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Effect of thermal treatment of activated carbon fiber felt for reuse in removal of methylene blue from a synthetic wastewater

Efecto del tratamiento térmico del fieltro de fibra de carbón activado para su reutilización en la eliminación de azul de metileno de un agua residual sintética

O.G. Rojas-Valencia[1,2]*, M. Estrada-Flores[1], C.M. Reza-San-Germán[1], E. Torres-Santillán[1], J. Hernández-Fuentes[1], J.L. Ledezma-Martínez[1]

[1] *Instituto Politécnico Nacional, ESIQIE, UPALM, Edificio Z5 segundo piso. San Pedro Zacatenco, Alcaldía Gustavo A. Madero, Ciudad de México, México.*
[2] *Universidad Tecnológica de México - UNITEC México - Campus Atizapán.*

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Abstract
This work proposes reusing of acrylonitrile activated carbon fiber felt (ACFF) for the removal (adsorption) of a cationic dye (methylene blue, MB) from synthetic wastewater. After every removal process, ACFF was thermally treated (calcinated) for reuse. The surface morphology was characterized by high-resolution scanning electron microscopy (HRSEM), and Fourier transform infrared spectroscopy (FTIR) analysis helped to identify functional groups that allowed adsorption process. Batch experiments were carried out at 27 °C with a pH of 10. Results revealed that after calcination, the MB adsorbed turned to carbon deposits, which desorbed during others removal processes, this allowed reusing of ACFF for several adsorption cycles with no significant change in adsorption capacity. The adsorption data followed Langmuir isotherm with the kinetics of pseudo-first-order, which suggests that chemisorption was carried on for removal of MB. The novelty of this work focuses on reusing, through calcination of ACFF, which allows removal efficiency more than 99% up to ten cycles, whereby removal of methylene blue from wastewater by activated carbon fiber felt could be cheaper than other proposed methods.
Keywords: reuse, activated carbon fiber felt, removal, methylene blue, calcination.

Resumen
Este trabajo sugiere la reutilización del fieltro de fibra de carbono activado de acrilonitrilo (FFCA) para la adsorción de azul de metileno de un agua contaminada sintética. Después de cada proceso de eliminación, la FFCA fue tratada térmicamente (calcinada) para su reutilización. La estructura morfológica de la superficie se caracterizó por microscopía electrónica de barrido de alta resolución (MEAR) y el análisis de infrarrojo con transformadas de Fourier (IRTF) se utilizó para identificar grupos funcionales que permitieron el proceso de adsorción. Se llevaron a cabo experimentos por lotes a 27 ° C con un pH de 10. Los resultados revelaron que después de la calcinación, el azul de metileno adsorbido en procesos de remoción anteriores, se convirtió en depósitos de carbono que fueron desorbidos durante los diferentes procesos de eliminación, esto permitió la reutilización de la FFCA durante varios ciclos de adsorción sin un cambio significativo en la capacidad de remoción del colorante Los datos de adsorción se acoplaron a la isoterma de Langmuir con una cinética de pseudo primer orden que sugiere que la eliminación del azul de metileno fue a partir de quimisorción. La novedad de este trabajo se centra en la reutilización, a través de la calcinación, de la FFCA que permite una eficiencia de eliminación de más del 99% hasta por 10 ciclos, por lo que la eliminación de azul de metileno presente en agua contaminada, por fieltro de fibra de carbono activado podría ser más barata que otros métodos propuestos.
Palabras clave: reutilización, fieltro de fibra de carbón activado, remoción, azul de metileno, calcinación.

1 Introduction

The world's population reached 7.7 billion in mid-2019, is expected to reach 8.5 billion in 2030, 9.7 billon in 2050 and 10.9 billion in 2100 (United Nations Department of Economic and Social Affairs Population Division, 2019), which means, an increase in the requirement for transport, health, food, textile industry services, etc. The growth of world's population brings about an increase in

* *Corresponding author. E-mail*: os.lastfrid@gmail.com
Tel. 57-29-60-00, Fax 55073
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environmental pollution, mainly by dyes presents, from textile industry, in effluent streams that empty into water bodies, bringing with it adverse effects on human and marine life (Pérez-Osorio *et al*., 2019), therefore, more and better wastewater treatment techniques are needed. Nowadays, there is a more significant concern to solve a wide variety of environmental pollution problems for the survival of life on our planet, including humans. This a subject of increasing concern to environmentalists and scientists. Anthropogenic water pollution is an activity mainly due to effluents coming out from industries. Textile wastewaters are one of the most important sources of pollution, it is estimated that 10-15% of dyes are lost in the effluent during dyeing process (Senthilkumaar *et al*., 2006). The discharge of these wastewaters influences the natural appearance of the rivers, harming aquatic life and the photosynthesis process of aquatic plants (Carmen *et al*., 2012). The polluting effects of textile dyes against the aquatic environment can also result toxic effects due to their longtime presence in the environment (Gita *et al*., 2017).

