The Use of Waste From Bauxite Ore in Sorption of 3,5-Dichlorophenol From Waste Water

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Usage of the low-cost sorbent for the removal of 3,5-dichlorophenol from the wastewater was investigated. Industrial solid waste (red mud) was selected as a low-cost sorbent. The effects of adsorbent dose, contact time, and temperature on sorption process were monitored. The initial concentration of the monitored pollutant was 4 mmol.L^{-1}. The results show that the mass of the adsorbent and the contact time with the sorbate has a relatively high impact on the sorption process. However, the temperature affects the sorption at a lower level. The highest efficiency of sorption 95.62% was achieved by the red mud (amount 2.5 g) after 48 hours contact with 3,5-dichlorophenol at 25 °C. At the temperature of 50 °C (amount of red mud 2.0 g) the efficiency after 48 hours was 92.77%. Relatively high sorption efficiency of the test material makes it preferable and very attractive for the use as an alternative sorption material.

Key words: *bauxite ore, sorption; 3,5-dichlorophenol; red mud; wastewater*

Introduction

Chlorophenols are found widely in the wastewater from various industrial processes such as the manufacture of plastics, dyes, drugs, pharmaceuticals and pesticides, and are also considered as a priority and hazardous pollutants because of their high toxicity, carcinogenicity and resistance to biodegradation (Zhou et al., 2014). The consumption of phenolic-polluted waters results in central nervous system (CNS) paralysis, protein deterioration, liver, kidneys and pancreas disorders in human. Therefore, in order to protect the environment and human health, the removal of phenolic compounds from water and wastewater is essential (Nourmoradi et al., 2016).

In recent years, various methods have been proposed for the removal of phenolic compounds from aqueous solutions such as electrochemical oxidation (Polcaro et al., 1997; Cañizares, et al., 2003), emulsion liquid membrane processes (Ng et al., 2010; Park et al., 2006; Sentyakov et al., 2016), ion exchange (Lee et al., 1996), membrane filtration (Das et al., 2008), reverse osmosis (Kargari et al., 2005), photocatalytic degradation (Alimoradzadeh et al., 2012; Czaplicka, 2006), oxidation with hydrogen peroxide (Mokrini, et al., 1997), wet oxidation (Chaliha et al., 2006). An alternative for wastewater treatment is the use of adsorption process, which is efficient in the removal and recovery of persistent organic pollutants (Moreira de Oliveira et al., 2013; Hind et al., 1999; Li et al., 2016; Ciahotný et al., 2016). Many researchers have shown that activated carbon is an effective adsorbent for organic compounds removal, especially for phenolic compounds (Ahmaruzzaman, 2008). Activated carbon is the most common material that displayed good sorption capability for many organic pollutants due to its high-surface-area, pore volume, and porosity (Nourmoradi et al., 2016). However, its high initial cost and the need for a costly regeneration system make it less economically viable as an adsorbent. Taking these criteria into consideration, the search for a low cost and easily available adsorbent has led many investigators to search more economic and efficient techniques as adsorbents (Ahmaruzzaman, 2008).

Thus, this study has been undertaken with the aim to study the adsorption behaviour of an industrial waste red mud for the removal of 3,5-dichlorophenol. Red mud is a solid waste generated during the production of aluminium oxide by the Bayer process during which the bauxite is leached with sodium hydroxide at

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an increased temperature and pressure (Zavastin et al., 2012). Red mud is considered as major industrial hazardous waste that causes environmental problems and generates multiphase problems in the society. The utilisation of red mud is realistically a significant problem in the alumina industry (Samal et al., 2013). The integrated utilisation of red mud has thus been intensively investigated, especially in terms of construction materials like cement, landfill etc. (Wanchao et al., 2014). The waste materials obtained from the alumina industries and thermal power plant need necessary treatment and confined disposal to manage them properly. Each year, about 90 million tonnes of red mud are produced globally. Red mud is a highly alkaline waste material with pH 10–12.5 mainly composed of fine particles containing aluminium, iron, silicon, titanium oxides and hydroxides. Due to the alkaline nature and the chemical species present in red mud, this solid waste causes a significant impact on the environment, and proper disposal of waste red mud presents a huge challenge where alumina industries are installed (Pirkanniemi et al., 2002; Ordóñez et al., 2002).

