



Original Article

Pellet activated carbon production using parawood charcoal from gasifier by KOH activation for adsorption of iron in water

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Abstract

The objective of this research was to produce pellet activated carbon (PAC) using parawood charcoal from the gasification process. Solutions of 0.1-2.0 M KOH were chosen for chemical activation followed by thermal activation in a tube furnace at 600-800°C for 30-120 min. The percent yield was 48.47wt% using the optimum conditions of 2.0 M KOH and 700 °C for 90 min. The results of iodine number and methylene blue adsorption of PAC were 602.75 and 202.04 mg/g, respectively, which indicated that the pore size was mostly in the range of mesopores and macropores. The Brunauer-Emmett-Teller surface area of PAC was found to be 705.47 m²/g. Iron adsorption was carried out by feeding solutions of iron in various flow rates (0.1-1.0 l/min) and concentrations (5-15 mg/l) through a filter cartridge. The PAC had higher iron adsorption compared to commercial activated carbon. Steam regeneration can effectively be performed to reuse the PAC as a recovery resource.

Keywords: activated carbon, extrusion, iron adsorption, rubber wood charcoal

1. Introduction

Activated carbon (AC) is a very high surface area carbon that can be produced from any carbonaceous material rich in elemental carbon (Jiwalak, 2002). The production of AC has been done from many kinds of raw materials, such as agricultural by-products and residual wastes (Kalderis *et al.*, 2008). A gasification process refers to the thermochemical conversion of wood as biomass to obtain electrical energy, tar, and solid residuals. Parawood (*Hevea brasiliensis*) is the most appropriate fuel to be used with a gasifier and wood charcoal is generated in the amount of one out of four of wood input. Charcoal is a component of the gasifier solid residuals that is composed of unreacted carbon with various amounts of siliceous ashes. The irregularly shaped particles have a well-defined pore structure and excellent potential as an adsorbent and precursor to AC. The charcoal is normally collected in an industrial area and sold at a very low price. To maximize the

economics and environmental benefits in energy generation system, the value added utilization of the charcoal needs to be considered as a precursor for AC production.

The production of AC consists of a carbonization process by burning the precursor at 200-500 °C to get charcoal followed by physical or chemical activation. The benefit of using charcoal as a precursor in AC production is the single step approach to reduce the manufacturing costs. The extrusion of pellet wood charcoal with a binder is a critical step to increase the strength of the AC. The binders have included polyvinyl alcohol, starch, and oil which take into account the porosity and pore size of the AC (Abechi *et al.*, 2013). Sulfuric acid, phosphoric acid, and potassium hydroxide have been used as activating agents to stimulate charcoal in chemical activation for AC production (Hirunpraditkoon *et al.*, 2011). The chemicals can impregnate the pores and cause the decay of the bitumen and organic compounds in the charcoal. The advantage of this method is the ability to operate at faster and at lower temperatures than a physical method. Potassium hydroxide (KOH) has been widely used in AC preparation with a greater specific surface area and a good pore development. KOH also enhances the specific surface area and the formation of -OH functional

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groups on the carbon surface (Viswanathan *et al.*, 2009). Both mesopores and micropores are formed as a result of the intercalation of potassium into the carbon network during the activation (Teng *et al.*, 2011).

In groundwater, iron is in the form of iron (II) and the concentrations are usually in the range of 0.5-10 mg/l. The iron precipitate can cause damage to clothing during washing. Normally, drinking water contains iron <0.3 mg/l. If the human body has too much iron, it can lead to health problems (McFarland I 2015). The use of AC as an alternative treatment to iron contaminated water using adsorption principles is an interesting option. Regeneration of the AC is important as an economical adsorption technique amongst several methods that are currently under research and application. Steam regeneration works especially well and is widely popular due to favorable economics and the effectiveness to regenerate activated carbon. After regeneration of an adsorbent bed, the condensed adsorbate can be recovered by separation techniques.