In textile industries cationic dyes are applied to fibers such as silk, wool, nylon and modified acrylic fibers using neutral to acid dye baths (Butani *et al*., 2017). Methylene blue (MB, [7-(Dimethyl-amino) phenothiazine-3-ylidene]-dimethyla-zanium chloride) is an example of cationic dye that is most commonly and continuously used for dyeing cotton, wool, and silk (Vargas *et al*., 2011). This cationic dye always contaminates wastewater from industries related to the use and synthesis of MB; this fact is worrisome because MB is a toxic with carcinogenic effects. Toxic consequences have been reported in animals exposed to MB include hypothermia, hemoconcentration, acidosis, hypercapnia, hypoxia, increases in blood pressure, corneal injury, changes in respiratory frequency amplitude, conjunctival damage, and Heinz body formation (Fallon *et al*., 2013). Methylene Blue (MB) is one of the most used dyes due its many applications, mainly as medical treatment and textile dye. In spite of its several applications, it has a number of negative impacts on human beings and animals (Suárez-Vázquez *et al*., 2019). Thus, removal of MB remains a central issue in sanitation of wastewater. For this reason, different methods have been applied, such as coagulation (Villabona-Ortíz *et al*., 2020; Zahrim *et al*., 2013) , chemical precipitation (Tünay *et al*., 1996), membrane filtration (Karisma *et al*., 2018) using polyimide based membranes (Gunawan *et al*., 2019; Febrianto *et al*., 2019), solvent extraction (Pandit *et al*., 2004), reverse osmosis (Abid *et al*., 2012), physicochemical methods (Dos Santos *et al*., 2004), biological methods (Bhatia *et al*., 2017), carbon filtration systems (Rodriguez *et al*., 2020), fluidized bed reactor (Quintal-Franco *et al*., 2020), etc. have been applied for the removal of textile dyes. Among them, adsorption has found to be an efficient and economics process due to its simplicity of operation. Removal of textile dyes by different materials is considered superior to other techniques because it is economical, with high performance and easy operation (Téllez-Pérez *et al*., 2017), moreover, it is one of the most used and effective processes for the removal of synthetic dyes that are difficult to mineralize by conventional methods (Che-Galicia *et al*., 2014). To remove unwanted hazardous compounds from contaminated water at low cost, attention has been focused on various naturally occurring adsorbents such as chitosan, zeolites, fly ash, coal, and various clay minerals (Aziz, 2013; Rahman *et al*., 2013). Today, the most commonly adsorbent used for dye or heavy metal removal is activated carbon (powdered or granular) (Pathania *et al*., 2017). Activated carbon has extended surface area, high capacity of adsorption, higher surface activity degree and also micro-pore structure which is suitable for eliminating dyes from wastewater (El-Sayed *et al*., 2014; Hesas *et al*., 2013; Saytili *et al*., 2015), however, its regeneration is expensive (do Amaral Junior *et al*., 2017).

The MB removal using activated carbon has been widely studied, however, many of studies about wastewater treatment, employ powdered (Corral-Escárcega *et al*., 2017) or granulated activated carbon (Méndez-Hernández and Loera, 2019; Rodríguez *et al*., 2020), however, the difficult separation of these kind of activated carbon from the aqueous solution restricts their practical application. Some disadvantages of powdered and granular activated carbon, involve the need for a packed bed which result in a pressure drop (Balanay & Lungu, 2016). In addition, the difficulty of operation is increased due system bulkiness. Another kind of super-adsorbent carbon material exists called activated carbon fiber (ACF), it is thought to be one of the best adsorbents, since it is an excellent microporous material (Ramos *et al*., 2004) with low mesoporosity and with absence of macroporosity if it is well-produced (Lee *et al*., 2014). ACF is commercially produced by the pyrolysis of carbonaceous materials of synthetic polymers such as rayon (Su *et al*., 2011), pitch (Alcañiz-Monge *et al*., 2008), saran (Yang *et al*., 2014), polyacrylonitrile (Yusof *et al*., 2012) and phenolic resin (An *et al*.,

2009) followed by an additional activation process. Activated carbon fiber felt (ACFF) is a variant of activated carbon fiber which has unique characteristics such as lower density, making it ideal for applications requiring low weight. ACFF is widely used for diverse applications such as air purification, water treatment, chemical (adsorption and desorption for organic compounds and solvents), even in military areas for protection garments and masks (Kuruvilla, 2013). Despite all the advantages and applications of ACF and ACFF, their use has been limited due to their relatively high cost.

