Desorption and Re-Adsorption of Procion Red MX-5B Dye on Alumina-Activated Carbon Composite

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ABSTRACT

The alumina-activated carbon has the ability to adsorb and desorb the procion red MX-5B. The research evaluated the influence of desorption agent, contact time, and temperature on desorption process of procion red MX-5B dye with alumina-activated carbon composite and the adsorption capacity of the composite after desorption process. The desorption agents used in desorption process were solution with pH 2−10, H_2O_2 30 % (v/v), methanol 70% (v/v) and ethanol 70% (v/v). The variation of contact time was in the range from 30 to 270 min and the temperature was set between 30−75 °C. The result concluded that the highest desorption efficiency up to 98.56% was achieved using ethanol 70% (v/v) for 240 min at 45 °C. The desorption kinetic followed the pseudo-first-order with the release constant (k_{des}) of 6.56×10^{-2} min^{-1}. The SEM micrograph showed there is a more porous surface on the composite after the desorption compared to before the desorption. The EDX analysis indicated that alumina content in the composite was reduced after desorption process. FTIR spectra of the composite before and after desorption process showed a peak of Al−O at 592 and 590 cm^{-1} which was proved that alumina still exists in the composite after the desorption process. The alumina-activated carbon composite was re-used to adsorb procion red MX-5B dye. After three times of desorption and re-adsorption process, the capacity adsorption was decreased from 12.38 to 7.38 mg/g.

Keywords: *alumina-activated carbon composite; procion red MX-5B dye; desorption, adsorption*

ABSTRAK

Komposit alumina-karbon aktif mempunyai kemampuan mengadsorpsi dan mendesorpsi zat warna procion merah MX-5B. Penelitian ini mempelajari pengaruh reagen pendesorpsi, waktu kontak, dan suhu pada proses desorpsi zat warna procion merah MX-5B dari komposit alumina-karbon aktif dan kemampuan adsorpsi komposit alumina-karbon aktif setelah proses desorpsi. Reagen pendesorpsi yang digunakan pada proses desorpsi meliputi larutan dengan pH pada interval 2–10, H_2O_2 30% (v/v), metanol 70% (v/v) dan etanol 70% (v/v). Variasi waktu kontak pada rentang 30–270 menit dan suhu antara 30–75 °C. Hasil penelitian menunjukkan bahwa efektivitas desorpsi terbesar diperoleh ketika menggunakan etanol pada waktu 240 menit dan temperatur 45 °C yakni sebesar 98,56%. Kinetika desorpsi mengikuti pseudo orde-satu dengan konstanta desorpsi (k_{des}) sebesar $6{,}56 \times 10^{-2}$ min^{-1}. Hasil analisis morfologi menggunakan SEM menunjukkan bahwa pori-pori pada permukaan komposit setelah desorpsi lebih banyak dibandingkan sebelum proses desorpsi. Hasil EDS menunjukkan berkurangnya kandungan alumina pada komposit setelah proses desorpsi. Spektra FTIR komposit sebelum dan setelah desorpsi menunjukkan adanya serapan yang sama yaitu serapan Al-O yang muncul pada bilangan gelombang 592 dan 590 cm^{-1}. Hal ini menunjukkan masih adanya alumina pada komposit setelah proses desorpsi. Komposit alumina-karbon aktif mengalami penurunan daya serap terhadap zat warna procion merah MX-5B setelah proses desorpsi sebanyak 3 kali yaitu dari 12,38 menjadi 7,38 mg/g.

Kata Kunci: *komposit alumina-karbon aktif; zat warna procion merah MX-5B; desorpsi, adsorpsi*

INTRODUCTION

Synthetic dye is often used as coloring agent in the textile industry. The effluent dye discharged into the environment can be a contaminant for surface water and groundwater [1]. Procion red MX-5B dye has a formula of $C_{19}H_{10}Cl_2N_6Na_2O_7S_2$ with molecular weight of 615.33400 g/mol [2]. Several methods have been studied to reduce synthetic dye contained in wastewater such as photocatalytic, chemical oxidation and adsorption [2-4]. Adsorption is considered as the most effective method to reduce dyes contaminated in textile industrial wastewater [5]. Adsorption has several advantages such as relatively low-cost, easy and

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simple to be carried out, and provide high sorption efficiency [6].

