*Desalination and Water Treatment*
www.deswater.com

doi: 10.1080/19443994.2016.1168133

57 (2016) 29137–29142
December

# Regeneration of spent activated carbon from industrial application by NaOH solution and hot water

Su-Huan Kow*, Muhammad Ridwan Fahmi, Che Zulzikrami Azner Abidin, Soon-An Ong, Naimah Ibrahim

*School of Environmental Engineering, University of Malaysia Perlis (UniMAP), Kompleks Pusat Pengajian Jejawi 3, 02600 Arau, Perlis, Malaysia, emails: suhuan_kow@yahoo.com (S.-H. Kow), drfahmi@unimap.edu.my (M.R. Fahmi), zulzikrami@unimap.edu.my (C.Z.A. Abidin), saong@unimap.edu.my (S.-A. Ong), naimah@unimap.edu.my (N. Ibrahim)*

Received 2 September 2015; Accepted 7 March 2016

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ABSTRACT

Spent activated carbon (SAC) must either be disposed of at a landfill or regenerated and reused in industries. Regeneration of SAC is a more preferable option as it can cut down replacement and disposal cost as well as reduce secondary pollution. Chemical regeneration is more suitable and economically feasible option for small-scale industrial application as compared to thermal regeneration. In this study, SAC is regenerated using sodium hydroxide (NaOH) solution and hot water. The aim of this study is to evaluate the effectiveness of NaOH and hot water regeneration of SAC. The optimal regeneration condition for SAC was determined and the regeneration performance was assessed by iodine and methylene blue (MB) adsorption test. Finding shows the optimal regeneration condition of carbon regeneration is 15 mL of 6 N of NaOH solution per g of carbon with 30 min of contact time. The estimated surface area, micropore volume, and total pore volume of batch and column regenerated SAC were 899.1 $m^2/g$, 0.8702 $cm^3/g$, 0.9521 $cm^3/g$; and 849.0 $m^2/g$, 0.8519 $cm^3/g$, and 0.9450 $cm^3/g$, respectively. The regeneration efficiency of regenerated SAC is comparable to fresh activated carbon with regeneration efficiency more than 90% in iodine adsorption test and 98% in MB adsorption test.

*Keywords:* Spent activated carbon; NaOH regeneration; Adsorption test; Iodine number; Methylene blue number

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## 1. Introduction

Activated carbon (AC) is a carbonization product of carbonaceous materials which utilizes the principle of adsorption to prevent environmental pollution. AC has been used for decades in industries as adsorbent to remove impurities from gases [1] or liquids [2] and to polish effluent for meeting stringent discharge standards [3]; as catalyst to improve process efficiency [4] and as gas container to store pure gas [5]. The adsorption performance of AC is determined by available surface area for physisorption and available adsorption site for chemisorption [6]. Despite the fact that

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*Corresponding author.

*Presented at the 2nd IWA Malaysia Young Water Professionals Conference 2015 (YWP15) Aspiring Beyond Water Solution, March 17–20, 2015, Langkawi Island, Malaysia*

AC has excellent adsorption ability, the available surface area and adsorption sites are limited [7]. In industrial applications, AC requires to be replaced with fresh activated carbon (FAC) once the area and sites of AC were covered by adsorbate in which the AC loses its adsorption capability and became spent activated carbon (SAC). The fate of SAC is either disposed of at a landfill, regenerated or reused in industries [8]. Regeneration of SAC refers to restoration of the available surface area and adsorption site on SAC or removal of adsorbate on SAC via desorption and/or decomposition. Many researches had studied on methods for carbon regeneration due to the fact that regeneration is cheaper than replacement of SAC. Furthermore, regeneration and reuse of SAC is also a practical way to reduce secondary pollution and conserve natural resources [9,10].

Carbon regeneration methods can be categorized into four major groups based on the mechanisms and agents involved in regeneration, namely thermal regeneration, chemical regeneration, microbiological regeneration, and vacuum regeneration [11,12]. However, microbiological regeneration and vacuum regeneration are not feasible for industrial application as the former requires long regeneration time [13–15] and the latter is not well investigated on its regenerative performance [12]. Thermal regeneration methods such as thermal swing adsorption [16], steaming [17], electrothermal swing adsorption [18], microwave regeneration [19], and gasification [20], involve heating process to desorb or degrade adsorbates on SAC. On the other hand, chemical regeneration methods include regeneration with liquid water [21], NaOH [22] and solvents [23], supercritical regeneration [24], electrochemical regeneration [25], and thermal oxidative regeneration [26] involve extraction, pH changes, or degradation in SAC regeneration. While many researchers have done extensive research and review on the carbon regeneration methods, solely thermal regeneration is used in industries [20].