Red mud is produced during the Bayer process for alumina production. Bauxite ores are usually a mixture of minerals rich in hydrated aluminium oxides. However, they also contain iron, silicon and titanium minerals. After the digestion of bauxite ores with sodium hydroxide at elevated temperature and pressure, aluminium oxide is dissolved in the solution, and the solid residue is red mud. The amount of the residue generated, per tonne of alumina produced, varies greatly depending on the type of bauxite used, from 0.3 tonnes for high-grade bauxite to 2.5 tonnes for the very low grade (Legube et al., 1999; Xu et al., 2001).

As red mud has strong alkalinity, which will cause some potential risks to its reuse, pre alkalinity will produce beneficial effects. In the past years, several methods have been proposed, such as acid neutralization, seawater wash treatment, heat treatment and the combination of above three treatments (Neagu et al., 2013) Acid neutralization is widely used for red mud treatment and this method can remove alkali metals and other inorganic impurities as well as some organics. It is generally found that increase the surface area and pore volume, favouring adsorption. The heat treatment can decompose unstable compounds and organics. However, it can also cause particle aggregation or sintering (Shaobin et al., 2008).

Red mud was studied as a possible low-cost material for sorption of various dangerous materials, for example, heavy metals (Grudić et al., 2013; Liu et al., 2016). Its use produce significant benefits in terms of environment and economics by reducing landfill volume, contamination of soil and groundwater, and release of land for alternative uses.

The production of red mud is associated with the production of alumina by bauxite from bauxite, mostly imported from Hungary, under the conditions of ZSNP Žiar nad Hronom about 70,000 tons of red sludge of 15 % Al_2O_3, 13 % SiO_2, 45 % Fe_2O_3, 6 % TiO_2, 2 % CaO, 7.5 % Na_2O, residual annealing loss (Fig. 1). This mud is in dusty form, making its negative impact on the environment even more pronounced (Soldán et al., 2014).

Fig. 1. Chemical composition of red mud.

Material and methods

Preparation of adsorbent

Red mud was obtained from a waste pond in Žiar nad Hronom in Slovakia. The main objective of the pre-treatment of red mud samples was to remove possible organic impurities, which could negatively affect the sorption properties of the sorbent. Sorbent sample was annealed in a muffle furnace VEB Electro Bad Frankenhausen LM 312.11 at temperature 500 °C during 2 hours followed by cooling in a desiccator. The obtained sample of red mud was then divided into fractions (< 500 μm) using sieve analysis.

Surface morphology and elemental characterisation

Because the chemical composition, porosity, particle size and thus the specific surface area of sorbents, affects sorption process a topographic, elemental and chemical character of sorbents was obtained by using

scanning electron microscope (SEM) of JEOL JSM 7600 F with FEG cathode with detector Energy-dispersive X-ray spectroscopy (EDX) of Oxinst MAX 50. The topography of the red mud was observed at an accelerating voltage 20 kV, a current of 2 nA and a working distance of approximately 15 mm. The chemical composition of the sorbent was carried out using the software INKA at a magnification of 1000.

Fourier Transform Infrared Spectroscopy

FT-IR spectrums of the samples were obtained using a Fourier Transform Infra-Red Spectrometer (FTIR) of Varian 660 MidIRDual MCT/DTGS Bundle. The ATR equipment for measuring solid particles of red mud was used. Spectrum was measured 100 times at a resolution of 4 cm^{-1}, with a filter 1.28 kHz and a rate of 5 kHz, recorded and evaluated by using software with the library VarianResolutionsPro Sadtler Canadian forensics.

Adsorption experiments

The sorption effectiveness of 3,5-dichlorophenol on alternative adsorbent (red mud) was studied. Also, the effect of sorption conditions (Tab. 1) on its course was evaluated.

Tab. 1. Monitored sorption parameters of 3,5-dichlorophenol removal.

The amount of sorbent [g]	Temperature [°C]	Time [h]
0.1	25	0
0.5	30	2
1.0	35	4
1.5	40	6
2.0	45	24
2.5	50	48

The initial concentration of 3,5-dichlorophenol was 4 mmol/L, pH of the 3,5-dichlorophenol solution was 5.14 and the temperature of 25 °C. The sorption experiments were carried out using HEIDOLPH shaker Promax 1020, at a frequency of 160 T min^{-1}. The effect of temperature was investigated in MEMMERT 100-800 ensured oven. Subsequently, the samples were taken and placed into a centrifuge of type Janetzký T30 in set times of sorption for 10 minutes at a speed of 2000 to 3000 rpm in order to remove small sorbent particles, which are undesired.

