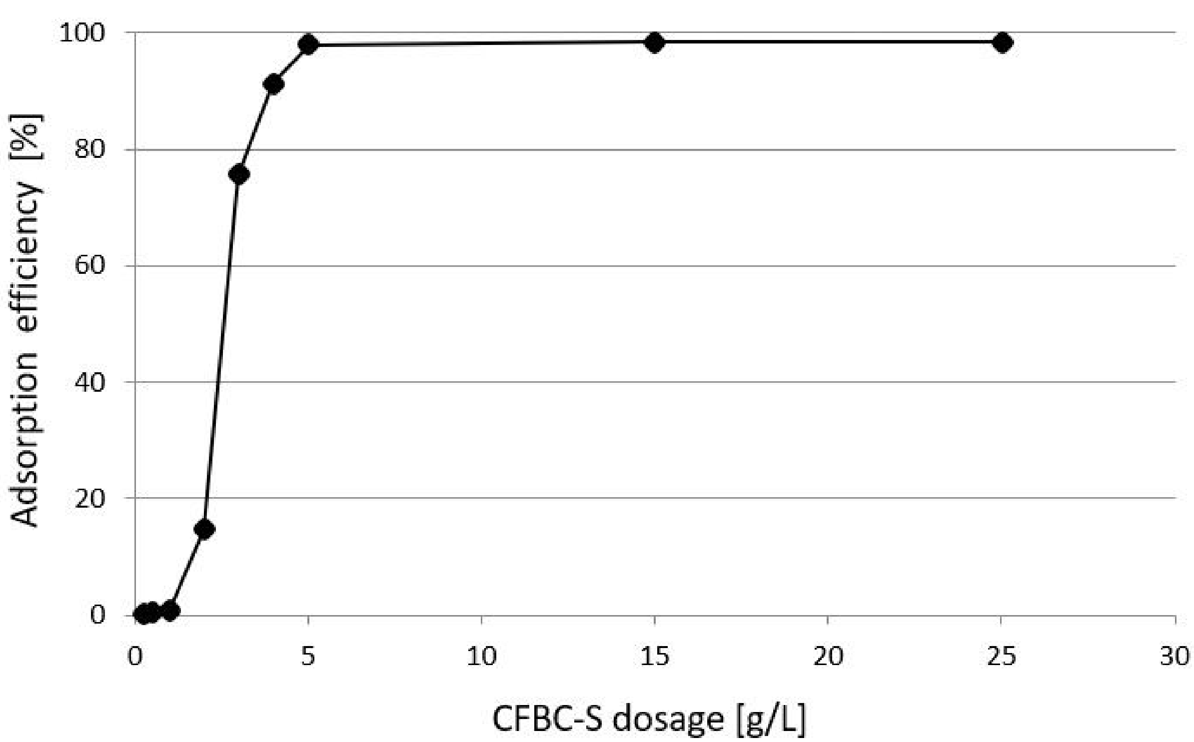

Figure 4

The impact of adsorbent dosage on adsorption efficiency of Cu(II)