This study aimed to produce a pellet activated carbon (PAC) from parawood charcoal derived from the gasification process using KOH as an activating agent and activation in a tube furnace. Charcoal pellet (CP) extrusion is performed by a mixture of charcoal powder and cassava starch binder that is extruded into a cylindrical shape. PAC was produced by soaking the CPs in KOH solution, calcining in a tube furnace, and measuring the % yield of the product. The PAC properties were measured by iodine and methylene blue adsorption. The ability between PAC and AC to remove iron was compared and tested in a filter column. Lastly, steam regeneration of the spent PAC was investigated to desorb iron from the pores of the PAC for economic use.

2. Experiments

2.1 Charcoal pellet preparation

The raw material for the PAC preparation was acquired from a gasification process of parawood for fuel based on carbonaceous materials conversion. The pelleting process of the charcoal was performed to produce CPs in four steps: i) crushing and screening to produce the charcoal powder with a standard sieve size of 37-74 microns, ii) moistening and combining the powder mixture with water and cassava starch binder, iii) forming cylinder-shaped agglomerate through extrusion, and iv) drying the final pellet product. The ratios of charcoal powder to cassava starch binder to water varied in proportions of 1:0.30:1, 1:0.35:1, and 1:0.40:1 by material weight. Then, the charcoal mixture was processed through an extruder at a diameter of 2 mm, gently cut with a blade to get the 1 cm length of CP for drying purposes. The effectiveness of proportional mixing in a pelleting process to create durable bonding in pellets was determined by testing the mechanical property of compressive strength.

2.2 Preparation of pellet activated carbon

KOH was used to chemically activate the CPs by a suitable proportion of charcoal and binder. The KOH solution was used to impregnate 200 g of CPs at a certain impregnation

ratio (weight of activating agent/weight of CP) of 2:1 v/w for 24 h. KOH solution concentrations of 0.1 to 2.0 molar were used to characterize the PAC. The soaked pellets were dried at 110 °C for 24 h and activated in a horizontal stainless steel tube furnace. The tube was 10 cm in diameter and 54 cm in length and was installed on the furnace at a heating rate of 10 °C/min under N₂ gas flow rate of 4 l/min. Thermal activation at temperatures of 600 to 800 °C coupled with different carbonization times of 30 to 120 min was employed to enlarge the surface area of the PAC from the charcoal. After cooling to room temperature, the prepared PAC was washed thoroughly with 5% HCl for 15 min followed by distilled water until the pH value of the filtrates became neutral (pH=7), and then dried at 110 °C for 24 h.

2.3 Characterization of PAC

In AC preparation, the yield is calculated using Equation (1) and is defined as the final weight of AC produced after activation divided by the initial weight of raw material on a dry basis (Jin *et al.*, 2010). The iodine number is a technique employed to determine the adsorption capacity of activated carbons. The iodine number can be determined to apply the ASTM D4607 standard method (Srikun, 2007) to approximate the surface area and microporosity of activated carbons in good precision. The surface activity of PAC towards methylene blue adsorption property was also measured (Analysis according to JIS K 1474-1991) (Pinkaw, 2013).

$$\text{Yield of activated carbon (wt\%)} = \frac{\text{weight of PAC}}{\text{weight of charcoal}} \times 100 \quad (1)$$

2.4 Application of iron adsorption from water

The continuous experiment was carried out to assess the sorption capacity of iron on PAC. The schematic diagram of the experimental setup used in this study is shown in Figure 1. The experiment consisted of iron-contaminated water in a storage tank, pump, valve, adsorption cartridge test, and filtered water reservoir. The adsorption cartridge was packed with 500 grams of PAC. Water filtration using PAC is an adsorptive process in which iron contaminant is adsorbed onto the carbon surface. The ability of the adsorption depends mostly on the operating parameters of water flow rate (F_w) and the concentration of the iron at the inlet. Concentration of 5-15 mg/l of iron solutions were prepared for feeding into the adsorption cartridge at flow rates of 0.1-1.0 l/min. Water samples were taken from the filtration inlet and outlet of the PAC packed bed to analyze the iron adsorption efficiency by spectrophotometry. The adsorption activity of PAC was also compared to commercially available AC. The amount of iron adsorbed was calculated from Equation 2 to indicate the experimental adsorption capacity:

$$q = \frac{(c_0 - c_t)V}{m} \quad (2)$$

where q is the amount of iron adsorbed by AC (mg/g), C_0 is the initial iron concentration (mg/l), C_t is the final metal concentration after filtration (mg/l), V is the solution volume (l), and m is the AC dose or weight (g).

