Utilization of Calcined Gypsum in Water and Wastewater Treatment: Removal of Phenol

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ABSTRACT

The release of phenol-containing effluents above the phenol permissible limit has triggered a lot of concern over the world due to their toxic nature. The adsorptive potential of gypsum on the removal of phenol was investigated. The effect of gypsum loading (0.5–3 g), contact time (2.5–20 min) and solution temperature (298 to 318 K) on the removal of phenol by gypsum was studied at neutral pH. The thermodynamics of the adsorption process was also studied. The kinetic data were fitted into the pseudo-second-order, Elovich, and intraparticle diffusion models. The removal efficiency of phenol increased along with the mass of gypsum, contact time and temperature. The results of the thermodynamics study indicate that the adsorption process is spontaneous and endothermic in nature. The change in free energy (ΔG^0) was found to increase with temperature. The values of the estimated ΔG^0 suggest that the phenol adsorption on gypsum is a physical adsorption process. Additionally, the kinetic data fitted best into the pseudo-second-order than the other kinetic models. This study proved that phenol can be used effectively for the reduction of phenol concentrations in water and wastewater.

Keywords: calcined gypsum; phenol; removal; thermodynamics parameters.

INTRODUCTION

The excessive release of phenol-containing wastes into the aqueous ecosystem due to industrialization is one of the greatest threats to health (Bemosmane et al., 2018; Pourakbar et al., 2018; Kaczorek et al., 2016). The major sources of phenolic compounds include the wastes from paper mills, pesticides production, petrochemical, paint, textile plants, oil refineries, and pharmaceutical industries (Mahvi et al., 2007; Azevedo et al., 2009,Baird; 1998). Phenols and phenolic compounds are considered as the priority and one of the most dangerous pollutants since they are harmful to organisms even at a low concentration (Rahmani et al. 2008; Pourakbar et al., 2018). Phenol and its degradation products are major aquatic

pollutants since they are toxic and carcinogenic in nature (Ahmadi and Igwegbe, 2018; Kulkarni and Kaware, 2013). Phenol is lethal to people and can result in either acute (short-term) and chronic (long-term) effects (Villegas et al., 2016). It is highly corrosive and causes harmful side effects such as diarrhea, nervous breakdown, impaired vision, sour mouth, and excretion of dark urine (Kulkarni and Kaware, 2013). Besides, it is relatively stable and soluble in water (Azevedo et al., 2009**.** Therefore, considering the health implication, proper treatment techniques are necessary for the reduction of the phenol level. Owing to its high toxicity and difficulty to reduce biologically, the limits for their existence in drinking water and effluents were set up (Sharan et al., 2009; Roostaei and Tezel, 2004; Salari et al., 2018). The

United States Environmental Protection Agency (USEPA) specified that the phenol concentration in drinking water and industrial effluents must be less than 1µg/L and 500 µg/L, respectively (Tao et al., 2013; Yan et al. 2006; Salari et al., 2018; Wu et al; 2001). Additionally, the World Health Organization (WHO) considers the maximum permissible limit of phenol in drinking water to be 0.002 mg/L (Salari et al., 2018; Roostaei et al., 2004). Furthermore, the EPA has set the concentration of phenol in wastewater below 2 mg/L (EPA, 2015; Pourakbar et al., 2018). The presence of phenol in water contributes to taste deterioration and odor (Sharan et al., 2009; Uddin et al., 2008; Moustafa et al. 1989). It has been reported that 700 million tons of phenol are produced per annum (Senthilvelan et al., 2014).

Many methods have been employed for the reduction of phenol including chemical oxidation (Kumar et al., 2010; Yavuz et al., 2007), photo-degradation (Wu et al., 2001; Mahvi et al., 2007; Azevedo et al., 2009), ion exchange (Caetano et al., 2009), electrocoagulation (Zazouli and Taghavi, 2012; Olya and Pirkarami, 2013; Bazrafshan et al., 2012), adsorption (Gholizadeh et al., 2013; Feng et al., 2015; Ahmadi and Igwegbe, 2018; Roostaei and Tezel, 2004), coagulation-flocculation (Bazrafshan et al., 2012), membrane filtration (Bemosmane et al., 2018), advanced oxidation processes (Rubalcaba et al., 2007; Esplugas et al., 2002), and other.