For the above, we suggest the reuse of activated carbon fiber felt (ACFF) for MB removal from a synthetic wastewater. ACFF has special characteristics, whose main advantage over other types of activated carbon is the ease of handling, because it can be presented in fabric, woven or yarn forms which makes density considerably lower, turning it ideal for applications requiring low weight (Gómora-Hernández *et al*., 2020). Therefore, the main purpose of the present research is to compare the adsorption capacity of ACFF by methylene blue removal from aqueous solutions by a removal-calcination process. This process sequence was carried out ten times under the same stirring, temperature, and pH conditions. The novelty of such a process is the ACFF reusability, through thermal treatment, which makes an alternative more economical for dye textile removal from aqueous systems.

2 Materials and methods

2.1 Materials

Cationic dye methylene blue ($C_{16}H_{18}ClN_3S{\cdot}xH_2O$), sodium hydroxide (NaOH, 98%), hydrochloric acid (HCl, 37%) and sodium chloride (NaCl, 98%) were purchased from Sigma Aldrich. Polyacrylonitrile (PAN) activated carbon fiber felt was purchased from KoThmex with the following features: fiber diameter of 6-11 (μm), surface area of 700-2000 (m^2/gr), micropore diameter of 0.4-1.0 (A), micropore volume of 1.5-2.0 (mL/g).

2.2 Pre-treatment of activated carbon fiber felt (ACFF)

To remove the adhered atmospheric dust, the ACFF was washed three times with distilled water and dried at 100 °C for 8 hours. After that, the dried ACFF was calcinated at 400 °C for thirty minutes to remove the volatile substances that might have been present. Finally, the ACFF was washed and dried once more, as mentioned above.

2.3 pH_{pzc}

For the point of zero charge (pH_{pzc}) determination (Attia *et al*., 2010) using a nitrogen bubbling to avoid the atmospheric CO_2 influence on pH, 50 mL of 0.1 M NaCl solution were placed in different flasks. Their pH value was adjusted from 2 to 11 by the addition of 0.1 M solutions of NaOH and HCl. When the desired pH value was obtained, 50 mg of ACFF was added to each flask and was stirred for 24 hours. The ph_{pzc} value is the point where the curve pH final vs. pH initial crosses the line pH initial = pH final (Adam, 2016).

2.4 Characterization

The ACFF was characterized by a high-resolution scanning electron microscope (JEOL 6701F) operated at 5.0 kV for surface morphological structure analysis. Initial and residual concentration of MB was determined at different shaking times, with a double beam UV/Vis spectrophotometer (PerkinElmer-Lambda 365) using a (1.0 cm) quartz cell at 665 nm. Functional groups on the ACFF surface were identified by PerkinElmer FTIR spectrometer "Spectrum two" brand.

2.5 Adsorption experiments

Synthetic wastewater was prepared dissolving 70 mg of MB powder in 1L of distilled water while 0.1 M NaOH and 0.1 M HCl were used to adjust the pH of the solution. Batch experiments were carried out by taking a piece of ACFF from 1.5 cm x 1.5 cm (55 mg) into 40 mL of solution at 27 °C. Residual dye concentration was analyzed by UV-Vis spectrophotometer at different time intervals (5, 10, 15, 20, 30, 40, 50 and 60 min) through calibration curve with different initial concentrations of MB.

The percent adsorption was calculated by using the Eq. (1);

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

where C_i is the initial dye concentration (mg/L), C_t is the residual concentration at different time intervals (mg/L). The adsorbed MB amount at time t, per g of ACFF, Qt (mg/g), was calculated using the Eq. (2);

$$Qt = \left(\frac{C_i - C_t}{m}\right) \cdot V \qquad (2)$$

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

2.6 *Thermal treatment (calcination) for reusability*

Once the piece of ACFF was used for MB removal for the first time, it was placed into a muffle furnace under atmosphere air at 400 °C for 30 minutes. After that, it was washed with distilled water and dried at 100 °C for 8 hours for subsequent reuse in a new adsorption process. It is important to emphasize that the same piece of ACFF was used for a removal-calcination process ten times to analyze its performance in reusability.

