Vol. 20, No. 3(2021) Mat2444
Revista Mexicana de Ingeniería Química
Materiales

High removal of toxic crystal violet dye using a thermally treated activated carbon fiber felt

Alta adsorción del colorante tóxico cristal violeta empleando una fibra de fieltro de carbón activado tratada térmicamente

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Received: April 23, 2021; Accepted: July 1, 2021

Abstract

In the present work, the reuse of activated carbon fiber felt (ACFF) is suggested to remove the cationic methyl violet, commonly called as Crystal Violet (CV), dye present in a synthetic solution. The morphological structure of the ACFF was analyzed by High Resolution Scanning Electron Microscopy (HRSEM). The dye removal processes were carried out in batch experiments at room temperature and pH of 10, by reusing the FFCA up to 10 times. After each removal process, the ACFF was thermally treated by calcination that allowed to transform the adsorbed dye into CO and CO_2 wherewith its useful lifetime was extended. The results show that the adsorption process follows the Langmuir isotherm with pseudo-first order kinetics, which suggests that a chemisorption was carried out by the covalent attraction between the carbonyl groups of the ACFF and the cationic species of the dye. The novelty of the work focuses on the reuse of the ACFF for up to 10 removal cycles with a high removal rate, which can lead to a reduction in the acquisition costs of the adsorbent material.

Keywords: activated carbon fiber felt, reusing, calcination, removal, crystal violet.

Resumen

En el presente trabajo se sugiere la reutilización del fieltro de fibra de carbón activado (FFCA) para la remoción del colorante catiónico violeta de metilo, también llamado cristal violeta (CV), presente en una solución sintética. La estructura morfológica de la FFCA se analizó mediante Microscopía Electrónica de Barrido de Alta Resolución (MEBAR). Los procesos de remoción del colorante se realizaron de manera discontinua a temperatura ambiente y pH de 10, mediante el reúso de la FFCA hasta 10 veces. Posterior a cada proceso de remoción, la FFCA se sometió a un tratamiento térmico (calcinación) que permitió convertir el colorante adsorbido en CO y CO_2 y extender el tiempo de vida útil de la FFCA. Los resultados muestran que el proceso de adsorción sigue la isoterma de Langmuir con cinética de pseudo primer orden, lo cual sugiere que se llevó a cabo una quimisorción por la atracción covalente entre los grupos carbonilo de la FFCA y la especie catiónica del colorante. La novedad del trabajo se centra en la reutilización de la FFCA hasta por 10 ciclos de remoción con alto porcentaje de remoción, lo cual puede llevar a una disminución en los costos de adquisición del material adsorbente.

Palabras clave: fieltro de fibra de carbón activado, reúso, calcinación, remoción, cristal violeta..

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https://doi.org/10.24275/rmiq/Mat2444
ISSN:1665-2738, *issn-e*: 2395-8472

1 Introduction

Emerging contaminants (ECs) are different compounds have been discharged by households and industries and they are generated due to the increase in common use objects, such as, pharmaceuticals and personal care products, plasticizers, food additives, wood preservatives, laundry detergents, surfactants, disinfectants, flame retardants, pesticides, natural and synthetic hormones, etc. (Sophia and Lima, 2018). They released continuously into the environment with a worried increasing rate and the potential hazards and impacts of these ECs on the ecosystem and human health have been gaining attention. Azo dyes are considerate emerging contaminants (Taheran *et al*., 2018; Ji *et al*., 2020). Different sources of contamination by dyes had been found in last years, however, the most polluting industry is the textile with colored discharges caused by azo dyes that represent near of the 70% of the dyestuffs used (Sen *et al*., 2016).

Methylene violet, commonly called as Crystal Violet (CV), is an azo dye that causes health problems in humans in contact with it, due to its corrosively and toxicity, the most common problems are severe damage to eyes, inhalation difficulty and skin irritation (Lu *et al*., 2020). However, CV has been used in many applications, particularly in textiles, paints and print inks (Nooraee Nia *et al*., 2017), medical applications where it is used as active ingredient in bacteria classification (Ehyaee *et al*., 2017). In chemistry as indicator in many reactions (Pak *et al*., 2017). It is used in food industry or cosmetics for coloring as pigment and dye (Horakova *et al*., 2016). Due to the low biodegradability property of synthetic dyes as CV, the incorporation of them in ecosystems cause negative ecological impact, mostly in the aquatic life, as the dye inhibits the passage of sunlight trough the water hindering the photosynthesis process (Mittal *et al*., 2020). It has been reported that CV concentration into wastewater is from 50-70 ppm which causes several damages to aquatic life (Morales-Álvarez *et al*., 2016).