For the determination of 3,5 – dichlorophenol concentration a gas chromatograph with MS detector GC-MS Agilent 5975C equipped with a capillary column (30 m x 0.250 mm - inner diameter) with a film of 0.25 µm and helium mobile phase was used. Parameters were chosen for the method of phenolitic compounds determination by the EPA Methods 82 70. Then, the percentage removal (Efficiency) was determined as shown below (expression 1):

$$Efficiency = (C_i\text{-}C_f/C_i)*100 \text{ \%} \tag{1}$$

where C_i and C_f are the initial and the final concentrations of 3,5-dichlorophenol in the solution respectively.

Results and discussion

Elemental characterisation

The chemical composition of red mud is shown in Table 2, and the values are expressed in w/w.

Tab. 2. Chemical composition used adsorbent [% w/w].

Elements	O	Mg	Al	Si	Ca	Cr	Mn	Fe	Ni
Red mud	44.85	2.90	9.04	7.15	0.81	0.83	0.21	33.75	0.46

SEM characterisation

SEM image of red mud is shown in Figure 2, and it demonstrates the positivity and surface area structure of the studied alternative cost adsorbent material.
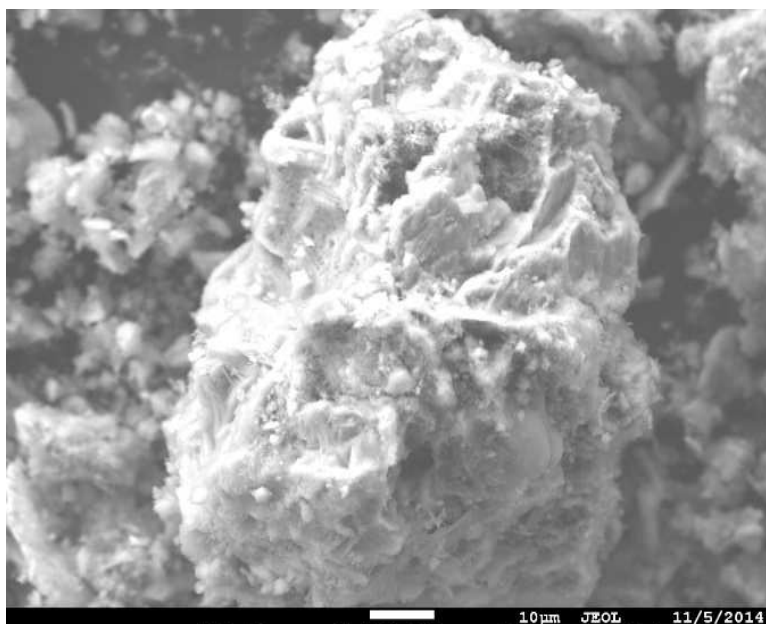
Fig. 2. Microscopic appearance of the sorbent surface (SEM) of red mud (fraction < 500 microns). Surface detail at a magnification of 1000.

FT-IR spectral analysis

Because the sorbent sample which we used was annealed, we watched the vibrations of individual functional groups in the sorbent which may change the observed sorption characteristics. In Table 3 the adsorption bands allocated to bonds in the observed spectra of the red mud sample are shown.

Tab. 3. FT-IR spectra of red mud at laboratory temperature and after annealing at a temperature of 500 °C.

Bonds	Wavenumber region [cm^{-1}]	
	RM (non-annealed sorbent)	**RM (annealed sorbent at 500 °C)**
C=O	1 440	1 433
Si-O	974	991
Al-O	874	872
O-Si-O	555	540
Fe-O	444	438
O-H, H-O-H	3 411	-

