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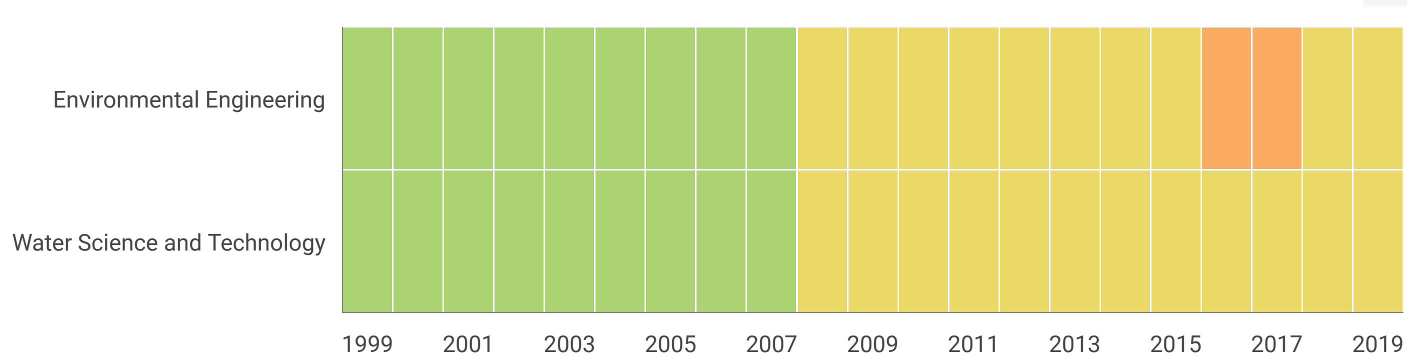

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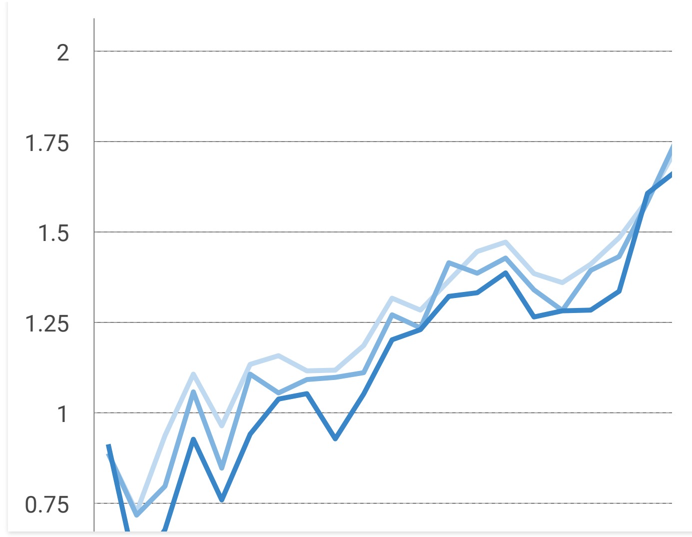