Many approaches have been used to achieve the elimination of dyes from water bodies, among which the following stand out: anodization and electrocoagulation (El-Ashtoukhy *et al*., 2017; Singh *et al*., 2016), micro remediation (Lira-Pérez J *et al*., 2019), microwave (Garzón-Pérez *et al*., 2020), ultrasonic, nanofiltration with modified membranes (Febrianto *et al*., 2019; Mittal *et al*., 2020; Febrianto *et al*., 2019; Gunawan *et al*., 2019; Sutedja *et al*., 2017), ultraviolet (Ayala *et al*., 2017), ozonation (Mendoza-Basilio *et al*., 2017), sedimentation (Solís *et al*., 2013), electro incineration, chemical coagulation, enzymatic (Solís-Oba *et al*. 2009), and different types of catalysis as: conventional catalytic processes (Raman and Kanmani, 2016), heterogeneous photocatalysis (Márquez-Ramírez *et al*., 2019; Lakshmi Prasanna and Rajagopalan, 2016), adsorption in batch (González-López et. al, 2021), piezocatalysis (Singh *et al*., 2020), remediation by nano zero-valent irons (Handojo *et al*., 2020) or a combination of the approaches. All they had been widely studied, but most of them are expensive or difficult to be reproduced in some labs. Adsorption approach has been used due to its simplicity and efficiency.

To reduce the dye of water systems, similar adsorption studies have been realized using different based carbon materials as adsorbent because it is one of the more eco-friendly and efficient materials for this purpose, between them: functionalized multi-walled carbon nanotubes (Sabna *et al*., 2016), organic powders obtained from natural sources (Lairini *et al*., 2017; Rahimian and Zarinabadi, 2020 ; Romero Cano *et al*., 2016; Gonzalez *et al*., 2013), or modified chitosan (Verduzco-Navarro *et al*., 2020); however, all the mentioned materials have the inconveniency that they must to be separated after each use or employ packed bed reactors which could causes pressure drops. For this reason, some research are focused in the using of pristine or modified carbon fiber felt, as the absorption of organic compounds from aqueous solutions (El-Shafey *et al*., 2017; Tang *et al*., 2007; Liu *et al*., 2019) where the studies were focused in the measurements of their properties but did not further explore the reusability as some researchers do it (Trellu *et al*., 2018; Rabbi *et al*., 2020; Acevedo-García *et al*., 2020; Pourali *et al*., 2021), included this group in a previous work (Rojas-Valencia *et al*.2020).

In this work, we suggest reuse of ACFF for high removal of toxic Crystal Violet dye. A thermal treatment of ACFF was carried out after every adsorption process for carbonize the removed dye and extend the half-life of adsorbent material. The % adsorption was determined until ten adsorption processes. The novelty of this work is that reusing of ACFF could make cheaper the removal process of dyes.

2 Experimental and methods

2.1 Materials

Cationic dye crystal violet (CV) ($C_{24}H_{28}N_3Cl$), sodium hydroxide (NaOH, 98%), hydrochloric acid (HCl, 37%) and sodium chloride (NaCl, 98%) were purchased from Sigma Aldrich. Polyacrylonitrile (PAN) Activated Carbon Fiber Felt (ACFF) was purchased from KoThmex with the following features: fiber diameter of 6-11 (μm), surface area of 700-2000 (m^2 g^{-1}), micropore diameter of 0.4-1.0 (A), micropore volume of 1.5-2.0 (mL g^{-1}). Polyacrylonitrile ACFF was chosen because his resistance to high temperatures and low cost.

2.2 Thermal treatment of activated carbon fiber felt (ACFF)

For thermal treatment of ACFF at different temperatures and times, and the point zero charge determination, was carried out from a previously published method by our work team (Rojas-Valencia *et al*., 2020).

2.3 Characterization

The ACFF was analyzed by a high-resolution scanning electron microscope (HRSEM, JEOL 6701F) operated at 5.0 kV for surface morphological structure observation. The initial and residual concentration of CV was determined at different removal times, with a double beam UV/Vis spectrophotometer (PerkinElmer-Lambda 365) using a (1.0 cm) quartz cell at 585 nm.