In Malaysia, the quantity of collected SAC in the year 2012 is 1,405.59 tonne [27] which is less if compared with other countries. As one of the scheduled wastes, SAC must dispose of at prescribed premises only and industries will be charged RM 2,790 per tonne of SAC [28] for disposal cost. Considering the fate of SAC and the cost involved—disposal cost and replacement cost of approximately RM 4,000 per tonne of FAC, regeneration and reuse of SAC in industrial is preferable. Though thermal regeneration is the most practicable method in industrial, it is not an economically viable option in Malaysia. This is due to thermal regeneration that requires high energy and a very high volume of SAC is required to make it economically sensible. Furthermore, thermal regeneration experiences carbon loss during the regeneration process [29]. Comparing to thermal regeneration, chemical regeneration has a more attractive economic factor [9]. Chemically regenerated spent activated carbon (RSAC) experienced negligible carbon loss and could potentially improve adsorption capacity.

In this study, chemical regeneration using NaOH and hot water was employed to regenerate SAC from industrial application. NaOH was chosen instead of acid because alkali has higher regeneration efficiency for SAC from dye and organic removal applications [3,7,30,31]. Though NaOH regeneration has been studied extensively, the regeneration procedure always involve repeated rinsing of the treated SAC with large amount of water to desorb organics and excess NaOH. Hot water was employed in this study as it can accelerate the extraction and desorption by using high temperature and minimal volume of water. The regeneration mechanism of NaOH and hot water involvesing pH changes, chemical reaction, extraction, and thermal desorption. During the regeneration process, hydroxyl ion caused the pH on the adsorbent surface to change, become negatively charged, and reduce the strength of Van der Waals force and chemical bonds between the adsorbate and adsorbent. This enables adsorbates that are unreactive to NaOH to be removed easily [3,7,21]. While adsorbates that are reactive with NaOH are removed by producing soluble salt [9] or other stable compounds [12]. Hot water wash after treating with NaOH can accelerate the rate of desorption of adsorbate from SAC, while extracting the water-soluble salt and excess NaOH solution in the SAC. Moreover, low energy requirement and cheap chemical as regeneration agents are other benefits for SAC regeneration using NaOH and hot water.

The aim of this study was to evaluate the effectiveness of NaOH and hot water regeneration of SAC. The optimal regeneration condition for SAC was determined using batch method; whereas the regeneration performance was assessed by iodine number and methylene blue number, and compared with RSAC and FAC.

## 2. Material and methods

SAC and FAC samples in this study were charcoal-type AC. The SAC sample was supplied by Meridian World Sdn. Bhd., an industrial waste management company located at Sungai Petani, Kedah, Malaysia. The SAC was previously used in industrial applications for odor removal. While the FAC used was Guaranteed Reagent (GR) grade commercial AC purchased from HmbG Chemicals.

### 2.1. Regeneration procedures

SAC is regenerated using NaOH and hot water (about 90˚C) via batch and column regeneration method. In batch regeneration, SAC was rinsed with tap water and dried at 105˚C for 24 h. Dried SAC and NaOH solution were enclosed in a 250 mL glass-stoppered flask and well mixed by using N-Biotek 101MT Orbital and Reciprocating Combi-Shaker. Alkali-treated carbon was rinsed with hot water, adjusted to pH 7–8, and dried in oven at 105˚C for 24 h to acquire batch regenerated spent activated carbon (BRSAC). In column test, SAC was loaded and packed in a perspex column (300 mm L; 50 mm OD). A peristaltic pump with adjustable flow capability is used to circulate solution to the vertically mounted packed-bed adsorption column. The carbon-loaded column was washed through with tap water and contacted with NaOH solution at predetermined optimal condition. The process was followed by hot water wash and dilutes acid wash. Lastly, the carbon was dried in oven at 105˚C for 24 h to acquire column regenerated spent activated carbon (CRSAC).