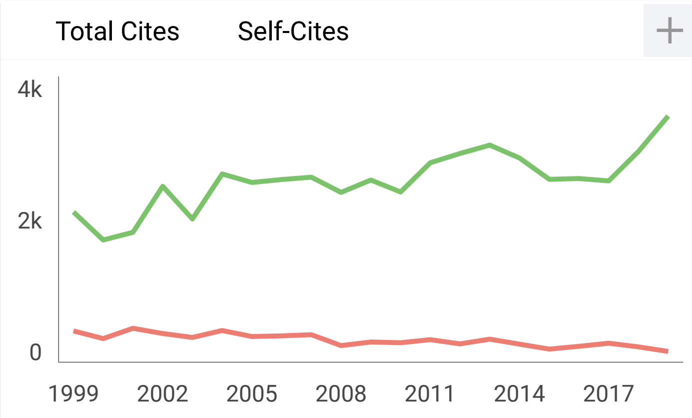

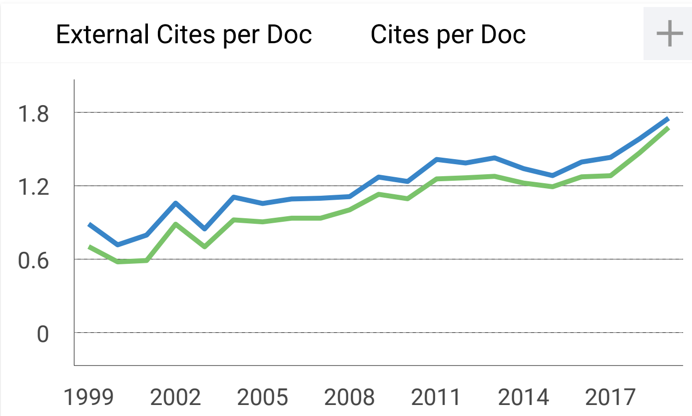

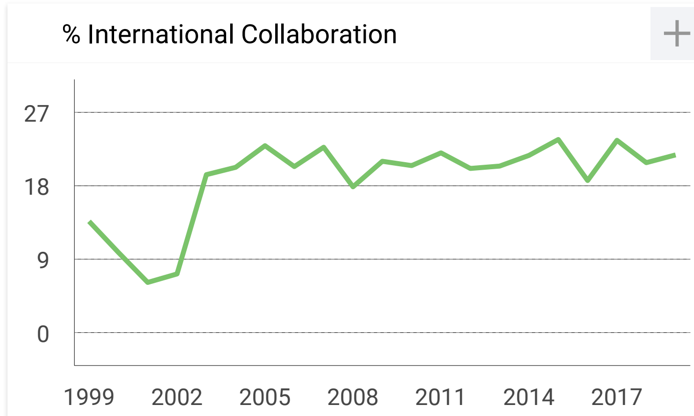

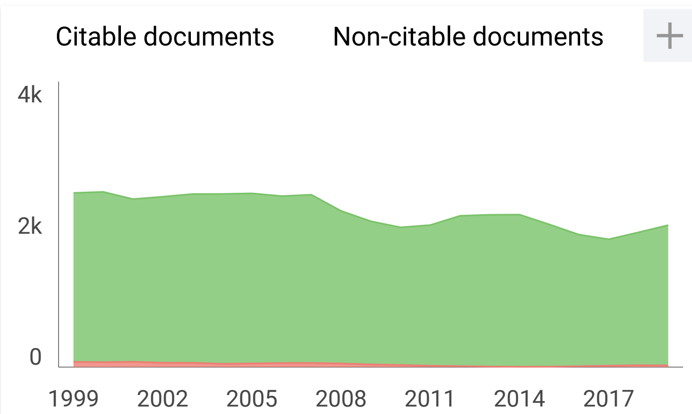

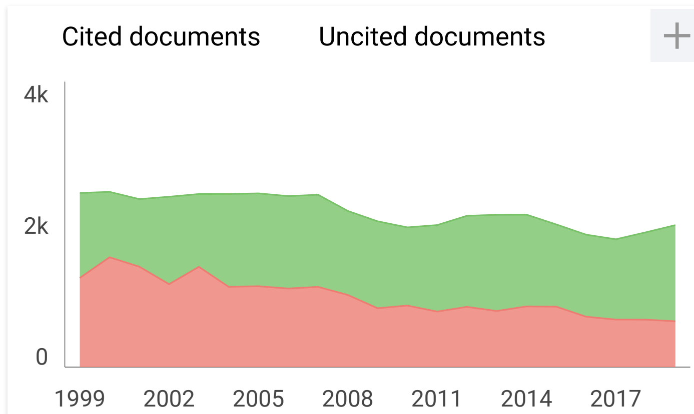

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Studies on desorption and regeneration of natural pumice for iron removal from aqueous solution

S. Indah, D. Helard and A. Binuwara

ABSTRACT

To make the adsorption process more economic and environmental friendly, it is necessary to study desorption and reutilization of the adsorbents. In the present study, the effectiveness of natural pumice in removal of iron from aqueous solution was investigated in several sorption-desorption cycles. The desorption characteristics of previously adsorbed iron ions on natural pumice were tested by various desorbing agents such as HCl, NaOH and aquadest. Among them, HCl showed the highest desorption efficiency (37.89%) with 0.1 M of concentration and 60 min of contact time. The removal efficiency of iron ions in reused natural pumice could be maintained up to 90% in the third cycle of adsorption. The results indicate that although complete desorption was not achieved, natural pumice from Sungai Pasak, West Sumatra, Indonesia, can be sufficiently reused up to three cycles of adsorption-desorption.