2.4 Adsorption experiments

An aqueous solution was prepared by dissolving 70 mg of CV powder in 1L of distilled water with an adjusted pH by 0.1 M NaOH and 0.1 M HCl solutions. The experiments were carried out by mixing a piece 200 mg of ACFF into 40 mL of solution at 27°C. Residual dye concentration was analyzed by UV-Vis spectrophotometer at 5, 10, 15, 20, 30, 40, 50 and 60 minutes, through calibration curve with different initial concentrations of CV. The percent adsorption was calculated by using the Eq. (1)

$$\%Adsorption = \left(\frac{C_i - C_t}{C_i}\right) \times 100 \tag{1}$$

Where C_i is the initial dye concentration (mg/L), C_t is the residual concentration at different time intervals (mg L^{-1}). The adsorbed CV amount at time t, per g of ACFF, Q_t (mg g^{-1}), was calculated using the Eq. (2)

$$Q_t = \left(\frac{C_i - C_t}{C_i}\right) \times V \tag{2}$$

Where m is the mass of ACFF (g), and V is the volume of the solution (L).

3 Results and discussion

3.1 HRSEM analysis

Activated carbon fiber felt was characterized by HRSEM for surface morphological structure analysis. Fig. 1 shows that ACFF is made up of several randomly arranged filaments with a clean surface, free of atmospheric dust, and organic impurities; therefore, there it is a complete surface area for dye removal.

3.2 Adsorption analysis

The removal of CV from aqueous solution was carried out at different times (5 min to 60 min). Fig. 2 shows that new ACFF removes 85% from CV at 5 minutes; while at 10 minutes, adsorption is near complete with 99.25% removal. However, the removal efficiency decreases with ACFF reuses. At 5 minutes, reusing ACFF from four to eight times, 42.5 % was removed; however, when it is reused for the tenth times, the removal percentage decreases to 28% at the same removal time.

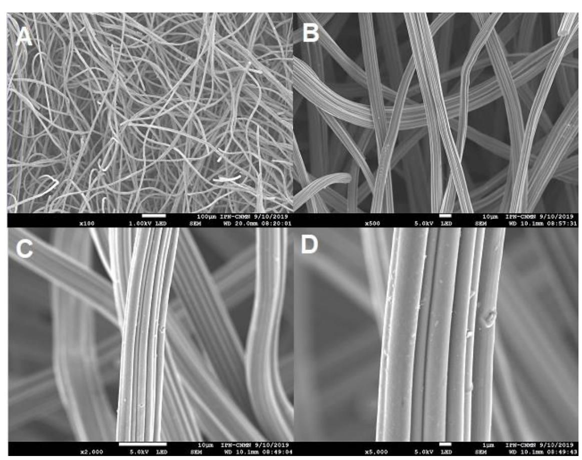

Fig. 1. HRSEM images of ACFF filaments at A (300 X), B (500 X), C (2000 X) and D (5000 X).

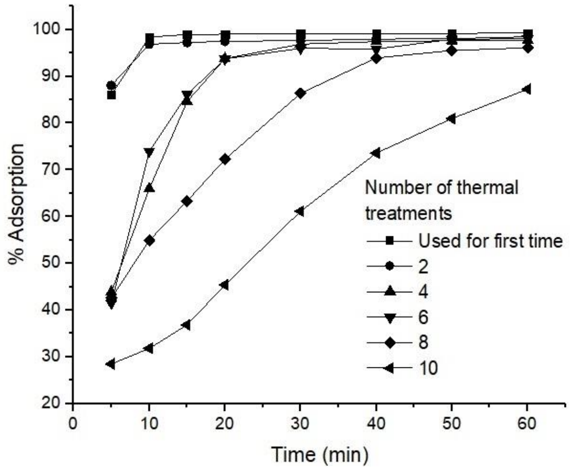

Fig. 2. Percentage dye removal for new and reused ACFF at different times.

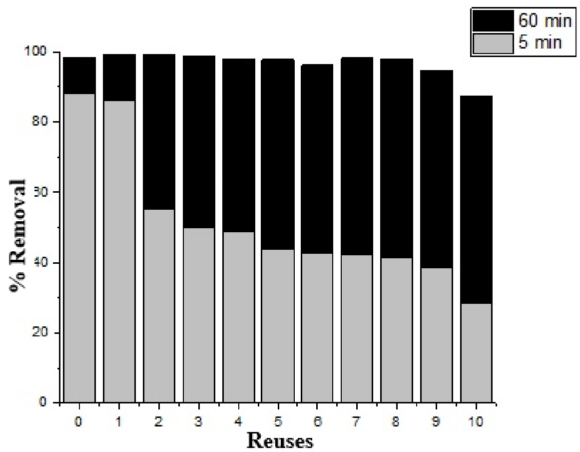

Fig. 3. Relation between ACFF reuses with removal performance.