The first three sets of experiment were conducted using batch method to determine the optimal operating condition for regeneration of SAC, such as concentration of NaOH, contact time, and volume of NaOH per g carbon. To determine the optimal concentration of NaOH, 1.0 g of SAC was contacted with 25 mL of 1, 2, 4, 6, 10, 15, and 20 N of NaOH for 4 h. In addition, the contact time was varied from 0.5 to 4.0 h in order to determine the optimal contact time. Furthermore, the volume of NaOH solution was varied from 10 to 50 mL to determine the optimal volume. The fourth set of experiment was conducted via batch and column method to evaluate the adsorption capacity of the RSAC. The adsorption capacity of FAC, BRSAC, CRSAC, and SAC were determined for comparative study. The regeneration efficiency of BRSAC and CRSAC were evaluated using iodine and MB number by comparing to those of FAC.

### 2.2. Analytical methods

Carbon adsorption test including iodine adsorption test and methylene blue (MB) adsorption test were conducted to evaluate the adsorption capacity of FAC, BRSAC, CRSAC, and SAC. 0.1 N Iodine solution was used as test solution for iodine adsorption test to determine the iodine number of carbon, which indicates the volume of micropore of the carbon. On the other hand, 1,000 mg/L of MB solution was used as test solution for MB adsorption test to determine the ability of carbon to adsorb large molecules.

Test procedure for iodine adsorption test adopted the ASTM D4607-94 procedure. Some modification was made, where adequate contact time was allowed to achieve adsorption equilibrium. A 250 mL glass-stoppered flask was used to enclose weighed RSAC and 0.1 N iodine solution, and shake for 48 h. Treated solution was filtered and titrated using 0.1 N sodium thiosulphate solution. As for MB adsorption test, a 250 mL glass-stoppered flask was used to enclose 0.1 g of carbon samples and 1,000 mg/L of MB solution, and contacted for 48 h to achieve adsorption equilibrium. The concentration of the treated MB solution was determined by using Hitachi U-2810 spectrophotometer. The MB number was evaluated using Eq. (1) below:

$$\text{MB number, } q_{\text{eq}}\ (\text{mg/g}) = (C_0 - C_e)/M \qquad (1)$$

where $C_0$ is the initial concentration of MB solution in mg/L; $C_e$ is the concentration of MB at equilibrium time in milligram per liter (mg/L); $V$ is the volume of the treated solution in liter (L), and $M$ is the mass of adsorbent in grams (g).

Surface area, micropore volume, and total pore volume of RSAC were estimated using iodine and MB number via modeling developed by Guerreiro [32]; whereas, the regeneration efficiency (RE) of SAC regeneration by NaOH solution and hot water was assessed using Eq. (2). The RE was evaluated using iodine and MB number of FAC and RSAC obtained in the carbon adsorption test:

$$\text{RE}\ (\%) = (A_{\text{Re}}/A_0) \times 100\% \qquad (2)$$

where RE is the regeneration efficiency in percent (%); $A_{\text{Re}}$ is the adsorption capacity of RSAC, and $A_0$ is the adsorption capacity of FAC.

## 3. Results and discussion

### 3.1. Optimal regeneration condition

#### 3.1.1. Concentration of NaOH

Fig. 1 shows the influence of concentration of NaOH solution on iodine number of RSAC. The experimental result shows that 1 N of NaOH solution can effectively restore adsorption capacity of SAC. The adsorption capacity of RSAC increases as the concentration of NaOH solution is increased from 1 to 10 N. The trend is the same as in the study of Sun et al. [9], a higher concentration of NaOH solution can result in higher regeneration efficiency. However, a further increase in concentration of NaOH from 10 to 20 N, did not improve the adsorption capacity of the carbon,

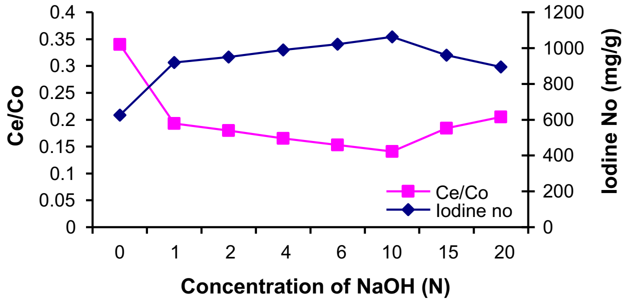

Fig. 1. Influence of concentration of NaOH solution on iodine number.