Key words | desorption, iron, pumice, regeneration

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INTRODUCTION

Adsorption is known as one of the major unit operations used for removal of various pollutants from water and wastewater. It can be selectively used for removal of certain pollutants by selection of a suitable adsorbent. Low cost adsorbents prepared from rice husk, coconut husk, pine bark waste, tea leaves, mushroom compost, crude olive stones, volcanics, zeolite, perlite, pumice and kaolinite have been used/found effective in removal of heavy metals (Acemioglu 2004; Ahluwalia & Goyal 2005; Ruggieri *et al.* 2008; Steinhauser & Bichler 2008; Abdulrasaq & Basiru 2010; Nieto *et al.* 2010; Malakootian *et al.* 2011; Kütahyalı *et al.* 2012; Kamarudzaman *et al.* 2013; Sepehr *et al.* 2014; Zhang *et al.* 2014; Keçeli 2015). In our earlier research, investigation was carried out for removal of iron and manganese by using adsorbents prepared from maize husk and natural pumice. The results showed that those adsorbents have potential for metals removal from water (Indah *et al.* 2016; Indah *et al.* 2017). However, the used adsorbent has to be discarded after it becomes exhausted. The disposal of adsorbent may generate a problem in the environment and the utilization of minerals like natural pumice as adsorbent may reduce the availability of natural resources. Therefore, the regeneration and reuse of the adsorbents is important to make the operation environmentally friendly and minimize the requirement for adsorbent. In this sense, desorption and reutilization of the adsorbents in adsorption-desorption cycles could help in reducing the residues.

The desorption can be carried out by means of various mechanisms such as ion exchange and complexation, where metals are desorbed from the adsorbent by a proper eluent to produce a small, concentrated volume of metal-containing solution, from which metals could potentially be recovered (Njikam & Schiewer 2012). Studies have shown that acidic, basic and neutral solutions can be used as desorbing agents (Aldor *et al.* 1995; Bux *et al.* 1996; Wankasi *et al.* 2005). Desorption can be also carried out by chelating agents (EDTA) or exchange with other ions (i.e. $CaCl_2$) (Beolchini *et al.* 2003; Sekhar *et al.* 2004). The choice of desorbing agent depends on the kind of adsorbent used and the metals adsorbed. An effective desorbing agent is one that desorbs the metal entirely without degradation of the properties of the adsorbent. After elution, concentrated metals can be recovered by electrochemical or other conventional techniques (Sekhar *et al.* 2004). The regeneration process can be necessary to maximize the function and capability of the adsorbent used, and the reuse of the adsorbent is required in order to make the process economic and environmentally friendly.

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In the present study, the characteristics for desorption and regeneration of iron ions adsorbed onto natural pumice was investigated with various desorbing agents since only few investigations have been reported on the regeneration of natural pumice. This paper reports a study on the ability of acidic, basic and neutral solutions to recover iron already adsorbed by natural pumice and the possibility of the adsorbent to be regenerated or not after exhaustion. In order to investigate the regeneration ability of the natural pumice, sequential adsorption-desorption cycles were repeated three times using the same adsorbent.

MATERIALS AND METHODS

Preparation of adsorbent

Pumice stone was collected at the riverside of Sungai Pasak, West Sumatra, Indonesia, as a byproduct of the process of sand mining in that area. It was washed with distilled water several times and dried out at room temperature. The stone was crushed and sieved in order to produce the desired particle size fractions. A scanning electron microscope (SEM, model S-3400N, Hitachi, Japan) was used to observe the surface morphology of that pumice.

Preparation of iron solution

Iron solutions with known concentrations were prepared by dissolving $Fe(NH_4)_2(SO_4)_2.6H_2O$ in deionized water.

Adsorption experiments

Batch adsorption experiments were carried out at room temperature (20–25 °C) by optimum dose of adsorbent (0.3 mg/L), contact time (60 min), diameter of adsorbent (<149 μm), pH of solution (5) and initial concentration (15 mg/L) as obtained in the previous study. In each experiment, 100 ml of iron solution was contacted with pumice in an Erlenmeyer flask and gently agitated at 100 rpm. After 60 min of contact time, the mixture was filtered by using Whatman's filter paper no. 42, and the concentration of iron in the filtrate was determined by atomic absorption spectrometry (Rayleigh WFX 320, China). The amount of iron ions adsorbed by the pumice was obtained as the difference between the initial and final concentration of the solutions. All experiments were repeated three times, and results presented are consequently the averaged values of replicate tests.