However, amongst the above-mentioned methods, the adsorption process seems to be the most prospective for the removal of organic and mineral pollutants (Sarvani et al., 2018; Deng et al., 2010) and is effective either carried out on laboratory or industrial scale (Sarvani et al., 2018; Rodrigues, 2011). Adsorption has been extensively used to control the phenol pollution because of its cost-effectiveness, simplicity of operation, availability of adsorbent precursors and efficiency (Menkiti et al., 2018). Activated carbon is commonly used for adsorbing pollutants but it is very expensive and cannot be easily regenerated (Khoshnamvand et al., 2017). Ahmadi and Igwegbe (2018) studied the removal of phenol using acid-modified bentonite. The removal efficiency of 97.9% was obtained at contact time of 60 min, dosage of 0.6 g/L, pH of 6 and concentration of 50 mg/L; the experimental data was best described by the pseudo-second-order kinetic model. Menkiti et al. (2018) used chemically modified Nteje clay (CMC) to remove

phenol from petroleum effluent; 91.67% efficiency was reached at equilibrium time of 25 min and pH of 6. The data fitted best to the Langmuir and pseudo-second-order model with monolayer adsorption capacity of 3.487 mg/g. Other researchers utilized low-cost adsorbents for the removal of phenol such as pomegranate peel carbon (Afsharnia et al., 2016), Moringa peregrine tree shell ash (Zarei et al., 2013), bentonite (Senturk et al., 2009; Banat et al., 2000), charcoal (Halhouli et al., 1995; Mukherjee et al., 2007), bagasse fly ash (Srivastava et al., 2006), freshwater micro-algae Chlorella vulgaris (Baldiris – Navarro et al., 2018), tobacco residues (Kilic et al., 2011), Moroccan stevensite (Hamadaoui et al., 2018), sawdust (Dakhil, 2013; Douara et al, 2016), avocado kernel seeds (Rodrigues et al., 2011), water hyacinth (Uddin et al., 2008) and ostrich feathers (Monshouri et al., 2012).

Gypsum is more advantageous compared to other adsorbents because of its abundance in nature, low-cost and no pretreatment is required as an adsorbent (Rauf et al., 2009). Gypsum ($CaSO_4.2H_2O$) is a very soft sulfate mineral composed of calcium sulfate dihydrate (Bello et al., 2013; Cornelis and Cornelius, 1985). Gypsum was applied successfully and extensively for the removal of dye (Rauf et al, 2009), pharmaceutical drugs (Li et al., 2014) and heavy metals (Petruzzeli et al., 2015; Raii et al., 2014). Moreover, there is no report on the use of gypsum for the removal of phenol.

The present study aims at investigating the removal of phenol from aqueous solution using gypsum which is abundantly available. The effects of mass of adsorbent (gypsum), contact time and solution temperature on the removal of phenol were investigated using the batch method under neutral pH conditions. The thermodynamics and kinetics of the adsorption process were also studied.

MATERIALS AND METHODS

The gypsum was supplied by Rawabi for Mining (Tafila, South Jordan). It was crushed, milled and then calcined at 150–200°C. Phenol (99% purity) was obtained from a local supplier. Phenol stocks were prepared by dissolving accurate weight of phenol in 1000 mL of DDW. This stock solution was then used in the preparation of synthetic wastewater samples with different concentrations. The batch sorption experiments

were conducted in a series of 500-mL glass beakers containing 250 mL of the phenol solution. A determined amount of calcined gypsum (0.5–3.0 g) was added to these beakers. The experiments were conducted at an ambient temperature of 25°C and the desired temperature with a stirring time of 5–25 min. After the completion of the experiments, the samples were filtered. The filtrate of phenol solutions was then subjected to UV-visible analysis in order to determine the percentage of phenol removal (SPECTRA-COMP 602; Advanced Products, Milan, Italy). A calibration curve of absorbance against different concentrations of phenol was constructed. The samples of phenol solutions had been analyzed by UV before and after treatment in order to determine the amount of phenol removed. The amount of phenol adsorbed on gypsum, q_e(mmol/g) and the percentage of phenol removed ($\%R$) were calculated as follows (Igwegbe et al., 2016; Ahmadi and Igwegbe, 2018):

$$q_{e = (C_0 - C_t)} \times \frac{V}{M} \quad (1)$$

$$\%R = \frac{C_0 - C_t}{C_0} \times 100 \quad (2)$$

where: C_0 and C_t (mg/L) are the concentrations at the initial time,
$t=0$ and time, t, respectively.
V is the volume of phenol solution (L) and
W is the mass of gypsum (g).