3 Results and discussion

3.1 *HRSEM analysis*

After pre-treatment, ACFF was characterized by HRSEM for surface morphological structure analysis. Fig. 1A shows that ACFF is made up of several randomly arranged filaments. Figs. 1B, 1C, and 1D show different filaments diameters of 6.74 μm, 10.2 μm, and 1.7 μm. This fact validates the specifications provided by KoThmex. It shows a clean surface, free of atmospheric dust, and organic impurities; therefore, there it is a complete surface area for dye removal.

3.2 *Point of zero charge (ph_{pzc})*

To determinate the ph_{pzc}, the pH drift method was used. According to results shown in Fig. 2, ph_{pzc} is the crossing point between the curve of pH final vs. pH initial and the line pH initial = pH final, being the value of 3.14 for ACFF. According with published studies, at pH < ph_{pzc} the carbon surface has a positive net charge, while at pH > ph_{pzc} the surface has a negative net charge (Amaringo-Villa, 2013). By the cationic nature of MB dye, in this work, the adsorption experiments were carried on at pH>3.14, being a pH = 10 the optimum value for high removal (Said *et al*., 2014).

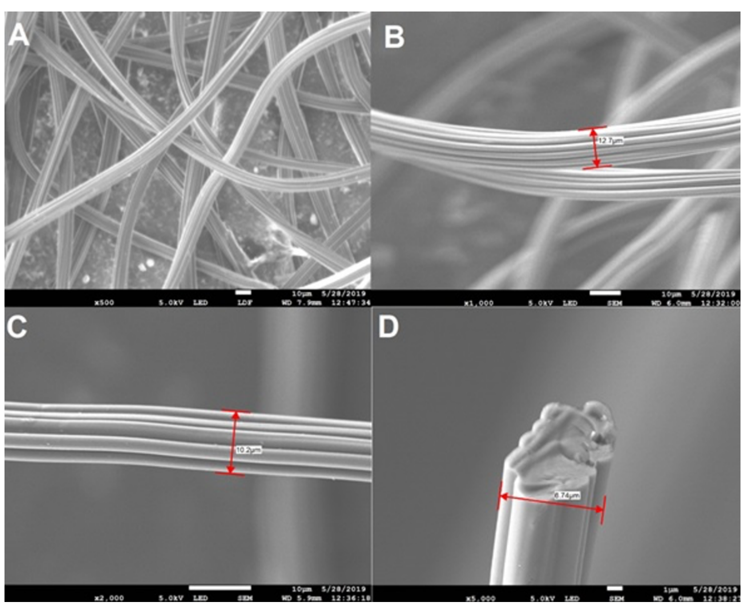

Fig. 1. HRSEM images of ACFF filaments with different diameters.

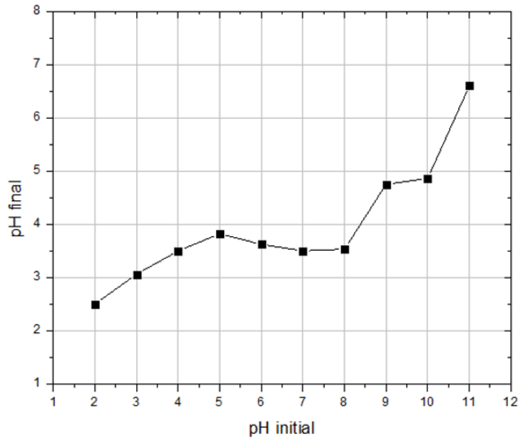

Fig. 2. Point of zero charge (pH_{pzc}) of ACFF by the pH drift method.

3.3 Adsorption analysis

The removal of MB from aqueous solution was carried out at different times (5 min to 60 min). Fig. 3 shows ACFF used for the first time removes 65% from MB at 5 minutes; it can also be appreciated that the removal efficiency improved with an increase in time exposure; a complete removal was achieved at 50 minutes, however, from 20 minutes adsorption is near complete with 99.13% removal. The removal efficiency decreases with ACFF reuse. At 10 minutes, reusing ACFF for the second time, 86.5% was removed; however, when it is reused for the tenth times, the removal percentage decreases to 66%. At 20 minutes, a difference can be observed in removal efficiency between ACFF reused for second and tenth time where the removals are 99% and 81.5%, respectively.