The percentage removal of iron were calculated by using the following equations:

$$\%R = \frac{100(C_0 - C_e)}{C_0} \quad (1)$$

The adsorption amount of iron per gram (g) of adsorbent (mg/g) is

$$q_e = \frac{C_0 - C_e}{W} \times V \quad (2)$$

where C_0 is the initial concentration of iron (mg/L), C_e is the equilibrium concentration of iron (mg/L), V is the volume of the solution (L), and W is the mass of the pumice (g).

Desorption and regeneration experiments

The reuse of the natural pumice was considered in consecutive sorption-desorption cycles. For the desorption experiment, previously adsorbed iron ions on natural pumice were transferred to a flask containing 100 mL of desorbing agent such as HCl, NaOH and distilled water. The mixture was shaken at 100 rpm using a shaker (Innova 2300, New Brunswick, USA) at room temperature for 1 hour. The desorption efficiency of iron ions from the natural pumice was calculated as the ratio between the amount of iron ions desorbed and the amount of iron ions adsorbed (Equation (3)). In the regeneration experiment, the iron-loaded adsorbent that was eluted by the desorbing agents was thoroughly washed three times with deionized water to remove any traces of desorbing agent, and then mixed again in wastewater containing iron ions for the next adsorption cycle. This procedure was employed for three consecutive cycles.

$$\textit{Percent of desorption} = \frac{\text{Concentration of metal desorbed}}{\text{Concentration of metal adsorbed}} \times 100\% \quad (3)$$

RESULTS AND DISCUSSION

Pore structure of pumice from Sungai Pasak, West Sumatra, Indonesia

Si, Al and Fe are the major elements in natural pumice from Sungai Pasak, as shown in Table 1 as determined by energy

Table 1 | Elemental composition of natural pumice from Sungai Pasak, West Sumatra, Indonesia

Element	% weight
O	56.38
Na	0.49
Mg	0.06
Al	3.89
Si	32.56
K	2.41
Ca	1.2
Fe	3

dispersive X-ray analysis (EDX). Other elements, except K, Ca, Na and Mg, were present in relatively smaller amounts (less than 3%). The EDX measurement indicated that the oxides of Si, Al and Fe were the major constituents of the pumice. Similar values were reported by Alemayehu & Lennartz (2009) and Asere *et al.* (2017). The elemental compositions of the pumice also indicate the absence of hazardous or carcinogenic substances, thus natural pumice is considered appropriate as an adsorbent to treat polluted water.

The SEM image showed the surface morphology of natural pumice from Sungai Pasak, West Sumatra as displayed in Figure 1. The image indicated that the pumice had a highly porous, smooth surface, with a cellular and irregular texture with larger cavities, which provides suitable sites for adsorption.

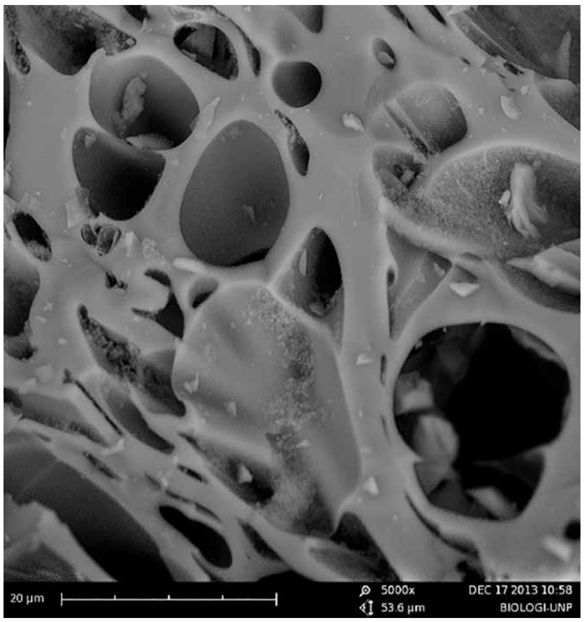

Figure 1 | SEM micrograph of natural pumice from Sungai Pasak, West Sumatra, Indonesia.