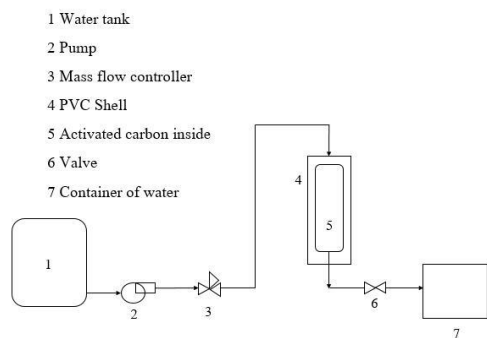


Figure 1. Schematic diagram of the experimental setup for application of PAC in iron adsorption from water.

2.5 Regeneration of spent activated carbon

The tank for steam regeneration of spent PAC was a 1 l stainless steel tank designed for high pressure. The spent PAC was placed on a perforated plate installed at 50% of the tank height over the level of water (300 ml). A regeneration temperature of 300 °C at 25 bar pressure and for 60 min was typically required for complete regeneration of the PAC. The temperature and pressure were monitored by a thermocouple and pressure gauge fixed at the top of the unit. The regenerated PAC was evaluated in the adsorption cartridge to measure the adsorption activity of iron adsorption from the water.

3. Results and Discussion

3.1 Effect of charcoal pellet preparation

All extruded formulations of CPs were hardened using cassava starch as a binder. The influence of the mixing proportions of charcoal to cassava starch to water at 1:0.3:1, 1:0.35:1, and 1:0.4:1 on the compressive strength was studied. The dry compressive strengths of all measured samples were in the range of 45-57 MPa (Figure 2). We observed an increase in strength with added cassava starch in the mixture up to the optimum ratio of 0.35 of cassava starch. At the ratio of 0.40 of cassava starch, the compressive strength did not increase further. After overnight immersion of the CPs in water, the % fraction of charcoal in water was found to be less than 1.25% cassava starch with the ratio of 0.35 added to the mixture.

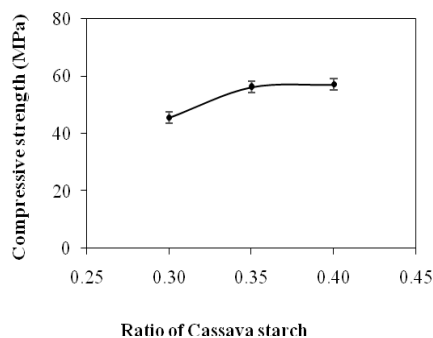


Figure 2. Effect of the cassava starch weight ratio added in charcoal powder extrusion mixture on the compressive strength of CP products.

3.2 Effect on PAC preparation

3.2.1 Effect of KOH concentration

The PAC was prepared by impregnation of CP with KOH solutions in the range of 0.1 to 2.0 molar, followed by carbonization in the tube furnace. At a carbonization temperature of 600 °C for 30 min, the yields, iodine numbers, and the adsorption of methylene blue of the different samples are shown in Figure 3. The results clearly showed that, as expected, an increased KOH concentration led to a decrease in the total yield of the process. Essentially, charcoal is not a single compound. The elemental compositions of charcoal are C, H, O, N, S, and ash with proximate analysis of fixed carbon >70%. In chemical activation, these structures decompose and most of the non-carbon elements are liberated in the form of liquid as tars and gases, leaving behind a rigid carbon skeleton in the form of aromatic sheets and strips. A higher concentration of KOH dispersed and reduced non-carbon elements that may obstruct pores. Thus, at the 2.0 molar concentration of KOH, the percent yield was 75.67, which was the lowest value compared to other concentrations (Figure 3a). The charcoal in contact with higher KOH concentrations resulted in the removal of more disorganized carbon and the formation of a well-developed microspore structure. The iodine number and methylene blue adsorption of PAC of the AC increased up to 470.5 and 116.2 mg/g, respectively (Figures 3b and 3c). Therefore, the 2.0 molar concentration of KOH was the optimal concentration because of the absorption ability of the PAC.