The study of desorption is conducted to assess the adsorption process and indicate the adsorption stability [1]. The dye can be released from the adsorbent which have saturated by dye. The adsorbent can be re-used to adsorb the dye. Desorption and regeneration of adsorbent could decrease the processing cost [7-8]. Generally, adsorbent regeneration was conducted using various solvents. The desorption process can be done using organic and inorganic solvents. Commonly used desorption agents in this process are methanol, ethanol, isopropanol, acid and base solution, NaCl, HNO_3, Fenton, and H_2O_2 which depend on the type of adsorbate and adsorbent. Other studies stated that temperature process and contact time also affect the desorption efficiency [9-12].

Activated carbon is the most widely used adsorbent in the adsorption process. The adsorption capacity of activated carbon can be increased by modification of its surface using metal oxide [13]. The metal oxides which can be composited with activated carbon to increase adsorption capacities are Fe_3O_4, $CuFe_2O_4$, $MnFe_2O_4$, SiO_2, TiO_2, and Al_2O_3. Alumina (Al_2O_3) is considered as one of metal oxide with several advantages, i.e., good mechanical property and thermal stability [14]. Alumina is also known to have pore system and active sites on its surface. Alumina was reported by several researchers for having the ability to adsorb orange G, yellow monochlorotriazine, and yellow dichlorotriazinecolor black G [5,15-16]. Surface modification of adsorbent using alumina was expected to increase the adsorption capacity as shown by modification of bentonite-alumina [17], alumina-chitosan [18], and activated carbon-alumina [19-20].

Modified alumina with activated carbon as an alumina-activated carbon composite can be used to adsorb procion red MX-5B dye. The adsorption process followed the pseudo-first order reaction and well fitted to Langmuir isotherm models [19]. In this research, we evaluated the desorption efficiency of alumina-activated carbon composite and the re-adsorption of procion red MX-5B dye after desorption process. In this study, the evaluated parameters of desorption process were various desorption agents, contact time and temperature. The pseudo-first-order kinetic model was applied to describe the kinetic desorption process.

EXPERIMENTAL SECTION

Materials

In this experiment, all chemicals used were of reagent grade and used without further purification. $Al(NO_3)_3$, NaOH, HCl, H_2O_2, and NH_4OH were purchased from Merck whereas procion red MX-5B was obtained from Sigma-Aldrich, distilled water, Whatman filter paper 42. The chemical structure of procion red MX-5B dye presented at Fig. 1.

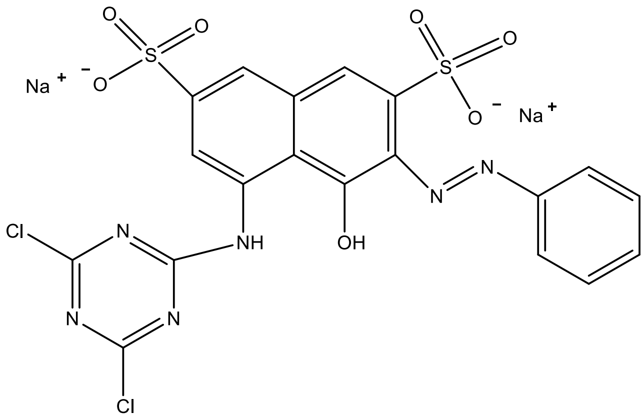

Fig 1. Chemical structure of procion red MX-5B

Instrumentation

The instrument used for the preparation of alumina-activated carbon composite, adsorption and desorption process were oven, hot plate, pH-meter Hanna HI 8424, incubator shaker Stuart SI500, furnace Naber 2804, UV-Visible spectrophotometer Shimadzu mini 1240. Characterization of the composite was analyzed by Scanning Electron Microscopy-Energy Dispersive X-Ray Spectroscopy (SEM-EDX) JEOL JSM 6510 and Fourier Transform Infrared (FTIR) Shimadzu 8400.