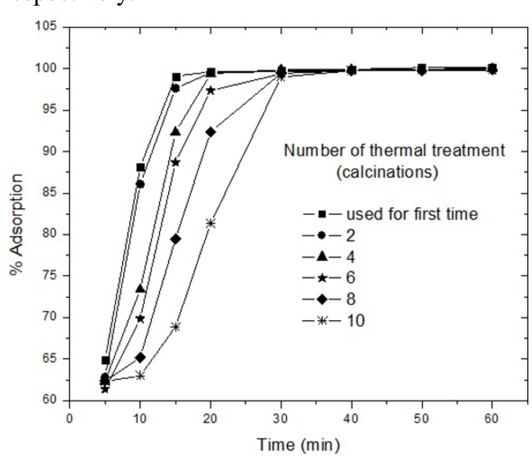

Fig. 3. Percentage dye removal for new and reused ACFF at different times.

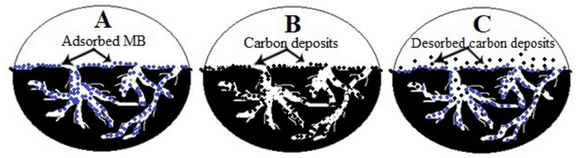

Fig. 4. Matrix of ACFF with adsorbed MB (A), after calcination (B) and being reused (C).

It is evident that for all reusing processes at 30 minutes, the removal percentage is near 100%. Adsorption occurs when the intermolecular attractive forces between the molecules of MB (adsorbate) and the ACFF (adsorbent) are higher than those among adsorbate molecules themselves (Gong *et al*., 2013) as shown in Fig. 4A. After using ACFF for the first time, it was treated by a thermal method, described above, for its reuse. After calcination, adsorbed MB is turned to CO, CO_2 (EC, 2007), and, as shown in Fig. 4B, 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 MB amount decreases, however, after a few minutes and as consequent of stirring, the carbon deposits could be desorbed to the aqueous solution (Fig. 4C), thereby, more MB molecules can be adsorbed. This fact is consistent with Fig. 3.

3.4 Adsorption isotherms

Adsorption isotherms are a graphic representation between adsorbate mass and adsorbent mass at constant temperature. The adsorption data of MB on ACFF were analyzed by Freundlich logarithmic and Langmuir model. The first one describes a relation between the adsorbed amount of adsorbate per gram of the solid at equilibrium (Q_e) and the concentration (C_e) in solution at the equilibrium (mg/L) (Ayawei *et al*., 2017). The Freundlich model is described in Eq. (3);

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

where Q_e is the adsorbed amount of MB at equilibrium (mg/g), C_e is the MB equilibrium concentration (mg/L), K_F is the adsorption capacity, and 1/n is the heterogeneity factor. In Langmuir isotherm, the adsorbent surface has specific homogeneous sites, and the adsorbate forms a monolayer on the surface (Şahin *et al*., 2013), the Langmuir model is described in Eq. (4);

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

where C_e and Q_e are the same parameters described above, Q_{max} and b are constants associated with capacity and affinity adsorption (Ibupoto *et al*., 2018).

Fig. 5 shows the Langmuir and Freundlich isotherms for ACFF. The correlation coefficient (R^2) determines the fitting quality. It can be appreciated that Langmuir isotherms for ACFF used for first time (A) and reused for ten times (C) describe the adsorption process more accurately than Freundlich isotherms (B) and (D), this can be confirmed because, their correlation coefficient is near to one, besides that, for Freundlich isotherms, if 1/n is a negative value the adsorption process is more likely to be Langmuir isotherm. When ACFF is used for the first time the maximum adsorption capacity (Qmax) value for MB removal at 27 °C, is 33.55 mg/g, whilst, for ACFF reused ten times Q max is 30.30 mg/g. The results show that thermal treatment (calcination) for ACFF achieve almost the same adsorption capacity during several removal process using the same ACFF piece.

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

$$R_L = \frac{1}{1 + b \cdot C_0} \qquad (5)$$

where b is Langmuir constant and C_0 is the initial MB concentration (mg/L). If $R_L > 1$, the adsorption is not favorable; if R_L=1, the process is linear in nature; if R_L>0, the process is favorable; if R_L=0, is an irreversible process (Singh *et al*., 2019). Table 1 shows Langmuir and Freundlich isotherms for ACFF reused for 2, 6, 8 and 10 times and according to the R^2 coefficient and R_L factor, during removal process the adsorbed MB amount per ACFF unit mass and remaining MB concentration at 27 °C, under equilibrium conditions, is best described by Langmuir model, which is consistent with isotherms shown previously.