Desorption studies

Desorption studies contribute to clarifying the mechanism of metal ion removal and recovery from metal-loaded adsorbent and also for the regeneration and recycling of spent adsorbents, which in turn may reduce operational cost and protect the environment. Figure 2 shows the desorption efficiencies for iron ions using three kinds of desorbing agent were in the range of 25–37%. It was also noticeable from the figure that desorption in the acidic media for iron ions was more rapid than in basic and neutral media. At the end of the study time of 60 min, over 36.57% and 37.89% of iron was recovered from the pumice adsorbent by the acidic reagent for desorption 1 and 2, respectively. With NaOH as a basic reagent, the desorption efficiencies were 25.78% and 26.75% for desorption 1 and 2. Moreover, 31.87% and 32.39% of desorption was recorded for distilled water (neutral media). A slight increase in desorption efficiencies was observed (approximately 1%) at the second desorption cycle (desorption 2) possibly due to the accumulation of iron that could not release at the second adsorption (adsorption 2).

The highest iron recovery from the iron-laden pumice was observed in an acid medium. This is probably because in acidic medium the functional groups in the natural pumice become protonated and do not attract the positively charged metal ions, and so the protons replace the bound metal ions. The poor desorption of less than 30% observed in basic medium may be due to the coordinating ligands being deprotonated, hence bound metal ions find it difficult

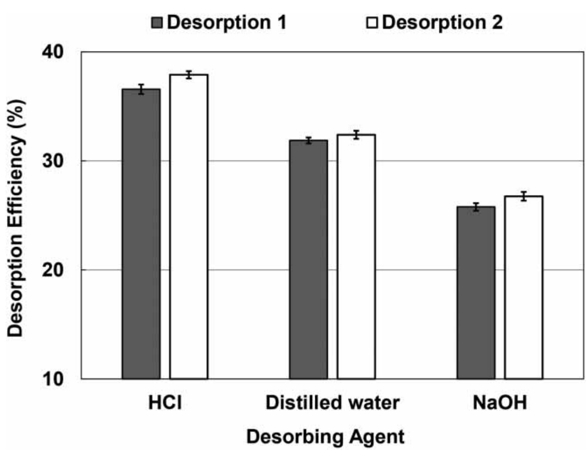

Figure 2 | Effect of desorption media on the recovery of iron from iron loaded natural pumice.

to be detached from the adsorbent. Similar results was obtained by Wankasi *et al.* (2005), who studied desorption of Pb^{2+} and Cu^{2+} from Nipa palm (*Nypa fruticans Wurmb*) biomass. The result confirmed that acidic medium (HCl) constitutes the best desorbing agent as it has the highest desorption efficiency, over 70 and 60% for Pb^{2+} and Cu^{2+}, while basic and neutral media were less than 20% and 4% of both metal ions. In addition, Li *et al.* (2009) also investigated the regeneration of modified spent grain used for lead removal. They used desorbing agents like HCl, NaOH, NaCl and ultrapure water in their research. They observed that HCl was the most efficient desorbing agent with 86% of efficiency, whereas NaOH, NaCl and ultrapure water were not effective in removing the adsorbed lead. When HCl is used as a desorbing agent, the adsorbent surface is completely covered by H^+ ions while the coordination sphere of chelated metal ions is disrupted. Thereafter, the metal ions cannot compete with H^+ ions for adsorption sites and subsequently metal ions are released from the adsorbent surface into the solution. At the end of the desorption process, the adsorbent becomes totally protonated to be ready for the next adsorption cycle (Li *et al.* 2009).

Although complete desorption was not achieved, it is clear from the results that the HCl as an acidic medium was the best desorbing and recovery agent, compared with the NaOH as a basic medium and distilled water as a neutral medium. Desorption is a time dependent variable. The residence time is very essential because the higher the residence time, the longer the contact between the desorbing agent and the metal loaded adsorbent (Wankasi *et al.* 2005). However, residence time also must be relatively small in order to protect the deterioration of the biomass and enhance its recyclable lifetime. In this study, the desorption time was 60 min. It may be possible to obtain 100% desorption if the contact time is extended. Nevertheless, the reusability of the pumice must be considered, because the longer the adsorbent is in contact with the desorbing agent, the faster the adsorbent may lose its reusability.