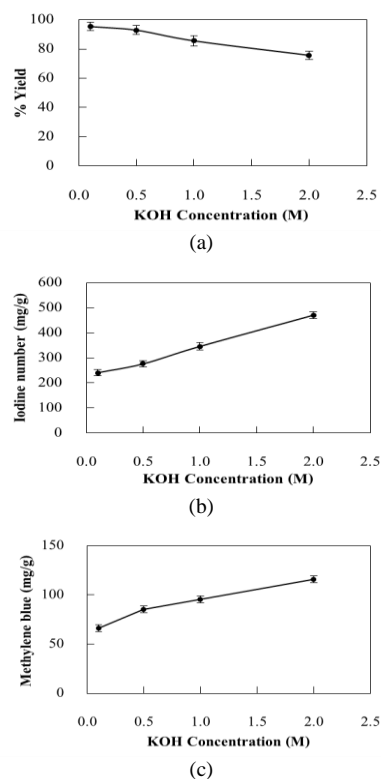


Figure 3. The effect of KOH concentration on % yield (a), iodine number (b), and methylene blue adsorption (c) of PAC at carbonization condition of 600°C for 30 minutes.

3.2.2 Effect of carbonization temperature

The effect of carbonization temperature was studied in ranges of 600-800 °C for 30 min using a KOH concentration of 2.0 M for chemical activation. Yield, iodine number, and methylene blue adsorption of the PAC obtained in different experimental conditions are given in Table 1. It was found there was an increase in iodine number value when the temperature increased from 600 °C to 700 °C and a decrease when increased from 700 °C to 800 °C. This can be explained by transformation into a macro- and mesoporous structure after the pore walls were demolished at high temperatures (Saeed *et al.*, 2003). It was seen that, at this temperature range, the methylene blue adsorption increased according to the increase of temperature. The yield of PAC, obtained from the impregnation of 2.0 molar KOH at 700 °C from charcoal as a raw material, was measured as 60.16% and the iodine number was 524.17 mg/g. The optimum operating temperature condition was found at 700 °C which indicated the best adsorption ability. The use of a lower temperature, compared to physical carbonization, is compensated by the interaction between the chemicals and the carbon skeleton.

Table 1. Effect of carbonization temperature in tube furnace on properties of PAC.

Carbonization Temperature (°C)	Carbonization Time (min)	Yield (%)	Iodine number (mg/g)	Methylene blue (mg/g)
600	30	75.68	470.50	116.5
700	30	60.16	524.17	159.53
800	30	59.97	508.44	161.91

3.2.3 Effect of carbonization time

The carbonization time was varied in the range of 30-120 min at 700 °C for PAC preparation using the 2.0 molar KOH for chemical activation. Yield, iodine number, and methylene blue adsorption of PAC at different carbonization times are given in Table 2. Increasing the carbonization time led to a decrease of yield because greater porosity occurred which could be determined from the reduced weight of PAC. The optimum carbonization time was found at 90 min, which was determined from the good performance of the iodine number (602.75 mg/g) and adsorption activity of methylene blue (202.04 mg/g). It can also be seen that, at the carbonization time of 120 min, the iodine number decreased. The excessive processing may cause serious degradation of the carbon layer which is detrimental to the carbon structure.

Table 2. Effect of carbonization time in tube furnace on properties of PAC.