RESULTS AND DISCUSSION

Effect of gypsum mass

The effect of gypsum mass on the reduction of phenol concentration was studied by varying the mass at the initial phenol concentration of 0.3809 mol/L. As seen in Fig. 1 and Table 1, as the mass of gypsum was increased from 0.5 to 3 g, the percentage of phenol removed on the adsorbent (gypsum) was enhanced steeply from 29.06 to 84.25 %. Therefore, the maximum phenol removal was achieved at the highest mass of gypsum studied (3 g). The improvement in phenol removal with increasing mass of gypsum is as a result of the increase in the number of adsorption sites and adsorbent surface area (Sahu et al., 2017; Afsharnia et al., 2017). The amount of phenol per unit mass of the gypsum, q_e decreased with the increasing adsorbent loading from 177.75 to 31.28 mmol/g. This is because the active adsorption sites of the adsorbent were not fully exploited (unsaturated) at a higher adsorbent loading compared to lower adsorbent mass (Gorzin and Abadi, 2018; Radnia et al., 2012). A smaller mass of adsorbent will be saturated more quickly than a larger one (Larous and Meniai, 2012).

Effect of contact time

In order to investigate the effect of time on the adsorption of phenol using gypsum, the time of contact varied from 2.5 to 20 min using an
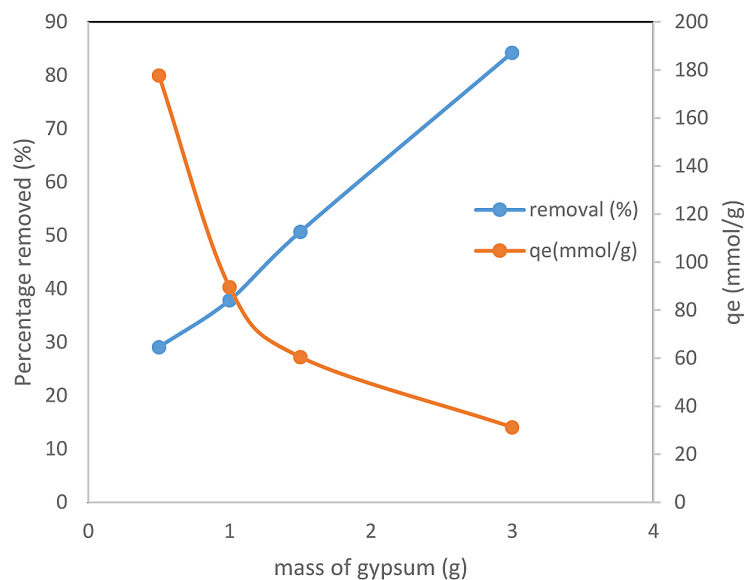

Fig. 1. Effect of mass of gypsum on phenol removal
(Contact time = 5 min, temperature = 25°C, initial phenol concentration = 0.3809 mol/L, pH = 7)

Table 1. Effect of gypsum mass, contact time and temperature on phenol removal at $C_0 = 0.3809$ mol/L

Operating parameter		C_e (mol/L)	Phenol removal (%)	q_e (mmol/g)
Mass of gypsum	0.5g	0.2702	29.06	177.75
	1g	0.2367	37.86	89.65
	1.5g	0.1879	50.67	60.53
	3g	0.06	84.25	31.28
Contact time	2.5 min	0.323	15.20	9.65
	10 min	0.2052	46.13	29.28
	20 min	0.1189	68.78	43.67
Temperature	298 K	0.1676	556	35.55
	308 K	0.1179	69.05	43.83
	318 K	0.1128	70.38	44.68

adsorbent mass of 1.5 g. Figure 2 and Table 1 show that the percentage of phenol removed and the adsorption capacity q_e was increased rapidly with time. Maximum phenol removal was attained at the highest contact time studied. This may result from the fact that the frequency of collision of the adsorbate and the adsorbent was increased with retention time, which also increased the process of adsorption (Ahmadi and Igwegbe, 2018; Buhani et al., 2018). Moreover, the adsorption sites were more available with time.