contrariwise, the result shows a gradual decrease in the iodine number of the RSAC. This phenomenon is due to the contribution of hydroxyl group ($OH^-$ ion) during the desorption process. A high concentration of hydroxyl group can hinder the adsorption performance of carbon as the surface of the carbon was occupied by $OH^-$ ion, thus reducing the available adsorption site of the carbon particles [7]. 6 N of NaOH was chosen as the optimal concentration for the following experiment as dilute NaOH solution (4–8 N) is more suitable and cost-effective in carbon regeneration [7,9,30].

### 3.1.2. Contact time

Fig. 2 shows a contact time of 30 min was enough to wash out the adsorbate on the SAC; a longer contact time did not improve adsorption capacity of RSAC. Therefore, 30 min of contact time was chosen as the optimal contact time.

### 3.1.3. Volume of NaOH per g carbon

Fig. 3 shows there is a great improvement of adsorption capacity of SAC though with only 10 mL of NaOH solution. The adsorption capacity of SAC was further improved when it was contacted with 15 mL of NaOH solution. There were no significant improvements on adsorption capacity by increasing the volume of the NaOH solution, except with 50 mL of NaOH solution. However, 15 mL of NaOH solution is more cost-effective and thus, was chosen as the optimal volume for the SAC regeneration.

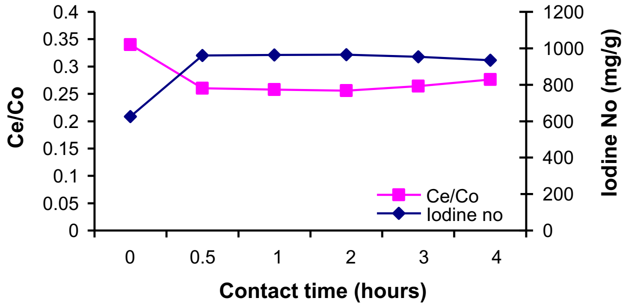

Fig. 2. Influence of contact time on iodine number.

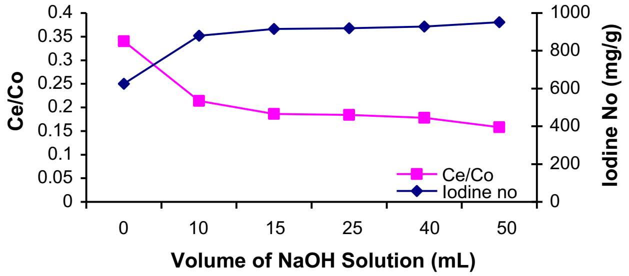

Fig. 3. Influence of NaOH volume on iodine number.

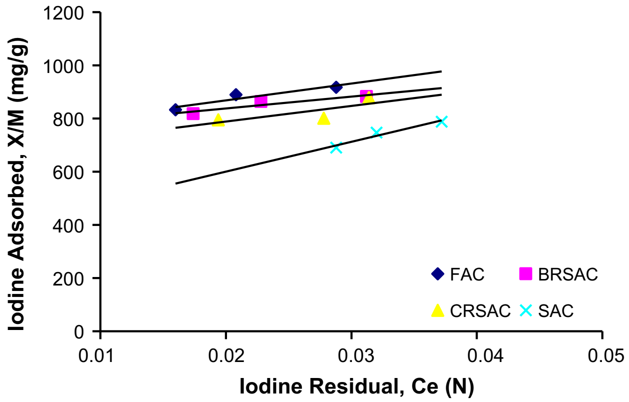

Fig. 4. Iodine adsorption isotherm of FAC, BRSAC, CRSAC, and SAC.