Carbonization Time (min)	Carbonization Temperature (°C)	Yield (%)	Iodine number (mg/g)	Methylene blue (mg/g)
30	700	60.16	524.17	159.53
60	700	55.45	550.92	125.43
90	700	48.47	602.75	202.04
120	700	49.48	570.95	200.68

3.3 Properties of AC

3.3.1 SEM micrographs

The SEM micrographs of the parawood charcoal, PAC, and regenerated PAC are shown in Figure 4. The SEM micrograph of the parawood charcoal indicated that a few scattered pores were generated on the surface as the volatile material had been released, which left most of the non-volatile char during carbonization of the charcoal in the gasification process (Figure 4a). The PAC had many different pores sizes on its surface which indicated that the structure had been destroyed and a dense porosity was formed through KOH activation (Figure 4b). The irregular pores were related to the decomposed organic matter and the gas generated in the activation process. The surface of the regenerated PAC regenerated had regular pores according to the heating and purging by steam that affected the pores of the adsorbent used (Figure 2c).

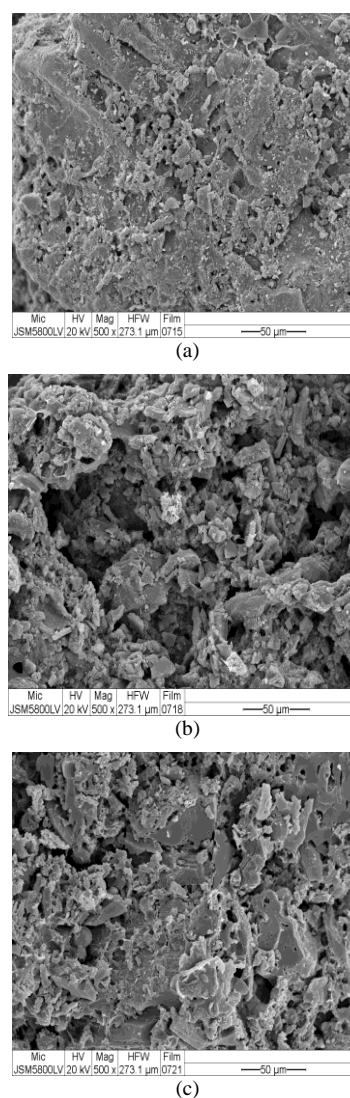


Figure 4. SEM micrographs of the para wood charcoal (a), PAC (b) and regenerated PAC (c).

A similar trend in the change of the surface morphology structures of waste printed circuits was found from the literature (Quan *et al.*, 2010). After the carbonization and activation process, the structure had different sizes and shapes of the pores. The pores were formed mainly from the removal of KOH, leaving a space that was previously occupied by the compounds (Teng & Wang, 2000).

The BET surface area of the PAC calculated by the Brunauer-Emmette-Teller (BET) method was 705.47 m²/g. The total pore volume and micro-pore volume of PAC were 0.4831 and 255.23 cm³/g, respectively.

3.3.2 Comparison of the properties to some other carbonaceous materials

In a comparison of the textural properties and maximum adsorption capacities of the PAC with other reported values for commercial AC (Ahmad *et al.*, 2012; Fierro *et al.*, 2008; Srivastava *et al.*, 2006), the PAC seemed to be an alternative precursor for the commercial AC preparation, in which its sorption capacity of iron was higher than the commercial AC. PAC could be employed as an efficient carbonaceous adsorbent as an alternative to the commercial AC for the removal iron from water.

3.4 Study of iron adsorption efficiency from water

3.4.1 Effect of the iron solution flow rate

The effect of flow rate on the adsorption of iron by PAC from aqueous solutions was investigated. The flow rates were 0.1, 0.5, and 1.0 l/min using 500 g of AC and the initial iron concentration in the water was 10 mg/l. The effects of flow rate on the breakthrough curve for iron adsorption from the water are shown in Figure 5. The results showed that a flow rate of 0.5 l/min could reduce the iron content efficiently and have a longer breakthrough time than flow rates at 0.1 or 1.0 l/min. These breakthrough curves showed that the column performed better at a lower flow rate which resulted in a longer breakthrough, exhaustion times, and percent removal. At a lower flow rate, the breakthrough time was longer than the higher flow rate because a longer time is required to reach its effective bed load. As the flow rate increased, the breakthrough curve became steeper since a shorter time was required to attain the effective bed load and hence the effective adsorption capacity of column reduced. This is attributed