Effect of solution temperature

Temperature changes the adsorption capacity of an adsorbent for a specific adsorbate (Banerjee and Chattopadhyaya, 2017). The influence of solution temperature on the phenol removal was investigated by varying the temperatures from 298 to 318 K (Fig. 3). As shown in Figure 3 and Table 1, the adsorption of phenol on gypsum was improved with increasing temperature. The highest removal of 70.39% was achieved at the highest studied temperature of 318 K. The removal of phenol was favored at a higher temperature because temperature increases the number of collisions between the adsorbent and the adsorbate, thereby enhancing the rate of adsorption (Afsharnia et al., 2017). Figure 3 also shows that the amount of phenol uptake by gypsum increased from 35.55 to 44.68 mmol/g. The rate of diffusion of the phenol molecules onto the gypsum surface was high with increased temperature.

Thermodynamics studies

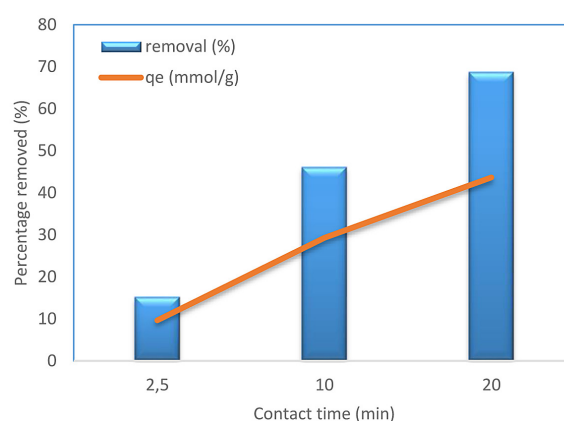The thermodynamic parameters including Gibbs free energy change (ΔG^0), entropy change

Fig. 2. Effect of contact time on phenol removal (Mass of gypsum = 1.5 g, temperature = 25°C, initial phenol concentration = 0.3809 mol/L, pH = 7)

(ΔS^0) and enthalpy change (ΔH^0) were calculated using Eqs. 3 and 4 (Adeogun and Balakrishnan, 2015; AlOthman et al., 2014):

$$\Delta G^0 = -RTLnK_d \qquad (3)$$

$$LnK_d = -\frac{\Delta H^0}{RT} + \frac{\Delta S^0}{R} \qquad (4)$$

where: K_d is a thermodynamic equilibrium constant obtained using Eq. 3 (Sogut and Caliskan, 2017):

$$K_d = \frac{C_0 - C_e}{C_e} \qquad (5)$$

where: ΔG^0 is the free energy change (kJ/mol),
T is the solution temperature (K),
R is the ideal gas constant (8.314 J/K/mol),
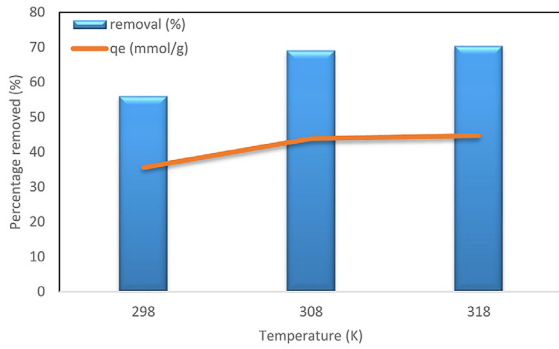C_0 is the initial phenol concentration and C_e is the final phenol concentration.

Fig. 3. Effect of solution temperature on phenol removal

(Mass of gypsum = 1.5 g, contact time = 5 min., initial phenol concentration = 0.3809 mol/L, pH = 7)

The values of ΔH^0 and ΔS^0 were obtained from the slope $(-\frac{\Delta H^0}{R})$ and intercept $(\frac{\Delta S^0}{R})$ of the Vant Hoff's plot of LnK_d versus $1/T$ (Fig. 4). The calculated thermodynamic parameters are presented in Table 2. The negative value of ΔG^0 indicates the spontaneous nature of the adsorption of phenol on gypsum. The value of ΔG^0 was found to increase along with temperature, which implies that the adsorption of phenol on gypsum was favored at increasing temperature. In general, the free
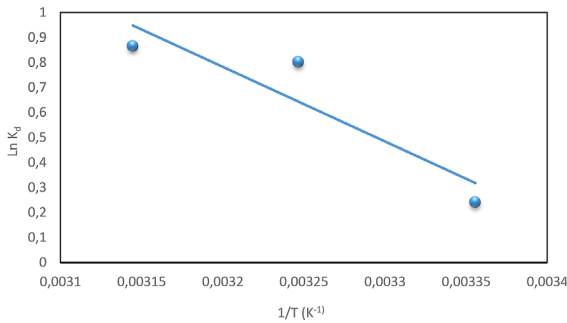

