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Adsorption and Regeneration of Sungai Pasak Pumice as An Adsorbent for Ammonium Removal in Water

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ABSTRACT

Pumice is known as a natural material with a porous structure, has the potential as an adsorbent to remove various kinds of pollutant, such as metals and other chemical compounds. The abundance of pumice is found in Sungai Pasak area and it never been utilized. This study was performed to see the ability of Sungai Pasak pumice as an adsorbent for ammonium removal in water along with its ability to be regenerated. The study was carried out triplo in batches to obtain optimum adsorption conditions which were then used in desorption experiments using HCl 0.1 M agent for regeneration process. The optimum conditions obtained: adsorbate pH 6, adsorbent dose 0.3 g/l, contact time 30 minutes, adsorbent diameter <63 μm and adsorbate concentration 4 mg/l. The results showed that pumice has ability to remove ammonium with adsorption capacity at optimum conditions were 47.06% and 6.27 mg/g with Freundlich's isotherm equation (R^2=0.997). Acid agents are able to adsorb ammonium from pumice with an average desorption percentage 88.89% after 2 times of reuse, proving that acidic agents are able to regenerate pumice adsorbents so they can be reused. The potential for adsorption and regeneration of the Sungai Pasak pumice can be utilized to treat water with ammonium such as ground water and waste water.

1. INTRODUCTION

Application of adsorbent from nature rocks such as zeolite, perlite and pumice have become a special focus nowadays. Pumice has light colour and consists of bubbles forming like a foam, literally known as silicate vulcanic rocks because of its phorous structure. Pumice constructed by fine capilars where adsorbate will adsorp into it (Edahwati and Suprihatin, 2011).

Ammonium generally found as one of pollutant which occur by industrial, domestic and agricultural activities such as fertilization. 33.7 mg of ammonium ion per kg body weight per day possibly affect metabolism, glucose tolerance and reduce cell's sensitivity over insulin (Fawel, et al., 1996). The quality standards for ammonium in drinking water is 1.5 mg/l based on Ministry of Health of Republik Indonesia Regulation No. 492/2010 and 0.25 – 25 mg/l in Ministry of Environment of Republik Indonesia Regulation No. 5/2014 about Waste Water Quality Standards. Previous research about pumice as an adsorbent had proven that it can remove various of pollutant, such as organic materials (Edwardo, et al., 2012) and removal of oil and grease (Miskah, 2010) with removal efficiency 98%; 89.78% and 69% respectively.

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Table1. Variation of Parameters

Parameter	Unit	References	Variation Used and Result	Variation this Experiment
Adsorbate pH	-	Halim, et al. (2013)	2-12; 7-8	6,7,8,9,10
		Xia, et al. (2016)	2-10; 7	
		Huang, et al. (2015)	4-9; 8	
Adsorbent dose	g/l	Xia, et al. (2016)	0.1-0.5; 0.3	0,3, 1, 3, 10, 30
		Zhang, et al. (2010)	0.5-6; 2	
		Sari (2016)	0.3-30; 0.3	
Adsorbent diameter	µm	Based on sieve available in the laboratory		<63, 63-125, 125-250, 250-500, 500-600
Contact time	mins	Moradi, et al. (2016)	60-180; 60	10,30,60,90,120
		Alshameri, et al. (2014)	30-180; 30	
		Huang, et al. (2015)	30-180; 120	
Adsorbate concentration	mg/l	Average of ammonium concentration in ground water of Padang City was 1.655 mg/l		0,5;1;2;3;4

Based on observations it is known that the use of pumice by the people in the West Sumatra region has not been maximized compared to its large presence especially pumice which is widely found in Sungai Pasak, Pariaman Regency as the rest of the sand mining activities by the surrounding community, it only stacked along the banks of the river approximately 1 – 5 kilograms per day of mining. Research on the utilization and regeneration of the Sungai Pasak pumice stone in pollutant removal from water has been done previously, such as iron (Indah et al., 2017), manganese (Indah, et al., 2017), chromium (Indah, et al., 2017) and nitrate (Helard, et al., 2018) with removal efficiency up to 78% and regenerated up to twice reuse with the removal efficiency ranging from 56 - 91%. This value is still within the same range as the efficiency before reuse.

Based on the research, this study completed the information about adsorption and regeneration of the Sungai Pasak pumice as an adsorbent for ammonium removal in ground water.