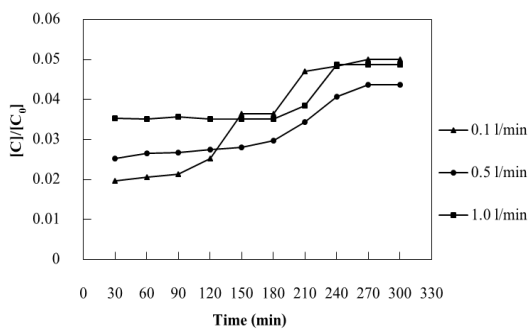


Figure 5. Breakthrough curves for iron adsorption in water on pellet activated carbon (PAC) at different flow rates (initial adsorbates concentration = 10 mg/L, weight = 500 g).

to the insufficient residence time of the adsorbate solutes in the column to allow for diffusion of the solute into the pores of the adsorbent (Girish & Murty, 2015; Mohammed *et al.*, 2016; Tan *et al.*, 2008). But in the last 120 min, the flow rate at 0.1 l/min showed a lower performance than the 0.5 and 1.0 l/min flow rates because the activated carbon was saturated with iron. The increase in the breakthrough time could be ascribed to the longer distance it takes the mass transfer zone to move from the entrance of the bed to the exit when the flow rate is inappropriate.

3.4.2 Effects of contact time and initial iron concentrations

The results of this experiment are displayed in Figure 6 which shows the effects of contact time on the adsorption of iron onto the PAC at different initial concentrations (5, 10, and 15 mg/l) at room temperature (30 °C). Adsorption studies were carried out for five hours. It was observed that the initial adsorption was rapid and a larger amount of iron was initially removed in a contact time of 30 min for all studied adsorbents. The adsorbed iron of 5 mg/l was found to vary linearly with time at the beginning of adsorption in the initial stages. Then saturation was attained which is called the equilibrium time. At 10 and 15 mg/l iron concentrations in the second stage, assigned to intra particle diffusion, shows a decrease in the slope due to the gradual increase in the adsorption rate after 120 min, which was not observed in the concentration of 5 mg/l. It was also predicted that the growing trend stopped when the state of equilibrium was reached. Adsorption of iron increased with increasing contact time and the adsorption capacity of PAC increases with an increase of the initial iron concentration from 5 to 15 mg/l for all studied adsorbents. Since the initial concentration provides an important driving force to overcome all mass transfer resistance, similar results have been reported in the literature for the removal of heavy metals (Akl *et al.*, 2013; El-Zahhar *et al.*, 2013; Youssef *et al.*, 2006).

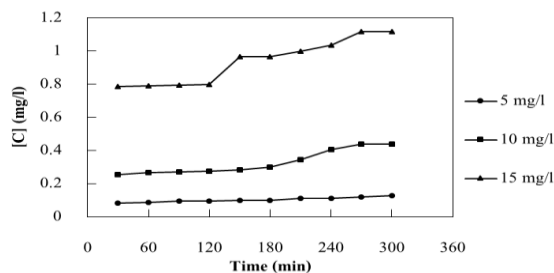


Figure 6. Effects of contact time on the adsorption capacity at different initial concentrations (iron solution flow rate = 0.5 L/min, weight = 500 g).

3.4.3 Comparative performance of commercial AC

The experiments were carried out with commercially available AC and the PAC for the adsorption of iron from water. The commercial AC had an iodine number of 1,150.50 mg/g and a methylene blue adsorption of 85.15 mg/g. Figure 7 describes representative results of the breakthrough curves for these adsorbents obtained under the operating conditions of adsorbent weight of 500 grams, flow

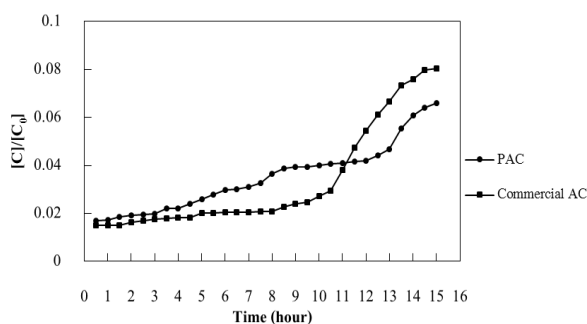


Figure 7. Breakthrough profiles of commercial AC comparing with PAC in iron adsorption efficiency. ($C_{inlet} = 5$ mg/L, $F_{water} = 0.5$ L/min, and $W = 500$ g).