Intense absorption band was observed in the wavelength of 1440 cm^{-1}, which we can associate with the stretching vibration of the C=O bond. The primary reason for the observation of these bonds was the presence of CO_2 chemically adsorbed on the surface of the red mud. We could see that in the sample that was annealed, the intensity of adsorption bands of C=O bonds decreased as a result of released CO_2 from the surface of the red mud. The following important absorption peak at 990 cm^{-1} corresponds to the vibration of the Si-O bond. By comparison of samples, we could see that the intensity of it after annealing decreased especially due to thermal degradation of mineral compounds, e.g. $Na(AlSiO_4)$. The low absorption peak at 870 cm^{-1} corresponds with Al-O bonds. The less significant absorption band at 530 cm^{-1} was corresponding to vibrations of O-Si-O groups. At 450 cm^{-1} we could observe the absorption band characteristic vibrations of Fe-O bonds. The identified absorption band at 3600-3400 cm^{-1} corresponds with the vibrations of OH bonds respectively H-O-H molecules of the adsorbed water. Samples which were annealed lose these bonds. This is due to the evaporation of moisture during annealing (Fig. 3).
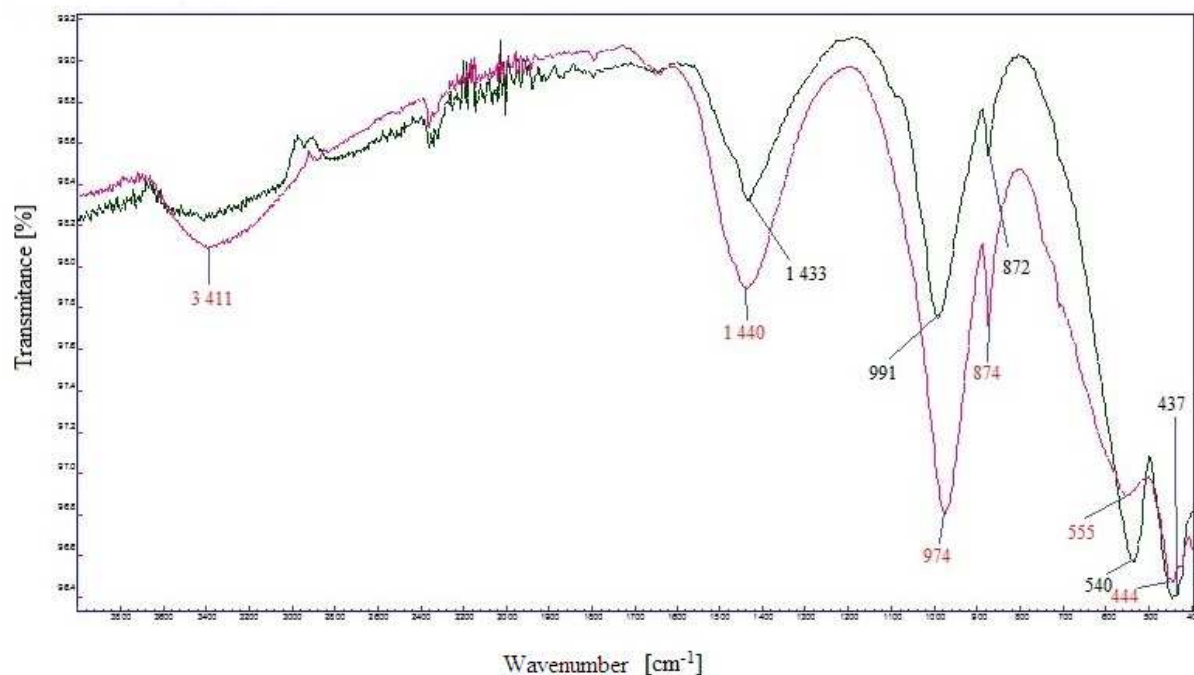
Fig. 3. IR spectrum of red mud before (red line) and after (black line) annealing.