Procedure

Preparation of alumina-activated carbon composite

Activated carbon was made from *Gelam* wood. The preparation of activated carbon was following procedure reported in the previous article [19]. Characteristic of activated carbon has surface area 535 m^2/g and pore volume of 0.56 cm^3/g. Synthesis of activated carbon-alumina composite (percentage of alumina is 20%) was conducted by a co-precipitation method using $Al(NO_3)_3$ and NH_4OH as precursors. A solution of 200 mL of 0.1N $Al(NO_3)_3$ was added to 50 g of activated carbon. The mixture was stirred at 120 rpm using a magnetic stirrer while 1 N NH_4OH is added gradually until pH reached ± 10. The reaction between $Al(NO_3)_3$ and NH_4OH produce an amorphous $Al(OH)_3$. Furthermore, the precipitate obtained was washed using distilled water and calcined at 350 °C for 3 h. The calcination process produces alumina (Al_2O_3) from the dehydroxylation of $Al(OH)_3$.

Adsorption experiment

Adsorption was carried out in batch method. In a typical process, 50 mL of procion red MX-5B dye with a concentration of 30 mg/L was added with 0.15 g alumina-activated carbon composite and shook for 2 hours at 150 rpm at room temperature. The suspension was filtered and the filtrate was analyzed using UV-Visible spectrophotometer at 542 nm [17]. The amount of dye adsorbed was following eq.1.

$$q_e = \frac{V}{m}\left(C_o - C_e\right) \quad (1)$$

where q_e, C_o, and C_e are the amount of procion red MX-5B dye adsorbed onto composite (mg/g), the initial concentration of dye (mg/L), concentration of dye at equilibrium (mg/L), respectively. Besides that, V and m are the volume of the solution (L) and the weight of the composite (g). The study was carried out by the following process: alumina-activated carbon composite adsorption, desorption process and re-adsorption. Eq. 2 describes the amounts of dye remaining on the adsorbent as a function of time.

$$q_t = q_e - C_t\left(V/m\right) \quad (2)$$

where q_t is the amount of dye remaining on the absorbent (mg/g) and C_t is the concentration of dye (mg/L) in the solution.

Desorption and re-adsorption process

The alumina-activated carbon composite which has adsorbed procion red MX-5B was desorbed by several desorption agents. Desorption process using 0.1 g of adsorbent added by 10 mL solution with pH in the range from 2−10 (adjusted by adding HCl and NaOH 1 M), methanol 70% (v/v), ethanol 70% (v/v), and H_2O_2 30% (v/v). The mixture was stirred using a shaker for 3 h at room temperature. Then, the suspension was filtered. The concentration of filtrate was carried out using UV-Visible spectrophotometer at 542 nm. The procedure of desorption was also performed for various contact time (30–240 min) and temperature (30, 45, 60, and 75 °C). Morphology analysis and elemental content of the composite before and after desorption were analyzed using SEM-EDX and the functional group was recorded by FTIR.

The kinetics of desorption study was obtained by pseudo first-order reaction [22-23]. The solvent used for desorption of procion red MX-5B dye is the best solvent. Composite used for all process were re-used in the re-adsorption process following the same procedure of adsorption [19]. Desorption and re-adsorption process was repeated three times to measure the desorption efficiency.