Fig. 4. Vant Hoff's linear plots of Ln K_d versus 1/T

Table 2. Thermodynamics parameters calculated for phenol adsorption on gypsum

Model	Kinetic parameters	
Pseudo-second-order	q_e(mmol/g)	87.72
	K_2 (g/mmol min)	4.357
	R^2	0.9997
Elovich	β (g/mmol)	0.0623
	α(mmol/g min)	11.253
	R^2	0.9898
Intraparticle diffusion	K_{pi} (mmol/g min$^{0.5}$)	11.789
	c_i	8.6795
	R^2	0.9988

energy change for physical adsorption is between -20 and 0 kJ/mol, but for chemical adsorption it is in the range of -80 to -400 kJ/mol (Babakhouya et al., 2010; Atkins, 1990). This suggests that the adsorption of phenol on gypsum is a physical adsorption process. The positive value of ΔH^0 indicates the phenol adsorption process is endothermic in nature (Adeogun and Balakrishnan, 2015; Babakhouya et al., 2010). The positive ΔS^0 value obtained denotes the increase in the degree of disorderliness of the adsorbed species (AlOthman et al., 2014) and the affinity of the gypsum adsorbent for phenol.

Adsorption kinetics

Adsorption kinetic models are used to study the adsorption rate and the probable rate governing step. The kinetics of the adsorption of phenol on gypsum was studied using the pseudo-second-order, Elovich, and intraparticle diffusion models.

The pseudo-second-order is stated as follows (Ho, 2006):

$$\frac{t}{q_t} = \frac{1}{K_2} + \frac{1}{q_e}t \qquad (6)$$

where: q_e and q_t is the amount of phenol adsorbed per unit mass of the adsorbent at equilibrium and at time, t (mmol/g).
K_2 is the pseudo-second-order rate constants (min^{-1}).

The pseudo-second-order plot is shown in Fig. 5. The constants, q_e, and K_2 (Table 3) were evaluated from the slope and intercept of the regression plot of t/q_t versus t (Fig. 5), respectively. The value of the regression coefficient, R^2 (0.9997) implies that the adsorption kinetic data obeys the pseudo-second-order model more so than other models. A study on the removal of phenol using adsorption onto modified *Pistacia mutica* shells (Sarvani et al., 2018) also followed the model.

The Elovich kinetic model is expressed as (Riahi et al., 2017; Abdelkreem, 2013):

$$q_t = (1/\beta)Ln(\alpha\beta) + (1/\beta)Ln\,t \qquad (7)$$

where: α is the initial adsorption rate (mmol/g min) and
β is related to the extent of surface coverage and the activation energy for chemisorption (g/mmol).

Table 3. Comparison of gypsum with other materials for phenol reduction

Adsorbent	Maximum removal	Conditions	Reference
Pomegranate peel carbon	95.96% (79.97 mg/g)	phenol concentration = 50 mg/l adsorbent dosage = 0.4 g/l Time = 120 min pH = 7 temperature = 30±1 °C stirring rate = 150 rpm Speed = 150 rpm	Afsharnia et al., 2016
Dates'stones	99%	Phenol concentration = 30 mg/L pH = 3 temperature = 50°C mass of activated carbon = 0.2g	Un et al., 2017
Rhazya stricta carbon	85%	solution pH = 3 adsorbent dose = 0.5 g/l temperature of 45 °C V = 50 mL Contact time = 4 h Phenol concentration=100 mg/L Speed = 100 rpm	Hegazy et al., 2014
Na-montmorillonite	100% (15 mg/g)	pH =5 Volume, V =100 mL Phenol concentration = 5 mg/L Contact time = 180 min Temperature = 23°C Speed = 180 rpm	Djebbar et al., 2012
Pistacia mutica shells	99% (15.2 mg/g)	Phenol concentration = 50 mg/L) Adsorbent dosage = 1 g/L pH = 4 Contact time = 45 min V = 100 mL Speed = 150 rpm	Sarvani et al., 2018
Bentonite	97.9% (8.2 mg/g)	Contact time = 60 min Initial phenol concentration = 50 mg/L Dosage of bentonite = 1 g/L pH = 4 Temperature = 25°C V = 100 mL Speed = 150 rpm	Ahmadi and Igwegbe, 2018
Gypsum	84.25% (31.28 mmol/g)	Contact time = 5 min, Mass of gypsum = 3 g, temperature = 25 °C Phenol concentration = 0.3809 mol/L. pH = 7 V = 250 mL	This study
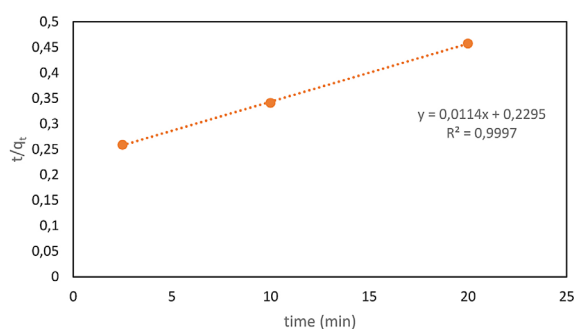
Fig. 5. Pseudo-second-order kinetic model plot of phenol adsorption onto gypsum