2. METHODS

This research was performed in Water Laboratory of Andalas University, Padang in 2016 as the project of Sungai Pasak pumice utilization for water pollutant removal.

2.1. Equipment and Material Preparation

Shaker (Haake SWB 20), analytical balance (ANDHR-60), pH meter (Eutech, pH 510), hot plate (Ciramec, SP131325), magnetic stirrer and UV-Vis Spectrophotometer (Shimadzu, UV-2600).

Pumice adsorbent is prepared by wash it with distilled water and dried out at room temperature, then grind into powder. Sieve shaker used to obtain pumice in various diameter. Energy dispersive x-ray (EDX) spectroscopy was applied to obtain information of oxide material of the pumice. Scanning electron microscopy (SEM) observation also performed to analyze the morphology of pumice stones.

2.2. Adsorption experiment

Adsorbent and the artificial solution with adjusted pH were put into erlenmeyer then stirred using a shaker at a speed of 100 rpm (Kitis, et al., 2007) at the specified contact time. After that, solution wasfiltered to using filter paper. The filtered solution was taken 25 ml to measure its ammonium concentration by adding 1-2 drops of seignette salt and 0.5 ml of Nessler reagent. Then the solution was homogenized and left for 10 minutes, then ammonium concentration was measured using a UV-Vis spectrophotometer. The adsorption capacity Q (mg/g) was calculated by following equation (Mekonnen, et al., 2015):

$$Q = \frac{C_{in} - C_{out}}{m} \, x \, V$$

C_{in} is the initial concentration of ammonium (mg/L), C_{out} is the equilibrium concentration of ammonium (mg/L), m is the mass of the pumice (g) and V is the volume of the solution (L).

Experiments were performed continuously starting from the variation of adsorbate pH. The optimum pH obtained then applied in the next variation experiment, the adsorbent dose. The optimum adsorbent dose then applied in the variation of adsorbent diameter experiment, and so on.

2.3. Regeneration experiment

Regeneration experiment applied with HCl 0.1 M as desorption agent. The desorption process was applied on pumice powder that had been used in the adsorption process with optimum conditions. The desorption adsorbent is reused for the adsorption process up to 2 times of reuse, which consist of adsorption I (initial), desorption I, adsorption II (reuse I), desorption II and adsorption III (reuse II). Ratio of adsorbents with desorption agents was 1:200. The result was represented as % desorption which calculated by following equation (Yalcinkaya, et al., 2002):

$$\%Desorption = \frac{Concentration\ of\ desorpted\ ammonium}{Concentration\ of\ adsorpted\ ammonium} \, x \, 100\%$$

3. RESULT AND DISCUSSION

3.1. Characteristic of Adsorbent

The main constituent elements of pumice are oxygen (O) and silica (Si). Both of these elements are found in the main constituent minerals, SiO_2. In addition, this pumice also contains quite a lot of aluminum (Al) which forms Al_2O_3 compounds. Besides aluminum, other metals such as magnesium (Mg) and iron (Fe) are also found in pumice. Alkaline elements such as sodium (Na), potassium (K) and calcium (Ca) are also contained in this pumice even though in a small percentage.

Pumice has a multi cavity structure so it has a very small density (usually <1 g/cm^3). The surface texture of pumice is irregular due to pore sizes that vary greatly from a few microns to <200 microns (Calvelri and Miraglia, 2003). Figure 1 shows that the pumice structure of the Sungai Pasak is very porous with varying cavity sizes. The number of pores found in pumice causes a larger surface area. This shows that pumice has a good structure as an adsorbent.

Table 2. Chemical Composition of Pumice Stone from Sungai Pasak, West Sumatera, Indonesia

Element	% Weigh	% Atom
O	56.38	70.53
Na	0.49	0.43
Mg	0.06	0.05
Al	3.89	2.89
Si	32.56	23.20
K	2.41	1.23
Ca	1.20	0.60
Fe	3.00	1.07