Effect of dosage

In Figure 4, we can see the impact of sorbent mass - red mud (0.1 g, 0.5 g, 1.0 g, 1.5 g, 2.0 g, 2.5 g) on the kinetics of the sorption over 2, 4, 6, 24 and 48 hours. We can see that the sorbent mass and the contact time of sorbate and sorbent have a relatively high impact on the efficiency of sorption process. The sorption efficiency increases with the increasing time With the weight of 0.1 g, 0.5 g, 1 g, 1.5 g and during the shortest monitored period of time (after 2 hours) the efficiency of red mud reaches 17.3 %, 25.66 %, 26.30 % and 24.87 %. During the longest monitored period of time (48 hours) the efficiency of red mud reaches 58.18 %, 61.64 %, 63.84 % and 63.48 %. With the weights of sorbent at 2.0 g and 2.5 g, the sorbent efficiency was relatively high (after 2 hours - 46.38 % and 56.08 %). The course of the sorption in comparison with the previous weights was different because the increase in the efficiency of the red mud sorbent after 6 hours was no longer so significant. This could be the reason for an adsorption equilibrium. After 48 hours the efficiency of the sorption was 89.70 % and 95.62 %.
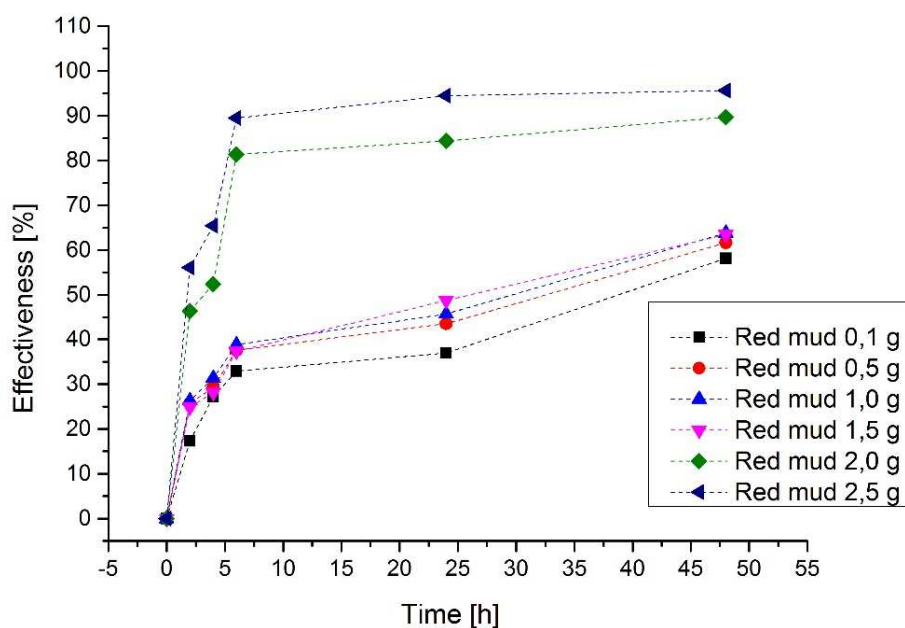

Fig. 4. Effect of dosage on % removal of 3,5-dichlorophenol.

Effect of temperature

In Figure 5, we can see the impact of the temperature (25 °C, 30 °C, 35 °C, 40 °C, 45 °C, 50 °C) on the sorption after 2, 4, 6, 24 and 48 hours at sorbent amount of 0.5 g. The hypothesis that the higher the temperature is the rate of sorption increases was confirmed. The adsorption equilibrium stabilisation has occurred earlier at a higher temperature. To compare the efficiency of the sorption and the impact of temperature on the process, sorption times of 2, 4, 6, 24 and 48 hours were chosen. Only very small differences could be observed with increasing temperature in the studied samples. Increase in the efficiency of the sorbent between each temperature was relatively low, about 0.5 – 3.0 %. After 2 hours of the sorption the efficiency at temperatures of 25 °C, 30 °C, 35 °C, 40 °C, 45 °C, 50 °C was following: 25.66 %, 28.70 %, 28.79 %, 29.28 %, 29.04 % a 29.21 %. At the longest period of time (48 hours) and at the highest reached temperature the efficiency was: 61.64 %, 62.09 %, 62.18 %, 63.53 %, 63.85 % and 64.74 %.
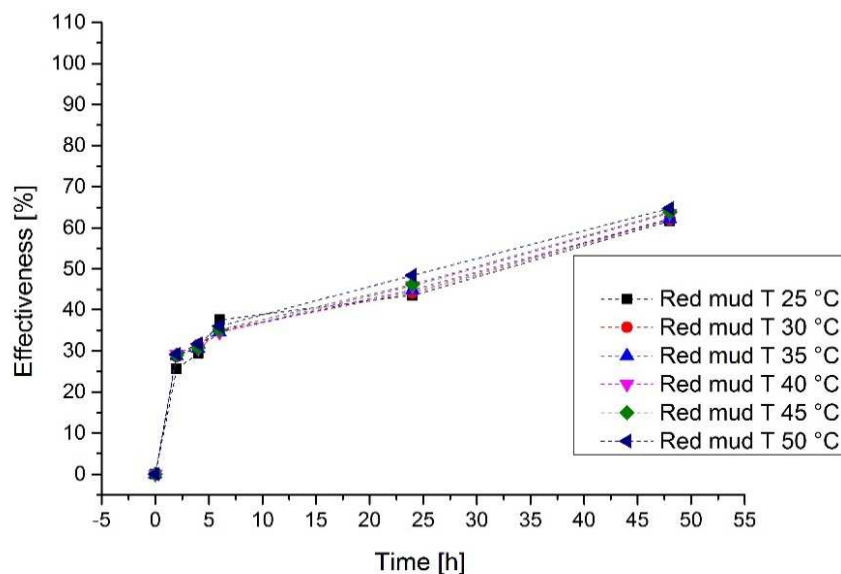

Fig. 5. Effect of temperature on % removal of 3,5-dichlorophenol (amount of the sorbent 0.5 g).