At 30 minutes, a difference can be observed in removal efficiency between ACFF reused for fourth and sixth time where the removals are 99%. It is evident that for the tenth time reuse, the percentage removal, at 60 minutes, is 85 %.

Fig. 3 shows the relation between ACFF reuses with remotion percent. It is appreciated that the new ACFF and with one reuse adsorb near 90% at 5 minutes, whilst at 60 minutes they can adsorb near 100 %, however, adsorption percentage diminishes with ten ACFF reuses until 28.5 % at 5 minutes and near 87.5% at 60 minutes.

Adsorption occurs when the intermolecular attractive forces between the molecules of adsorbate (CV) and the adsorbent (ACFF) are higher than those among adsorbate molecules themselves (Gong *et al.*, 2013). After using ACFF for the first time, it was treated by a thermal method (calcination), where adsorbed CV is turned to CO, CO_2 (EC, 2007), and carbon deposits are created too; this means that, during ACFF reusing at the beginning of removal process, there is less surface area, and therefore, the adsorbed CV amount decreases, however, after a few minutes and as consequent of stirring, during removal process, the carbon deposits could be desorbed to the aqueous solution, thereby, more CV molecules can be adsorbed.

3.3 Adsorption isotherms

Adsorption isotherms are the graphic representation between adsorbate mass and adsorbent mass in a solution at equilibrium pH and constant temperature. The adsorption data of CV on ACFF were analyzed Freundlich logarithmic and Langmuir model. The first one is a mathematical expression for adsorption equilibrium between a liquid and a solid material (Van Der Bruggen, 2020). Freundlich model is described in Eq. (3)

$$\log(Q_e) = \log k_F + \frac{1}{n} \log C_e \tag{3}$$

Where k_F is the adsorption capacity (L mg^{-1}) and $(1/n)$ is the heterogeneity factor, which indicates the magnitude of the adsorption driving force or the surface heterogeneity. Langmuir isotherm is a model of the absolute absorption based on monolayer filling of non-interacting molecules; the linearized Langmuir model is described in Eq. (4).

$$\frac{C_e}{Q_e} = \frac{1}{Q_{\max} k_L} + \left(\frac{1}{Q_{\max}}\right) C_e \tag{4}$$

Where C_e and Q_e are the same parameters described above, $Q_{\max}$ (mg g^{-1}) is the maximum saturated monolayer adsorption capacity and k_L (mg g^{-1}) is a constant related to the affinity between CV and ACFF.

Fig. 4 shows the Langmuir and Freundlich isotherms for ACFF. The correlation coefficient (R^2) determines the fitting quality. It can be shown that Langmuir isotherm for ACFF used for first time (A) describes the adsorption process more accurately than obtained at ten reuses (C) and Freundlich isotherms (B) and (D), this can be confirmed because, their correlation coefficient is near to one. The Langmuir constant indicates the extent of interaction between CV and the ACFF surface. If the value of k_L is relatively larger it indicates that there is a strong