RESULT AND DISCUSSION

Effect of Desorption Agents

Desorption of procion red MX-5B dye from alumina-activated carbon composite using solution at a various pH of 2−10, methanol, ethanol, and H_2O_2 are shown in Table 1. The higher pH of the solution shows more efficiency in desorption of procion red MX-5B dye. The surface properties of alumina and the functional groups on the adsorbent are affected by pH solution [12,15,24]. The alumina has a point of zero charge (pH_{pzc}) of 7.4. In solution with pH > pH_{pzc}, alumina would have negative charge [11,15,25]. Thus, in alkaline solution, there is electrostatic repulsion between anion of dye with the negatively charged of the surface of alumina [15]. In this study, the effect of pH to increase the desorption efficiency was insignificant. The desorption efficiency at pH 2 was 1.28% while at pH 10 was 3.01%.

Desorption using H_2O_2 30% solution gives the desorption efficiency of 1.24%. The desorption mechanism using H_2O_2 30% solution is oxidation reaction [26]. H_2O_2 released free radical of hydroxyl which will reacted with a dye compound [27]. The catalyst, such as metal ions, is needed to initiate the process. In this study, desorption process did not use catalyst that caused the slow rate of free radical

Table 1. Desorption of procion red MX-5B dye from alumina-activated carbon composite

Desorption agent	Conc. desorbed (mg L^{-1})	Desorption efficiency (%)
pH 2	0.327	1.28
pH 3	0.308	1.30
pH 4	0.327	1.34
pH 5	0.346	1.42
pH 6	0.404	1.64
pH 7	0.500	2.08
pH 8	0.635	2.45
pH 9	0.769	3.07
pH 10	0.750	3.01
Ethanol 70%	22.404	92.31
Methanol 70%	16.827	73.10
H_2O_2 30%	0.327	1.24

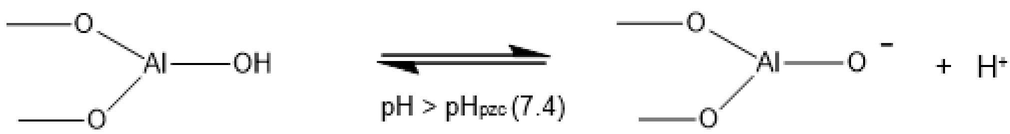

Fig 2. Structure of alumina at pH solution > pH_{pzc}

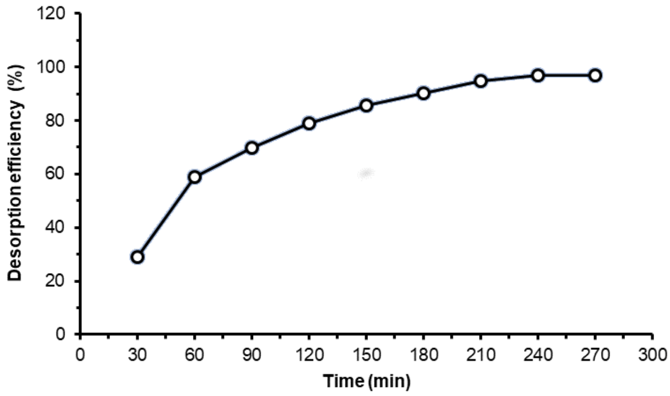

Fig 3. Effect of contact time to procion red MX-B58 dyes desorption from alumina-activated carbon composite (weight of adsorbent 0.1 g, pH solution of 6.3, volume of ethanol 10 mL at room temperature ± 30 °C)

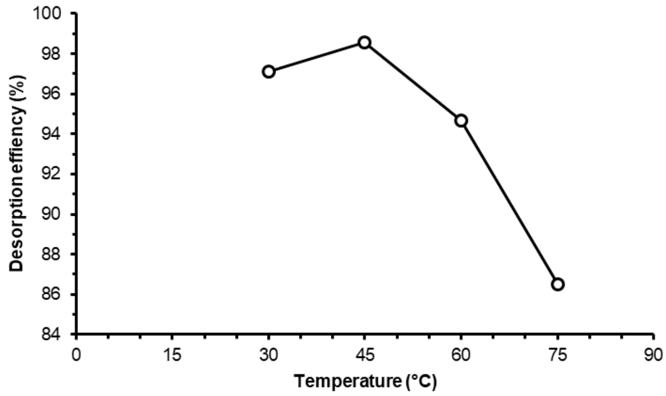

Fig 4. Effect of temperature to procion red MX-B58 dye desorption from alumina-activated carbon composite (weight of adsorbent 0.1 g, pH solution of 6.3, volume of ethanol 10 mL and contact time 240 min)