We could see that the studied samples (Fig. 6) were affected by the temperature of the sorption at the sorbent weight of 2.0 g and at 2, 4, 6, 24 and 48 hours. Increase in the efficiency at higher temperature was relatively low, in the range of 0.5 – 2.0 %. After 2 hours of the sorption the efficiency at the temperatures of 25 °C, 30 °C, 35 °C, 40 °C, 45 °C, 50 °C was following: 46.38 %, 48.91 %, 50.02 %, 50.34 %, 50.98 % a 51.36 %. At the longest period of time (48 hours) and at the highest reached temperature the efficiency was: 89.70 %, 90.69 %, 91.30 %, 91.28 %, 92.13 % and 92.77 %.
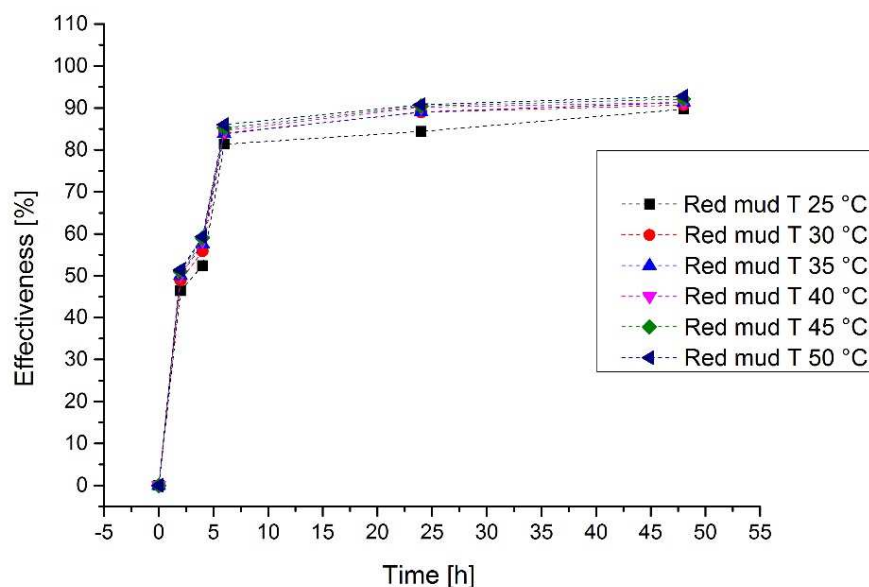

Fig. 6. Effect of temperature on % removal of 3,5-dichlorophenol (amount of the sorbent 2.0 g).

Conclusion

This study shows that the red mud could be used as an adsorbent for the removal 3,5-dichlorophenol from wastewater. Experimental data has shown that the efficiency of the sorption was significantly influenced by the amount of the sorbent (the observed amounts were 0.1 g, 0.5 g, 1.0 g, 1.5 g, 2.0 g, 2.5 g) and by the contact time of the sorbent and the sorbate (the observed times 2, 4, 6, 24 and 48 hours).

At the lowest mass of the sorbent (0.1 g) and the shortest sorbent sorbate contact (2 hours), the efficiency was 17.3 %. With the increasing weight and time of sorbent-sorbate contact, the efficiency has increased. Optimal conditions to achieve the highest efficiency of 3,5-dichlorophenol sorption using red mud were: the amount of red mud (2,5 g), sorption time (48 hours) and temperature (25 °C). Efficiency under these conditions was 95.62 %.

In our experimental measurement, the effect of temperature on the efficiency of sorption did not demonstrate a significant effect. With increasing temperature and a sorbent amount, the sorbent efficiency increased in the range of about 0.5 - 3.0 % for both sorbent bins.

However, it should be noted that the requirements for sorbent may vary and therefore the individual requirements of a particular case must be taken into consideration. Nevertheless, the results presented in this study should be confirmed by further testing.

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