Results of SEM analysis that observed the surface morphology of the natural pumice after iron adsorption and desorption are shown in Figure 3. It was revealed that desorption could affect the surface and pore condition of the natural pumice. After adsorption, it can be seen that small particles of amorphous precipitate adhere to the pumice

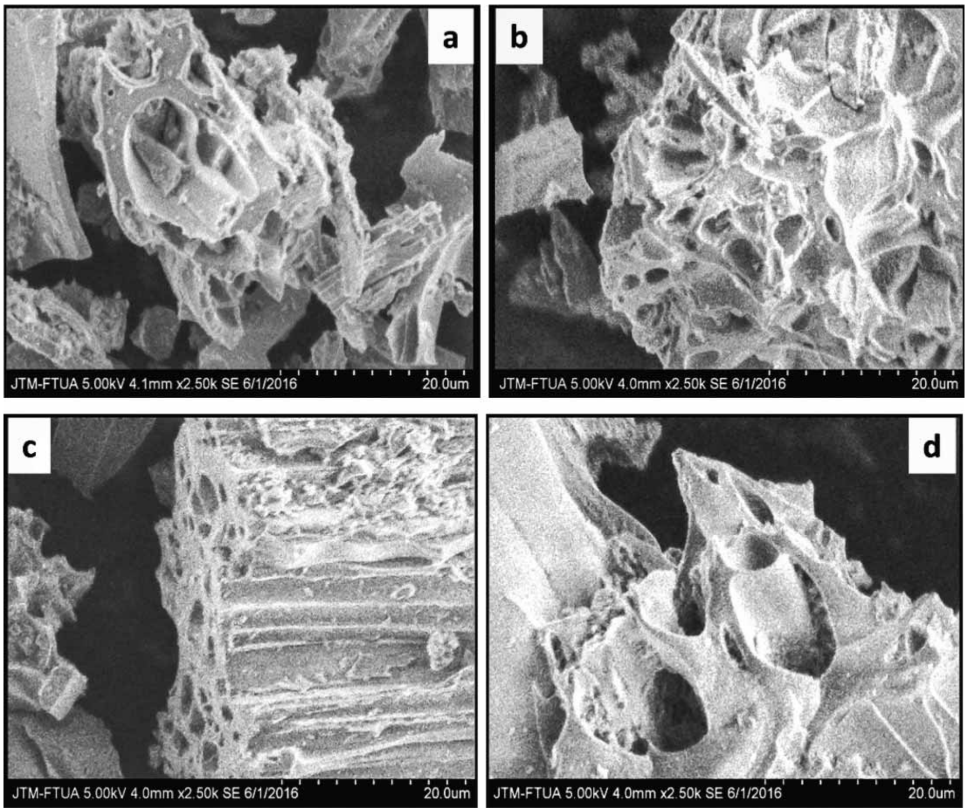

Figure 3 | SEM images of the natural pumice after iron adsorption (a), iron desorbed by HCl (b), distilled water (c) and NaOH (d).

surface, which may be due to the presence of iron ions on the pumice surface (Figure 3(a)). Different conditions of pumice surfaces were observed after desorption by different agents. A smoother surface, more porous and relatively regular texture were obtained in the natural pumice after being desorbed by HCl (Figure 3(b)), while a rough structure on surfaces was found in pumice desorbed by distilled water (Figure 3(c)) and NaOH (Figure 3(d)). This may due to the iron ions that still fill the natural cavities of the natural pumice.

Figures 4 and 5 show removal efficiency and iron uptake from three steps of adsorption with different desorbing agents. As shown in Figure 4, removal efficiency of iron ions for recycled pumice can be maintained at 90% even in the third cycle when using HCl as the desorbing agent. There is a slight decrease in removal efficiency obtained using pumice desorbed by distilled water and NaOH that may due to incomplete desorption of iron ions. Similar trends were observed by Gupta & Babu (2009) as well as Kwon & Jeon (2012) in using a regenerated adsorbent that could be maintained at high efficiency for more than two cycles.

Figure 6 shows the amount of iron retained by natural pumice at the end of each adsorption or desorption step with different desorbing agents. At adsorption 1, when the fresh adsorbent was used, approximately 45 mg/g of iron uptake was obtained. The efficiency of HCl as a desorbing agent was relatively higher than that of distilled water and NaOH, confirmed by the lowest iron retained by the adsorbent at the end of desorption 1 and desorption 2 found when using HCL as desorbing agents. The reuse of the remaining natural pumice in a second and third adsorption cycle (adsorption 2 and adsorption 3) slightly increased the iron uptake or was retained by the natural pumice. This may be related to chemical changes in the natural pumice produced by acid solution (HCl). Many researchers reported that acid solution like HCl can be used to modify the adsorbents in order to improve their adsorption capacity. It may due to the ability of the acid solution to removal of impurities on the adsorbent surface, decreased the organic content of adsorbent, increased porosity, breakdown of the cell wall or generation of new sorption active sites (Sepehr et al. 2014). Consequently, it could be concluded that natural pumice from Sungai Pasak, West Sumatra, Indonesia can be sufficiently reused up to three cycles of adsorption-desorption.