formation. Another reference shows H_2O_2 cannot be used as a solvent for desorption of remazol yellow dye from zeolite [24].

Desorption is affected by the availability of hydroxyl group, molecular weight, and desorbing agent hydrophobicity. The dye is more soluble in the alcohol than water due to the forming of hydrogen bond between hydroxyl groups in alcohol with the dye. The organic solvent has higher desorption efficiency than inorganic solvent [11]. The data are given in Table 1, indicates that alcohol gives higher desorption efficiency than the other solvents. Methanol and ethanol have one hydroxyl group with partition coefficient (K_{ow}) of methanol and ethanol are 0.15 and 0.48, respectively. A solvent with high K_{ow} (K_{ow} > 1) is more hydrophobic. The dyes compound will compete with hydrophobic groups of ethanol or methanol to be bonded to the adsorbent. If hydrophobicity of solvent is high, it will attach procion red MX-5B dye more strongly [9,11].

In this study, the desorption efficiency of methanol and ethanol are 73.10 and 92.31%, respectively (Table 1). The desorption efficiency is also influenced by the molecular weight [11]. Ethanol has a greater molecular weight than methanol. Thus methanol is more polar than ethanol. The dye itself is classified into organic compounds which have polar and non polar groups. However, the dye tends to be non-polar, so the dye is more soluble in ethanol than methanol.

Effect of Contact Time to Desorption Process

The procion red dye MX-5B desorption from alumina-activated composite was conducted using ethanol at various contact time in the range of 30–240 min. The desorption of dye at different time intervals in order to get the equilibrium time was achieved. The effect of contact time on the amount of dyes desorbed at room temperature (± 30 °C) and pH of solution 6.3 is shown in Fig. 3. From the figure obtained the longer contact time increased the amount of dye released from the adsorbent until an equilibrium is reached. The dye cannot be released completely. Therefore, there are still some dye which strongly bonded to the adsorbent. It is clear that the contact time of 240 min gave the highest desorption efficiency of 97.07%.

Effect of Temperature of Desorption

The effect of temperature on the desorption process is illustrated in Fig. 4. The Desorption used ethanol as a solvent, pH solution 6.3 and contact time of 240 min. The temperature of desorption was set up at 30, 45, 60, and 75 °C. The figure indicated that optimum temperature for desorption of procion red MX-5B dye from alumina-activated carbon composite is 45 °C with desorption efficiency of 98.56% and decreased at 60 and 75 °C. The decreasing of desorption is caused by evaporation of ethanol, hence reduce its ability to dissolve dyes from alumina-activated carbon composite.

Ethanol has a boiling point at 78.37 °C. The composition of ethanol and water in a solvent influences the boiling point. Each dye compounds had different optimum temperature of desorption, i.e. methylene blue was desorbed optimally by ethanol at 25 °C [28], reactive blue BF-5G through optimal desorption at 30 °C using isopropyl alcohol [11], reactive red gave optimum desorption from carbon nanotubes at 48 °C using NaOH [6], the yellow and red dyes were desorbed optimally from activated carbon using alcohol 20% at 40 and 50 °C [12].