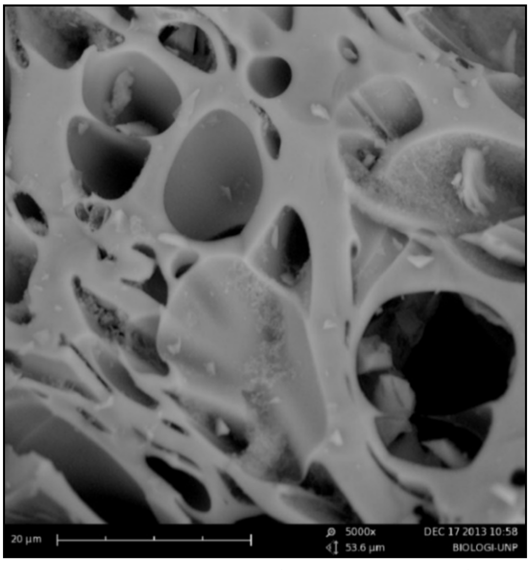

Figure 1. SEM micrograph of Pumice Stone from Sungai Pasak, West Sumatera, Indonesia

3.2. Adsorption results

3.2.1. Optimum pH

The largest adsorption capacity for the range was 6.02 mg/g obtained at pH 6. At pH >6 there is a decrease in ammonium removal efficiency because the ammonium equilibrium in solution is strongly influenced by the pH of the solution (Hedstrom, 2001). Ammonia can be eliminated if it is in the form of an ammonium ion. At alkaline pH, the number of OH^- ions causes a bond between ammonium ion (NH_4^+) and OH^- so that NH_4^+ changes to ammonia (NH_3). Thornton, et al. (2007) proved that the mesolite adsorbent was able to remove ammonium in water at 6-7 optimum pH with removal efficiency up to 70%. Huang et al. (2015) used zeolite adsorbents to remove ammonium in wastewater, obtained optimum pH 6-7 with 70% removal efficiency because at pH >7 ammonium ions would easily become ammonia (in non-ionic forms) which cannot be set aside by ion exchange.

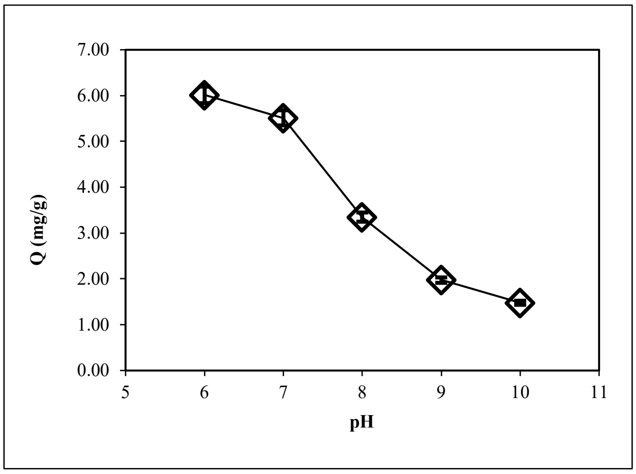

Figure 2. Ammonium Adsorption Capacity in Various pH

3.2.2. Optimum dose of absorbent

The largest adsorption capacity was 6.25 mg/g, obtained using the 0.3 g/l adsorbent dose. The adsorption capacity obtained is inversely proportional to adsorption efficiency. This is probably because there is clogging of the adsorbent at larger dose variation, so that the active side of the adsorbent is not entirely open in adsorbing ammonium and influences the effectiveness of adsorption.

According to Ahalya (2005) in the specific process the increase in mass of the adsorbent is proportional to the increase in the number of particles and the surface area of the adsorbent which causes the number of active sides of ammonium binder to increase, but the adsorption capacity is smaller. Meanwhile, the adsorption capacity of ammonium ions decreases with an increase in the adsorbent dose because the active side acting as an adsorbate binder on the surface is unsaturated so the amount of adsorbate adsorbed per unit weight of the adsorbent decreases when the adsorbent dose is high.