Although three cycles of adsorption-desorption equilibrium studies is not enough to sufficiently access the desorption capacity of a desorbing agent, the study has demonstrated that the selection of an eluant may be influenced by the consideration of the reusability of the pumice

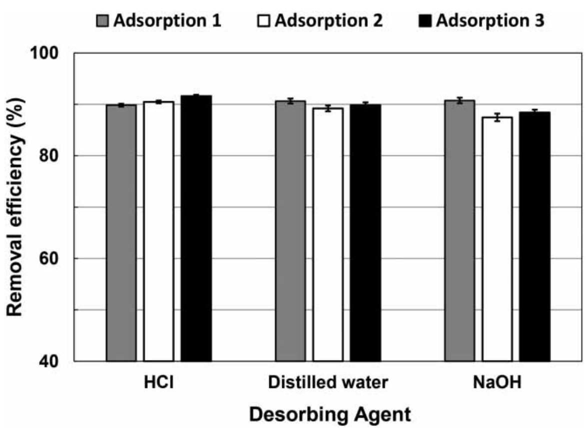

Figure 4 | Removal efficiencies of iron from three steps of adsorption with different desorbing agents.

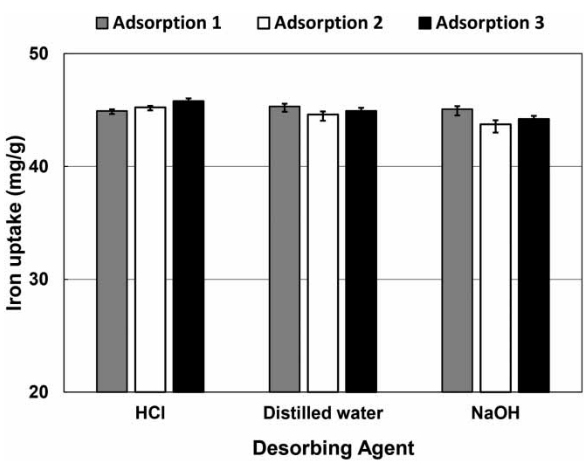

Figure 5 | Fe uptake from three steps of adsorption with different desorbing agents.

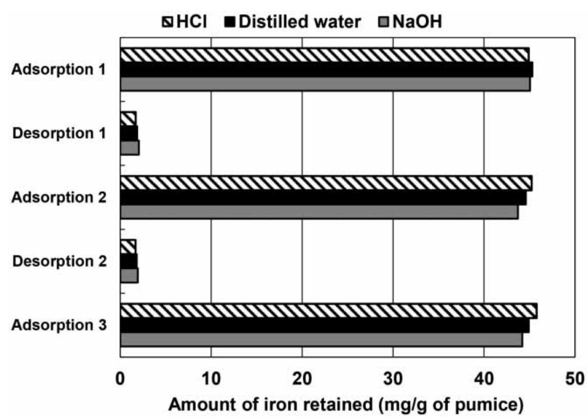

Figure 6 | Iron retained by the natural pumice after sorption-desorption cycles with different desorbing agents.

as well as the desorption kinetics. The results of this investigation are quite useful for further researches especially in trying concentration and time gradients to obtain the optimum working concentration for acid media and contact time for desorption.

CONCLUSIONS

This study focused on desorption and regeneration of natural pumice from Sungai Pasak, West Sumatra, Indonesia for iron removal from aqueous solution. For that, three adsorption-desorption cycles with different desorbing agents were conducted. The highest desorption efficiency was observed by using HCl 0.1 M as the desorbing agent within 60 min of contact time that offered 36.57% and 37.89% of efficiencies at desorption 1 and 2, compared with distilled water and NaOH. The experimental results also suggest that the removal efficiency of recycled natural pumice for iron ions can be kept up to 90% through three cycles, although there was a slight decrease when using distilled water and NaOH. Because of its excellent reusability, further investigations are needed to study some factors to increase the desorption efficiency of iron in natural pumice, like the optimum contact time and concentration of desorbing agents.

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