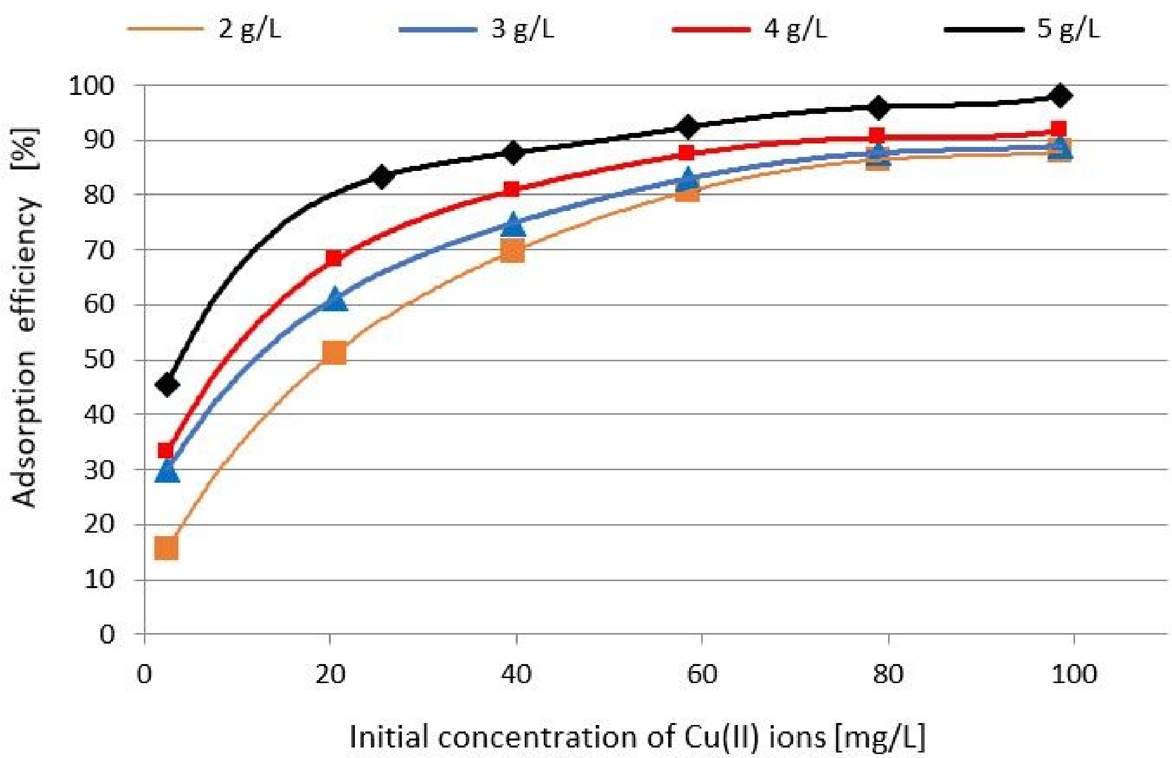

Figure 5

The impact of initial concentration of Cu(II) ions on adsorption efficiency

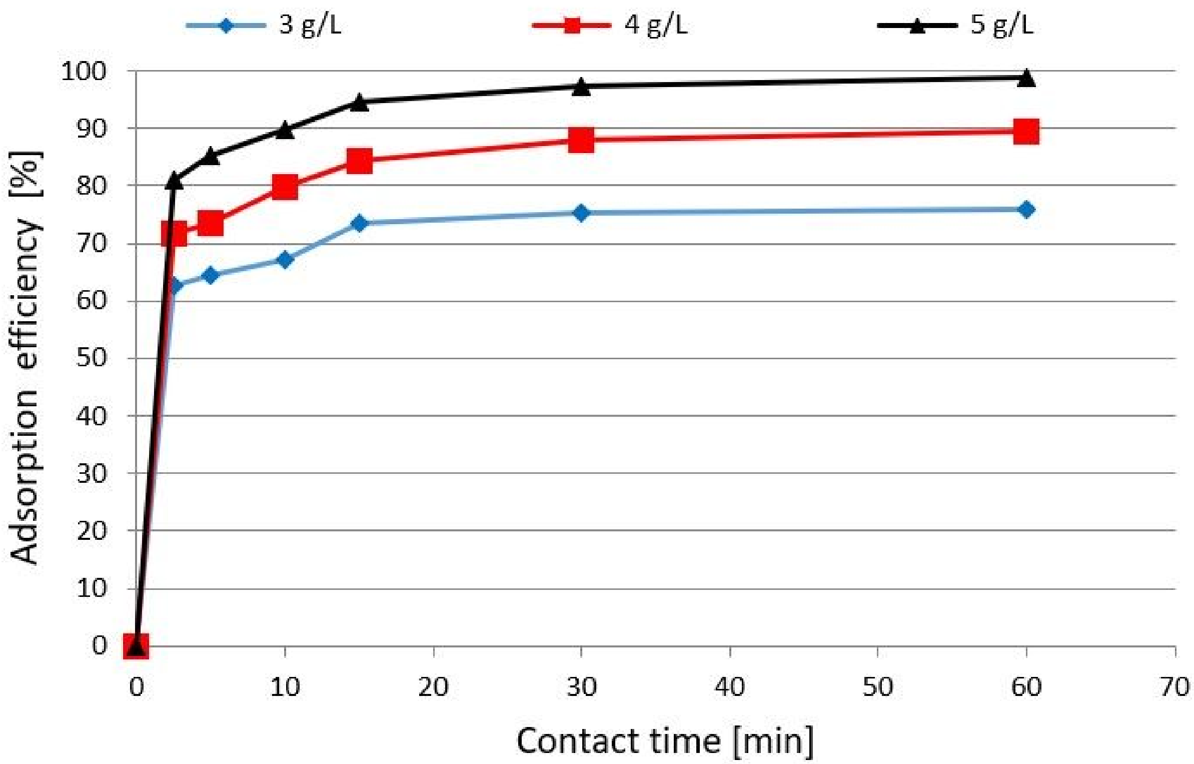

Figure 6

The impact of contact time on adsorption efficiency of Cu(II) ions

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