Kinetic of Desorption

The kinetic study is provided information mechanism on the adsorption process. Kinetic desorption In the present study expressed by a pseudo-first order rate equation as represented as eq. 3 [12,26].

$$\frac{q_t}{q_0} = \exp(-k_{des}t) \quad (3)$$

where q_t and q_0 are the concentration of procion red MX-5B dye in the composite (mg/g) at time t and the initial total procion red MX-5B dye in the composite (mg/g). k_{des} is a rate constant (min^{-1}) of pseudo first order desorption. The linear form of the pseudo first order kinetic desorption as given in eq. 4 [26].

$$\ln\frac{q_t}{q_0} = \ln\theta - k_{des}t + (1-\theta) \quad (4)$$

with θ as the value of desorbable fraction. Plot of ln q_t/q_0 and t are used calculated of k_{des} and θ. The pseudo first order kinetic desorption of procion red MX-5B dye using ethanol is represented in Fig. 5.

The kinetic study of desorption of procion red MX-5B dye was fitted to the pseudo first-order with the highest R^2 value of 0.9855. The value of k_{des} and θ are $6.56.10^{-2}$ min^{-1} and 1.152, respectively. Desorption process is influenced by the strong interaction between adsorbent and dye molecules, such as van der Waals forces or electrostatic attraction. The adsorption is indicated as chemisorption process if the desorption is irreversible [11].

Composite Morphology Before and After Desorption

Fig. 6 (a) and (b) display characterization result of alumina-activated carbon composite using SEM with 10,000x magnification. The morphology shows that the surface of alumina-activated carbon composite before desorption is denser and heterogeneous compared to after desorption. It indicates the surface of alumina-activated carbon composite become saturated by adsorbing procion red MX-5B dyes.

Formation of pore in the surface of alumina-activated carbon composite after desorption process confirmed that procion red MX-5B dye was successfully removed. EDX analysis of alumina-activated carbon composite was conducted to determine atomic composition contained within adsorbent which presented in Table 2. The decreased of Al content in the composite after desorption process is obvious along with O content. It appeared that alumina was also partly removed through desorption process as well as the procion red MX-5B dye.

FTIR spectra of alumina-activated carbon composite before and after desorption are shown on Fig. 7. The wavenumber in range of 557.0−748.3 cm^{-1} indicates Al−O bond [29]. This FTIR spectra of composite before desorption investigated at 592.1 cm^{-1}, whereas after desorption appears at 590.2 cm^{-1}.

Table 2. Elemental data of alumina-activated carbon composite before and after desorption

Element	Before desorption (%)	After desorption (%)
C	48.50	64.20
O	31.42	22.50
Al	15.56	10.23
Si	4.52	3.02
Au	-	0.05