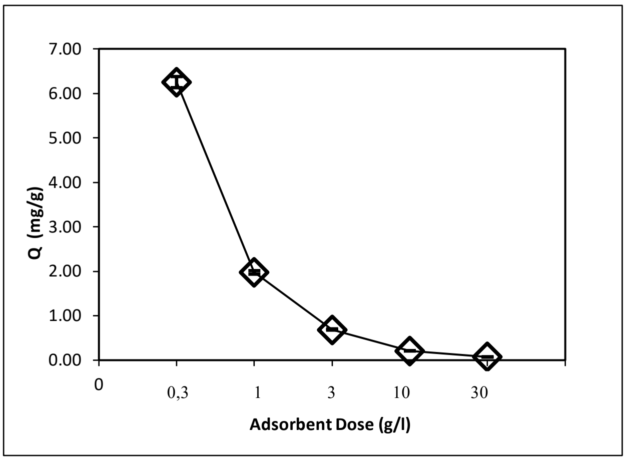

Figure 3. Ammonium Adsorption Capacity in Various Adsorbent Dose

3.2.3. Optimum contact time

The largest adsorption capacity occurs at 30 minutes contact time. According to Alshameri et al. (2014) in the removal of ammonium ions using zeolite adsorbents using a contact time range of 0-180 minutes that the efficiency of ammonium removal (NH_4^+) becomes slower with increasing contact time between adsorbents and adsorbates. This is due to the rapid diffusion of available adsorption sites for the achievement of fast balance. Efficiency decreases because when the adsorption of the outer surface of the adsorbent reaches its saturation point after equilibrium is achieved. Therefore, in this optimization experiment the optimum contact time for ammonium removal is 30 minutes, because it provides the greatest efficiency and adsorption capacity.

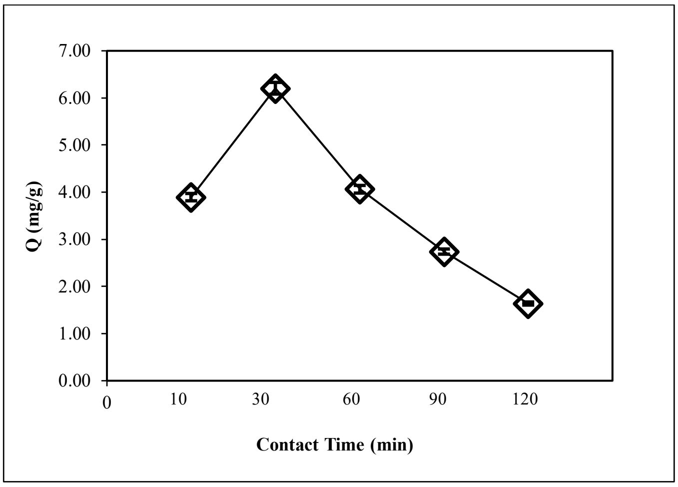

Figure 4. Ammonium Adsorption Capacity in Various Contact Time

3.2.4. Optimum Diameter of adsorbent

The largest adsorption capacity was 6.11 mg/g with an adsorbent diameter of <63 µm. In the optimization experiments with variations in the diameter of the adsorbent, the adsorption capacity obtained was directly proportional to the removal efficiency. This is in accordance with the results of the Lopez-nuñez, et al. (2014) and Zhang, et al. (2015) trial if the smaller the diameter of the adsorbent, the wider surface area obtained.

3.2.5. Optimum initial adsorbate concentration

At higher initial concentrations, the adsorption rate is much higher than the lower concentration. This is because higher concentrations provide greater competitiveness of ammonium ions to replace cation adsorbents during contact time. According to Shi, et al. (2013) the efficiency of ammonium removal by adsorption depends on the ratio between the concentration of ammonium ion in water and the active site of the adsorbent. If the concentration of ammonium ion is high while the active site of the adsorbent is small, the active site of the adsorbent will be saturated, resulting in a decrease in efficiency of removal. The optimum conditions for ammonium removal were chosen based on the highest removal efficiency and the greatest adsorption capacity was at the adsorbate concentration of 4 mg/l.