### 3.2. Regeneration performance

Batch and column regeneration method were conducted at optimal regeneration condition—15 mL of 6 N NaOH per g carbon with 30 min of contact time to regenerate SAC for adsorption analysis. The adsorption performance of the SAC, BRSAC, CRSAC, and FAC were evaluated through iodine and MB adsorption test. Fig. 4 shows the iodine adsorption isotherm of different types of carbon. The trend of adsorption performance of carbon is: FAC > BRSAC > CRSAC > SAC. From the graph, the adsorption isotherms of both RSAC via batch and column regeneration method are near to those of FAC, which indicates that the performance of BRSAC and CRSAC are almost as good as that of FAC.

The adsorption data was further processed by using least square fit method to estimate the iodine number of carbons. The finding shows that SAC from industrial odor application still has adsorption capacity of 625.0 mg I/g, which indicates that the micropores of the carbon are not fully occupied by impurities and thus still has adsorption capacity for iodine. On the other hand, the BRSAC and CRSAC have iodine number of 838.0 and 788.0 mg I/g, respectively, which is very close to the iodine number of FAC, 870 mg I/g. Fig. 5 shows the iodine and MB number of different types of carbon.

MB adsorption test was also carried out to estimate the MB number of different types of carbon. MB number defines the amount of MB that can be adsorbed by 1 g of carbon. The finding shows SAC has adsorption capacity of 235.5 mg/g for MB, indicating that the macropores and mesopores of the carbon still have active adsorption sites. On the other hand, the RSAC via batch and column method has MB number of 384.9 and 383.8 mg/g, respectively which is both very close to the MB number of FAC, 391.0 mg/g. The MB number of both the RSAC is as close to FAC with differences of approximately 8 mg/g.

The surface area, micropore volume and total pore volume of FAC, BRSAC, CRASC, and SAC were estimated using predetermined iodine and MB number via Guerreiro's modeling, and summarized in Table 1. RE of RSAC was assessed using predetermined iodine and MB number via Eq. (2). Results show regeneration via batch and column methods which were 96.3 and 90.5% in iodine adsorption test, while the RE is higher in MB adsorption test, 98.4 and 98.1%, respectively. This proves that regeneration of SAC using NaOH and hot water was able to recover more than 90% of micropore of carbon, and more than 98% of adsorption site at macro- and mesopores of the carbon.

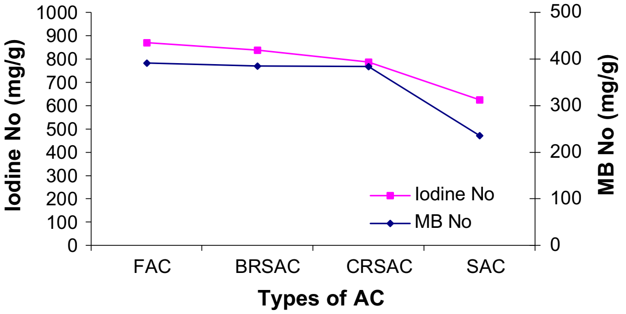

Fig. 5. Iodine no and MB no of FAC, BRSAC, CRSAC, and SAC.

Table 1
The estimated surface area, micropore volume, and total pore volume of FAC, BRSAC, CRASC, and SAC

| Types of carbon | Surface area ($m^2$/g) | Micropore volume ($cm^3$/g) | Total pore volume ($cm^3$/g) |
|---|---|---|---|
| FAC | 936.0 | 0.906 | 0.967 |
| BRSAC | 899.1 | 0.870 | 0.952 |
| CRSAC | 849.0 | 0.852 | 0.945 |
| SAC | 613.7 | 0.327 | 0.647 |

## 4. Conclusion

The optimal regeneration condition for SAC is 15 mL of 6 N of NaOH per g of carbon with 30 min of contact time. The estimated surface area, micropore volume, and total pore volume of BRSAC and CRSAC were 899.1 $m^2$/g, 0.8702 $cm^3$/g, 0.9521 $cm^3$/g; and 849.0 $m^2$/g, 0.8519 $cm^3$/g, 0.9450 $cm^3$/g, respectively. The RE of both BRSAC and CRSAC are more than 90% in iodine adsorption test and 98% in MB adsorption test, which indicates that the adsorption capacity of SAC was restored and is close to those of FAC. This proves NaOH and hot water can regenerate SAC effectively.

## Acknowledgment

The authors wish to express their gratitude to the School of Environmental Engineering, University Malaysia Perlis (UniMAP) for providing the equipments and technical support during this research.

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