rate of 0.5 l/min, and iron inlet concentration of 5 mg/l. The adsorption of iron using the commercial AC was similar to the PAC. The performance of PAC was a little superior to that of the commercial AC for approximately 15 h. In the first 11 hours, the commercial AC showed a better performance than the PAC. However, in the last 11 hours, the PAC was capable of absorbing up to 14 h. The experimental iron adsorption capacities on the PAC and commercial AC were 3.95 and 3.10 mg/g, respectively. In addition, PAC is 3 times cheaper than the commercial AC.

3.5 Regeneration of used PAC

Spent adsorbents are usually discarded as waste which is a serious environmental problem. AC can be regenerated and put to reuse in the same process line. Steam is readily available in the process industry and can be utilized for regeneration of spent adsorbents. The use of steam for AC regeneration has multiple benefits (Irfan *et al.*, 2013). The heating and purging properties of steam in the pores are important for adsorbent regeneration. This study included the regeneration of PAC with steam at 300 °C for 60 min, at a pressure of 25 bar in a batch regeneration system. and tests for adsorption experiment for the iron adsorption efficiency at $[C_0] = 5$ mg/L, $F_w = 0.5$ L/min, and 500 g of PAC. The comparison of iron adsorption efficiency between the fresh PAC and regenerated PAC is described in Figure 8. It was found that the regenerated PAC still had good performance in iron adsorption efficiency following the standards of WHO (iron not to exceed 0.3 mg/l or the breakthrough curve ($[C]/[C_0] < 0.06$) and the iron adsorption efficiency was as good as the PAC. The experimental results of the iron adsorption capacities of the PAC and regenerated PAC were 3.95 and 3.53 mg/g, respectively. Therefore, this research suggests that the regeneration of spent AC is reasonable. Reuse by regeneration of the adsorbent could provide a double reward by stabilizing the adsorbents and recovering a resource by reutilization and thereby minimizing demand for virgin adsorbents.

Regenerated PAC by steam can remove the iron adsorbed onto the PAC surface. The iodine number was 510 mg/g which suggested that the regenerated PAC can be effectively used again.

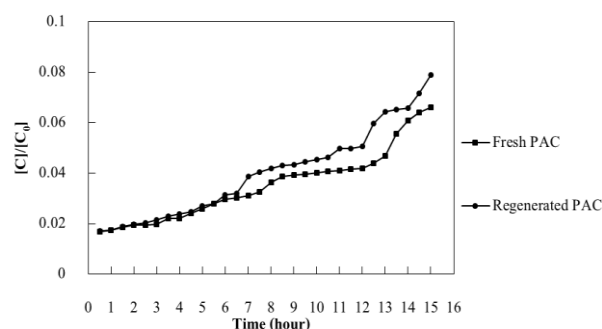


Figure 8. The breakthrough curve of iron adsorption efficiency for fresh PAC and regenerated PAC. ($[C_0] = 5$ mg/L, $F_w = 0.5$ L/min, and 500 g of PAC).

4. Conclusions

Parawood charcoal derived from the gasification process can be used for preparation of PAC. The percent yield of PAC prepared from parawood charcoal was 48.47wt% and the PAC was activated using 2.0 molar KOH at a temperature of 700 °C for 90 min. The results of iodine number and methylene blue adsorption were 602.75 and 202.04 mg/g, respectively, and the BET surface area of the PAC was 705.47 m²/g. The PAC filter cartridge had a higher iron adsorption capacity compared to the commercial AC. The steam regeneration process can effectively be performed for PAC reuse. PAC from this research work can be used as an excellent alternative. It is an effective and inexpensive material for removal of high amounts of iron from water.

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