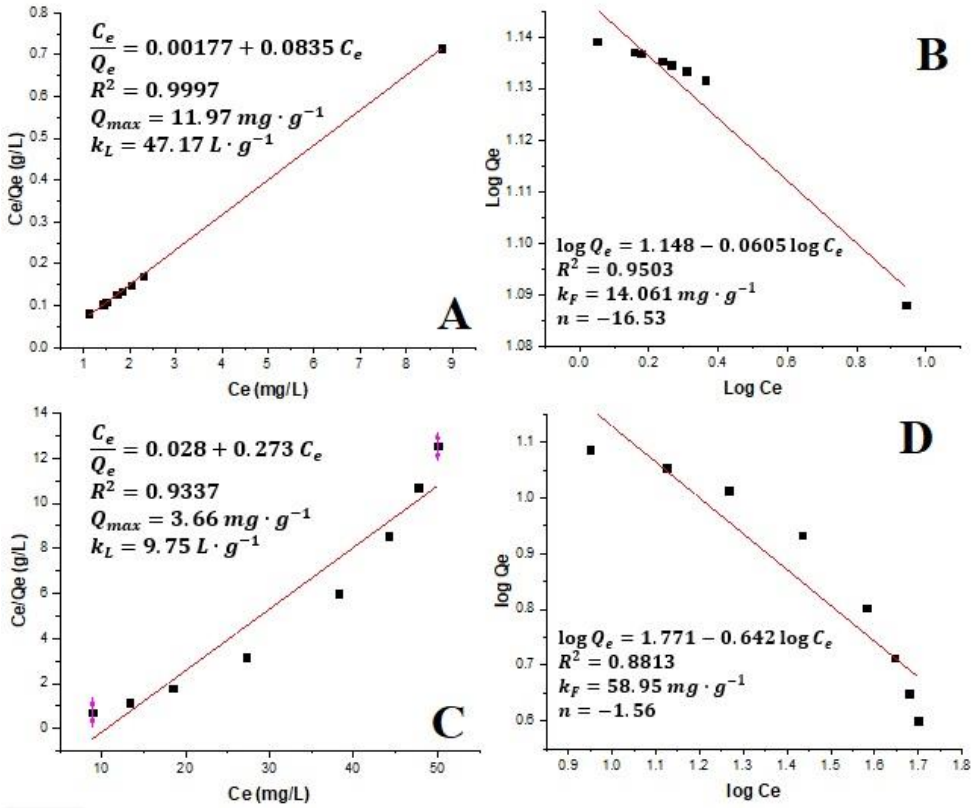

Fig. 4. Langmuir isotherm for ACFF used for first time (A) and ten times (C). Freundlich isotherm for ACFF used for first time (B) and for ten times (D).

interaction between adsorbate and adsorbent while smaller value implies a weak interaction, it means that when ACFF is used for the first time the maximum adsorption capacity ($Q_{\max}$) value for CV removal at 27°C, is 11.97 mg g^{-1}, whilst, for ACFF reused ten times $Q_{\max}$ is 3.66 mg g^{-1} which is directly related with Langmuir constant. Freundlich constant n<1 indicates that adsorption was a chemical process.

With the information provided by Langmuir isotherms, a separation factor (R_L) was determined with Eq. (5)

$$R_L = \frac{1}{1 + k_L C_0} \tag{5}$$

Where k_L is Langmuir constant and C_0 is the initial concentration of CV (mg L^{-1}). If $R_L > 1$ the adsorption is not favorable; if $R_L = 1$ the process is linear in nature; if $R_L < 1$ the process is favorable; if $R_L = 0$ is an irreversible process (Singh *et al.*, 2019). Table 1 shows Langmuir and Freundlich isotherms for all reuses of CFF and according to the R^2 coefficient and R_L factor, during removal process the adsorbed CV amount per ACFF unit mass and remaining CV concentration at 27 °C, under equilibrium conditions, is best described by Langmuir model. According with Table 1, the dimensionless separation factor values, from the Langmuir model, were $R_L < 1$, whereby adsorption processes carried out were favorable.

3.4 Adsorption kinetics

Kinetics describe the adsorption rate of CV in ACFF and determine the time in which equilibrium is reached. In this research, pseudo-first order and pseudo-second order were applied to experimental results. The pseudo-first-order model is described in Eq. (6) and pseudo-second order in Eq. (7)

$$\ln(Q_e - Q_t) = \ln Q_e - k_1 t \tag{6}$$

$$\frac{t}{Q_t} = \frac{1}{k_2 Q_e^2} + \left(\frac{1}{Q_e}\right) t \tag{7}$$

Table 1. Constants related to isotherms.

		Langmuir isotherm			Freundlich isotherm		
Reuses	R_L	R^2	Q_{max} (mg g^{-1})	k_L (L g^{-1})	R^2	k_F (mg g^{-1})	**n**
0	0.00031	0.999	11.97	47.17	0.95	14.06	-16.52
1	0.00025	0.999	11.83	45.91	0.961	13.58	-15.87
2	0.0005	0.979	10.78	28.15	0.95	17.36	-15.11
3	0.00098	0.977	7.092	24.53	0.79	19.16	-14.83
4	0.00093	0.955	6.894	19.23	0.787	23.36	-13.99
5	0.00076	0.947	6.086	18.58	0.781	25.87	-10.61
6	0.00075	0.929	6.035	18.23	0.819	36.25	-9.65
7	0.00073	0.955	5.595	18.03	0.741	39.17	-6.57
8	0.00073	0.995	5.489	17.77	0.707	41.21	-4.41
9	0.00072	0.945	5.412	11.67	0.845	42.21	-2.82
10	0.00146	0.933	3.663	9.75	0.881	58.95	-1.56