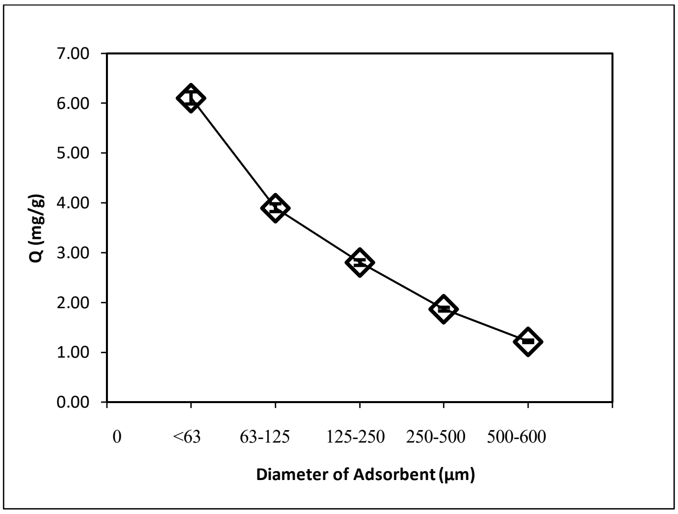

Figure 5. Ammonium Adsorption Capacity in Various Diameter of Adsorbent

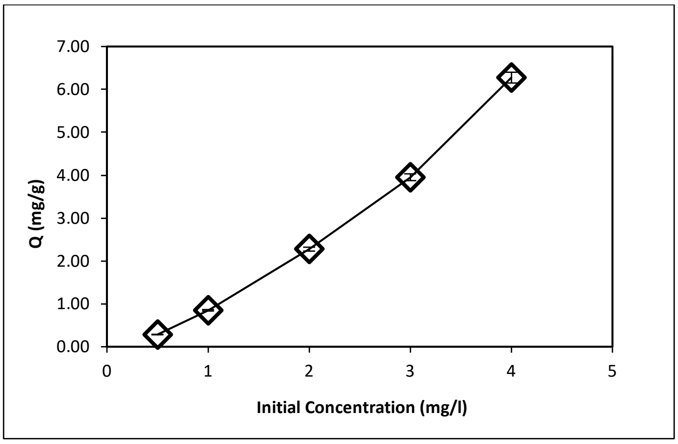

Figure 6. Ammonium Adsorption Capacity in Various Initial Adsorbate Concentration

3.2.6. Isotherm of adsorption process

The curve of the Langmuir and Freundlich isotherm equation was made using optimization experiments at variations in the concentration of the adsorbate. Based on these comparisons, the Freundlich isotherm is more suitable to follow the equilibrium model for the adsorption of ammonium with the Sungai Pasak pumice adsorbent. This means that the active side on the surface of pumice is heterogeneous and adsorption can occur in multilayer, affects the adsorption ability of each of these compounds also varies (Samarghandi, et al., 2012). In addition, the selection of Freundlich isotherm in this study shows that the adsorption mechanism that occurs is physical, where the physical adsorption of molecules is bound to the adsorbent by Van Der Walls force.

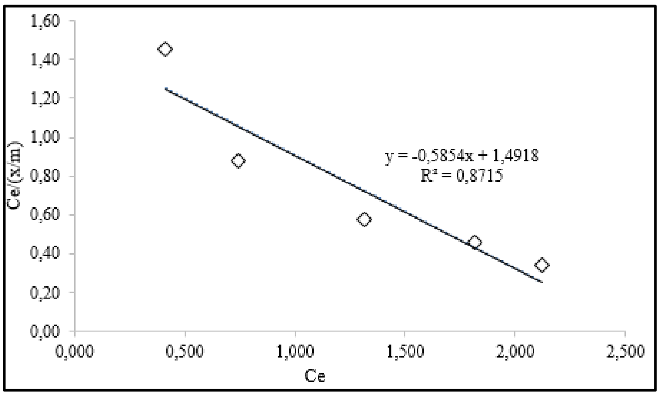

Figure 7. Langmuir Isotherm for Ammonium Adsorption with Pumice Stone Adsorbents

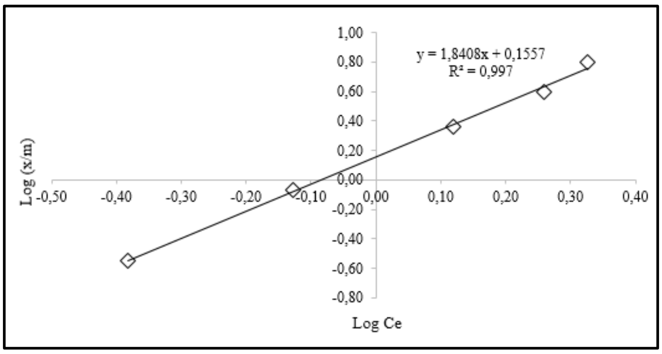

Figure 8. Freundlich Isotherm for Ammonium Adsorption with Pumice Stone Adsorbents

Table 3. Comparison of Isotherm Langmuir and Freundlich Line Constants

Isotherm Model	**Parameter**		
	q_m	K_L	R^2
Langmuir	-1,823	-0,3677	0,871
	K_F	**1/n**	R^2
Freundlich	1,431	1,8408	0,997