The plot of q_t versus *Ln t* is shown in Figure 6. The Elovich constants, β, and α were obtained from the slope and intercept, respectively. The data fitted well into the model. The studies by Abdelkreem (2013) and Ekpete et al. (2011) followed the Elovich model.

The intraparticle diffusion equation is given as (Igwegbe et al., 2016; Reddy et al., 2012):

$$q_t = k_{pi}\, t^{0.5} + c_i \qquad (8)$$

where: c_i is a constant that provides an idea about the thickness of the boundary layer,
k_{pi} is the intraparticle diffusion rate constant (mg/g min$^{0.5}$) and
q_t is the amount adsorbed (mmol/g) at time t (min).

The graph of q_t versus $t^{0.5}$ is represented in Figure 7. The intraparticle constants, k_{pi} and c_i were calculated from the slope and intercept, respectively. The data conformed to the intraparticle diffusion model. The plot did not pass through the origin implying that the intra-particle diffusion is not the only rate limiting step (Ma et al., 2013).
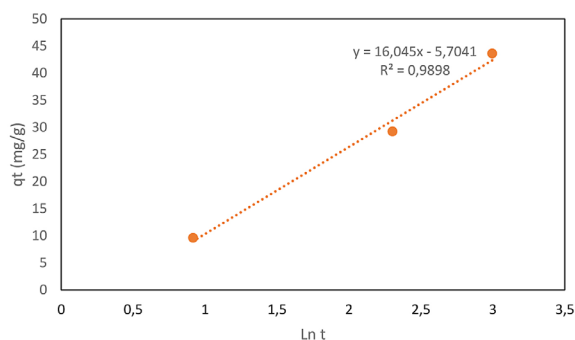
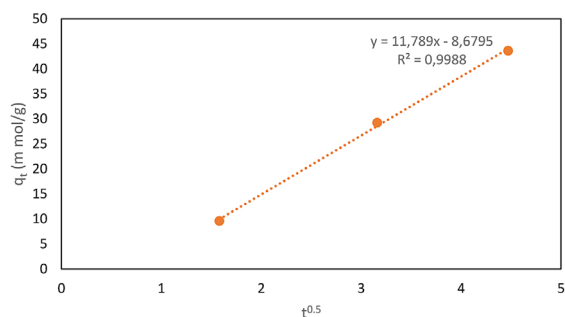**Fig. 6.** Elovich kinetic model plot of phenol adsorption onto gypsum

Fig. 7. Intraparticle diffusion plot of phenol adsorption onto gypsum

Generally, the phenol adsorption kinetic data fitted best into the pseudo-second-order kinetic model denoting a chemical adsorption process (Igwegbe et al., 2016).

Comparison with other adsorbents for phenol removal

The removal efficiencies and the adsorption capacities of phenol removal using different adsorbent materials are presented in Table 3. It indicates that gypsum is a potential material that can be applied for the reduction of phenol in aqueous environments, when compared with other materials.

CONCLUSION

The effectiveness of the adsorptive removal of phenol from its aqueous solution using gypsum (which is readily available) was investigated. The effect of gypsum mass, contact time and solution temperature on the adsorption process was studied. Phenol removal was found to increase along with adsorbent mass, contact time and temperature. The phenol adsorption process on gypsum

was found to be spontaneous and endothermic in nature. The adsorption data fit into the pseudo-second-order, Elovich, and intraparticle diffusion kinetic models. The results obtained from the study showed that gypsum can act as a potential adsorbent for the removal of phenol from its aqueous solutions.

Acknowledgement

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