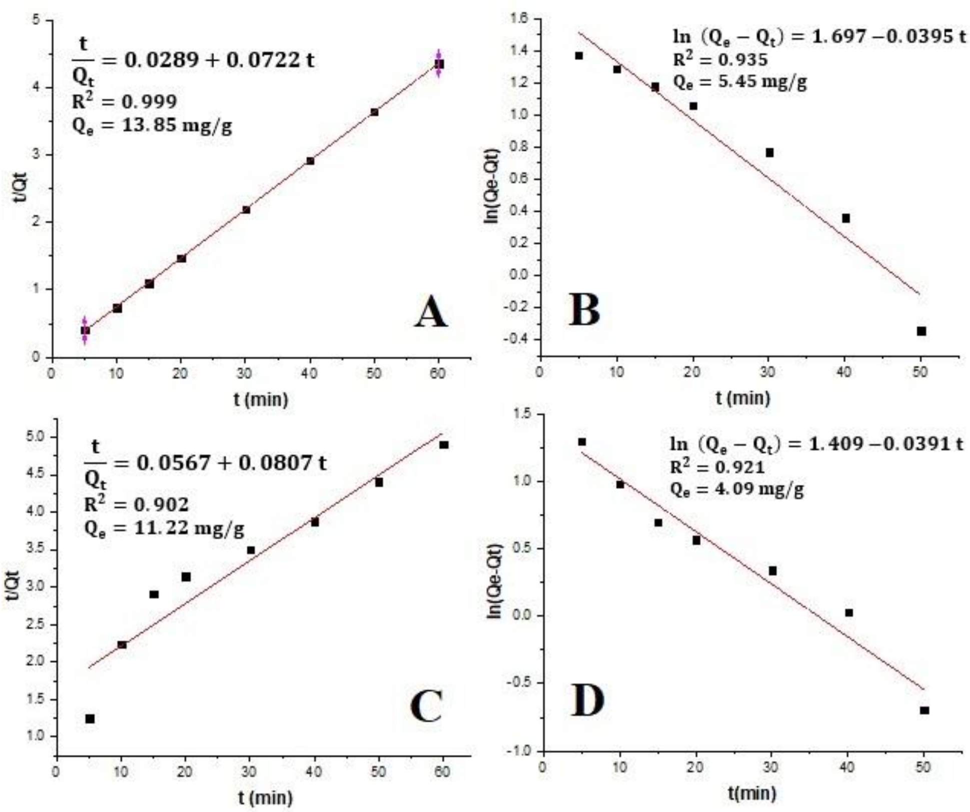

Fig. 5. Pseudo first-order kinetics model for ACFF used for first time (A) and ten times (C). Pseudo pseudo-second order (B) for ACFF used for first time and ten times (D).

Where, for Eq. (7), Q_e is the amount of CV adsorbed at equilibrium (mg g^{-1}), Q_t is the amount of CV adsorbed at time t (mg g^{-1}), k_1 (min^{-1}) and k_2 (g mg^{-1} min^{-1}) are the rates of pseudo-first and second order (Ibupoto *et al*., 2018). Fig. 5 shows pseudo-first order kinetics model (A) and pseudo-second order (B) for CV adsorption onto ACFF used for the first time and, pseudo-first order (C) and pseudo-second order (D) for ACFF reused ten times. It is clear from R^2 values shown that the pseudo-first-order kinetics model best describes the adsorption of CV on ACFF for first or for the tenth time, and it can be supported with the Q_e calculated, which is like the experimental Q_e. The pseudo-first-order kinetic model suggests chemical interactions between CV and ACFF by the presence of carbonyl groups present on the surface of ACFF and cationic species of CV (Rojas-Valencia *et al*., 2020).

Conclusions

The current study suggests reuse of thermally treated activated carbon fiber felt for removal of toxic crystal violet dye. Calcination step after every removal process allows thermal degradation of crystal violet dye to CO and CO_2 gases, which can be partially desorbed by magnetic stirring used in removal processes. Adsorption capacity of new ACFF is reduced from 11.97 mg g^{-1} to 3.66 mg g^{-1} at tenth reuse process. It can be attributed to the decrease in the surface are from ACFF because of partial desorption of crystal violet dye turned to CO and CO_2, whereby removal percentage decrease to 85 % at 60 minutes with tenth reuse. The reuse of ACFF after a thermal treatment allows to extend its useful lifetime and reduction in acquisition of adsorbent material costs.

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