According to Atkins (1999), the n value in Freundlich ishoterm if it is in the range of 1-10 adsorption process is said to be profitable, while for 1/n <1 indicates that favorable adsorbent because the interaction between adsorbates and adsorbents is strong, which caused adsorbate is hardly detached from the surface of the adsorbent. The n value in this study is 0.5434 with a value of 1/n >1, is 1.8408 so that pumice as an adsorbent in the ammonium adsorption process is unfavorable. This is due to the weak interaction between the adsorbate (ammonium) and the adsorbent using pumice so that the adsorbate is easily separated from the surface of the adsorbent and causes the adsorption process to be less optimum.

3.3. Regeneration results

The increase in adsorption capacity in the adsorption process II was related to the desorption process I had carried out on the adsorbent. In this case, it is possible that the desorption process with HCl agent is successful so that the ammonium ion which bound to the adsorbent pores is released and the pore is empty or refreshed. This affects the adsorption process which results in a higher adsorption capacity than before. In the desorption process, it is possible for the adsorbent to be activated by HCl agent which affects the pumice pores so that it can adsorb more adsorbates than before. In addition, the HCl agent in the desorption process can affect the pH of the solution in the subsequent adsorption process which possible to have an impact on removal efficiency and adsorption capacity obtained.Adsorbents which have been twice used in the desorption I and II were reused in adsorption III, obtained the decrease in the removal efficiency and adsorption capacity in the adsorption III (reuse II).

According to Wankasi, et al. (2005), protons (H^+) in acidic solutions replace positive ions on the surface of the adsorbent, in this case ammonium ions (NH_4^+). In proton exchange agents such as HCl, the H^+ ions released by HCl have a firm bond to the adsorbent surface. A decrease in % desorption in desorption II occurs because the ability of the desorption agent is not proportional to the amount of adsorbate found on the adsorbent. The adsorbate found on the adsorbent during the desorption II was an accumulation of adsorbate the remaining desorption I and II process. Both of these processes allow for considerable amount of adsorbate on the surface of the adsorbent which is not completely desorbed.

Table 4. Performance of Desorption Agent in Ammonium Removal after Twice Reuse

Desorption Agent	Removal Efficiency (%) of Adsorption			Adsorption Capacity (Q (mg/g))			%Desorption (%)	
	I	II	III	I	II	III	I	II
HCl 0,1 M	43,713	47,515	45,133	5,828	6,335	6,018	89,734	88,048

Ammonium% desorption obtained is quite high related to the adsorption mechanism which has an isotherm equation which is in accordance with the Freundlich model based on adsorption research. This shows that the adsorption process that occurs between the adsorbent of pumice powder and ammonium ion is a reversible physical adsorption process. Based on the research, obtained a value of 1/n >1 which indicates that there is a weak bond between the adsorbent and the adsorbate so that the adsorbate in the form of ammonium ion is easily released from the adsorbent. Wankasi, et al. (2005) stated that the length of contact time between pollutants and recovery reagents influences the rate of pollutant recovery by the adsorbent so that the possibility of obtaining 100% desorption can occur if the contact time between the adsorbent and the desorption agent can be extended.

4. CONCLUSION

Pumice of Sungai Pasak as a natural adsorbent because of its convenience and free to get has porous structure and this study had proven its ability in ammonium removal in water. The optimum conditions using artificial solutions in batch experiment at room temperature and 100 rpm of mixing speed were pH 6 of adsorbate, adsorbent dose 0.3 g/l, contact time 30 minutes, adsorbent diameter <63 µm and adsorbate concentration 4 mg/l with the efficiency of ammonium removal at optimum conditions was 47.06% and ammonium adsorption capacity was 6.27 mg/g. The corresponding isotherm equation is the Freundlich isotherm equation with Kf value 1.431 and the value of 1/n 1.8408 imply that the use of pumice adsorbent is unfavorable because the value of 1/n >1 means that the bond between the adsorbate and the adsorbent is infirm. Regeneration study was applied to maximize the utilization of pumice by reuse. Regeneration by desorption process with HCl agent obtained removal efficiency and adsorption capacity up to 47.515% and 6.335 mg/g. Further study is needed to optimize the ability of pumice as an adsorbent by pretreatment and advanced process modification.

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