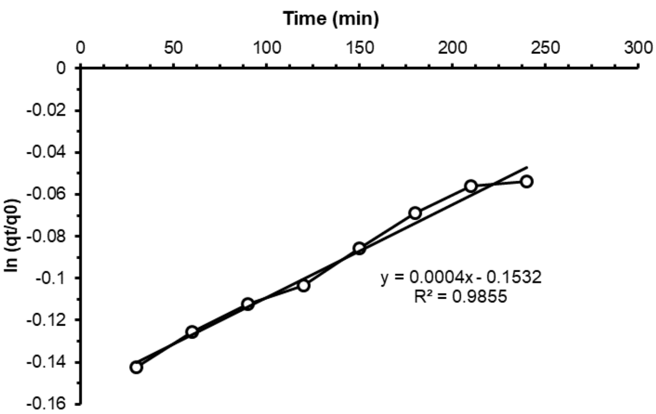

Fig 5. Pseudo first-order desorption kinetic

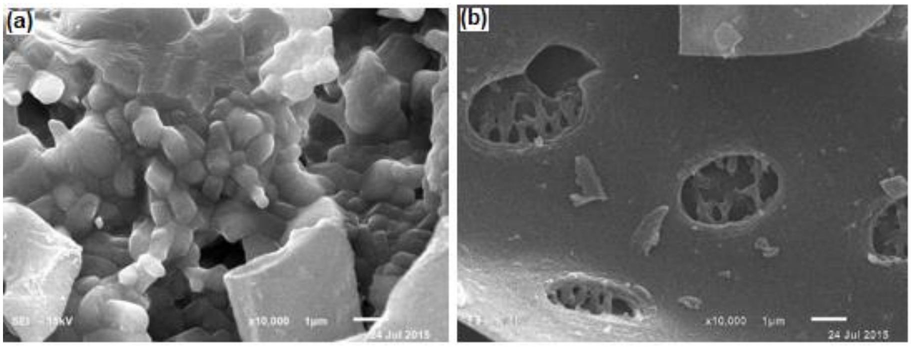

Fig 6. Morphology of alumina-activated carbon composite before desorption (a) and after desorption (b)

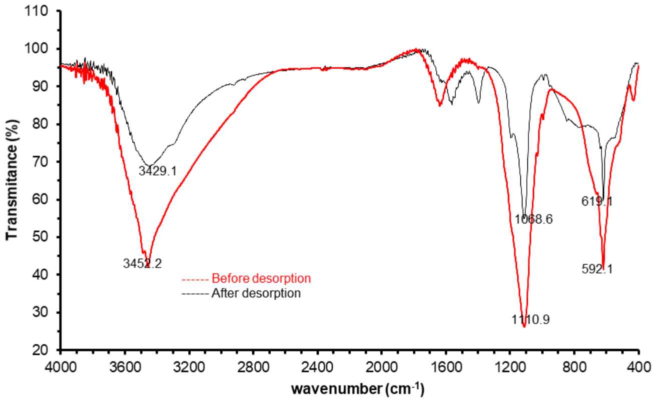

Fig 7. FTIR spectra of alumina-activated carbon composite before and after desorption

Table 3. Adsorption capacity of alumina-activated carbon composite after desorption process

cycle	Adsorption capacity (mg/g)	Desorption efficiency (%)
1	12.38	98.56
2	8.17	85.23
3	7.38	73.46

A sharp peak at 1110.9 and 1066.6 cm^{-1} indicated the C−O bond of alumina-activated carbon composite before and after desorption, respectively. Peaks at 3452.2 and 3429.1 cm^{-1} are assigned for H−O−H stretching. Generally, the FTIR spectra of the composite before and after desorption reveals the same pattern of wavenumber. Change in intensity of composite may be related to the desorption process [11]. According to the data, we can conclude that there is no structural change of composite due to desorption process.

Re-Adsorption Process of Procion Red MX-5B Using Alumina-Activated Carbon Composite

Alumina-activated carbon composite was re-used to adsorb red procion MX-5B dye in the same process of adsorption [19]. Desorption was conducted after adsorption using ethanol 70% at 45 °C for 240 min. The adsorption-desorption process was carried out for three cycles and calculated of composite adsorption capacity and desorption efficiency are shown in Table 3.

The repeated desorption process on the composite decreased the adsorption capacity of alumina-activated carbon composite. The decrease of adsorption capacity is depending on the functional groups in the adsorbent. Several dyes cannot be separated from the adsorbent, therefore block the pore of adsorbent [30]. From the EDX analysis, the amount of alumina is reduced after the desorption process, so the active sites in the composite are also reduced. The occupied of activated carbon surface sites by solvent molecules leading to the decreasing of adsorption capacity [9].

CONCLUSION

The alumina-activated carbon can be used as an adsorbent to remove procion red MX-5B dye in solution. The procion red MX-5B dye can be released from adsorbent and the adsorbent can be re-used for adsorption. In this study, the effective solvent for desorption of procion red MX-5B dye from alumina-activated carbon was ethanol. The highest desorption efficiency was obtained at 45 °C for 240 min. The desorption kinetic model followed the pseudo first-order. The desorption efficiency of alumina-activated carbon after three cycles of desorption and re-used for adsorption process decreased as calculated from 98.56 to 73.46%. Thus the alumina-activated carbon composite can be promising as an adsorbent to remove the pollutants, especially dyes.

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