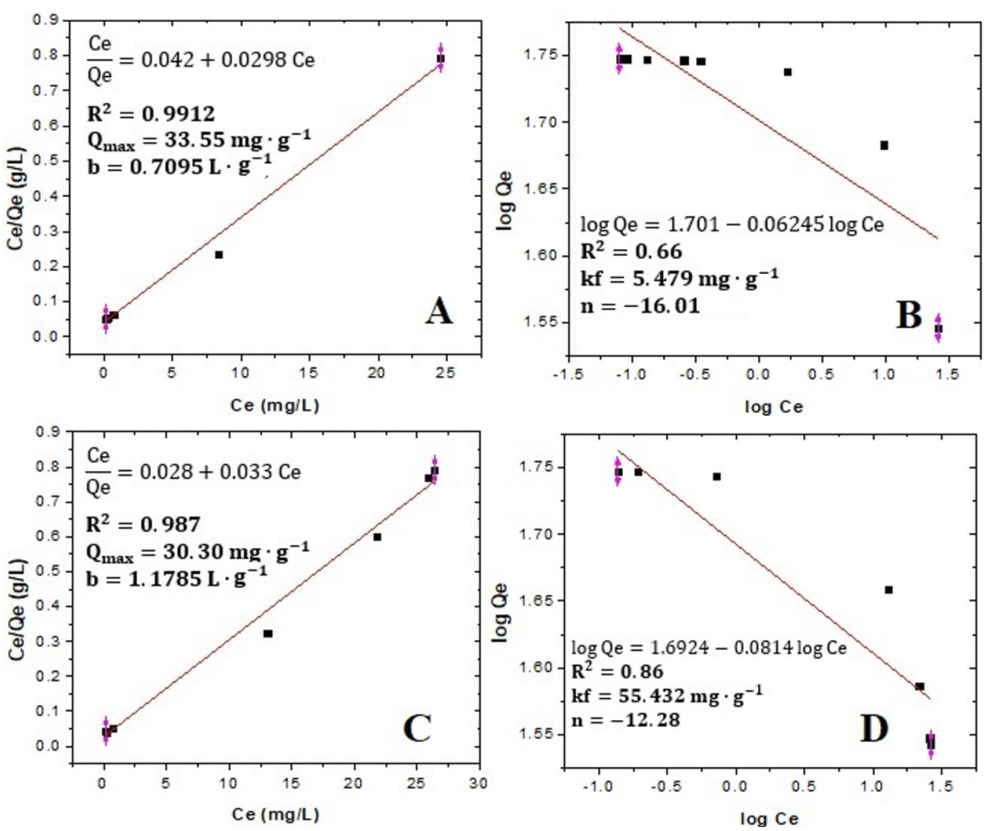

Fig. 5. Langmuir isotherm (A) and Freundlich isotherm (B) for ACFF used for first time. Langmuir isotherm (C) and Freundlich isotherm (D) for ACFF reused for ten times.

Table 1. Constants related to isotherms.

		Langmuir isotherm			Freundlich isotherm		
Reuses	R_L	**R^2**	Q_{max} **(mg·g^{-1})**	**b (L·g^{-1})**	**R^2**	k_f **(mg·g^{-1})**	**n**
2	0.0194	0.9887	33.04	0.7216	0.5629	5.4784	-15.59
4	0.0184	0.9905	32.78	0.7588	0.6595	3.4652	-15.05
6	0.0183	0.9888	32.12	0.7649	0.7298	5.464	-13.46
8	0.0139	0.9885	31.57	1.011	0.6987	5.4412	-12.86

According with Table 1, the dimensionless separation factor values, from the Langmuir model, were $0<R_L<1$, whereby adsorption processes carried out were favorable with affinity between ACFF and MB by bonding covalent.

3.5 Adsorption kinetics

Kinetics describe the adsorption rate of MB 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),

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

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

where Q_e is the amount of MB adsorbed at equilibrium (mg/g), Q_t is the amount of MB adsorbed at time t (mg/g), 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. 6 shows pseudo-first-order kinetics model (A) and pseudo-second-order (B) for MB adsorption onto ACFF used for the first time and, pseudo-first-order (C) and pseudo-second-order (D) for ACFF reused ten times.

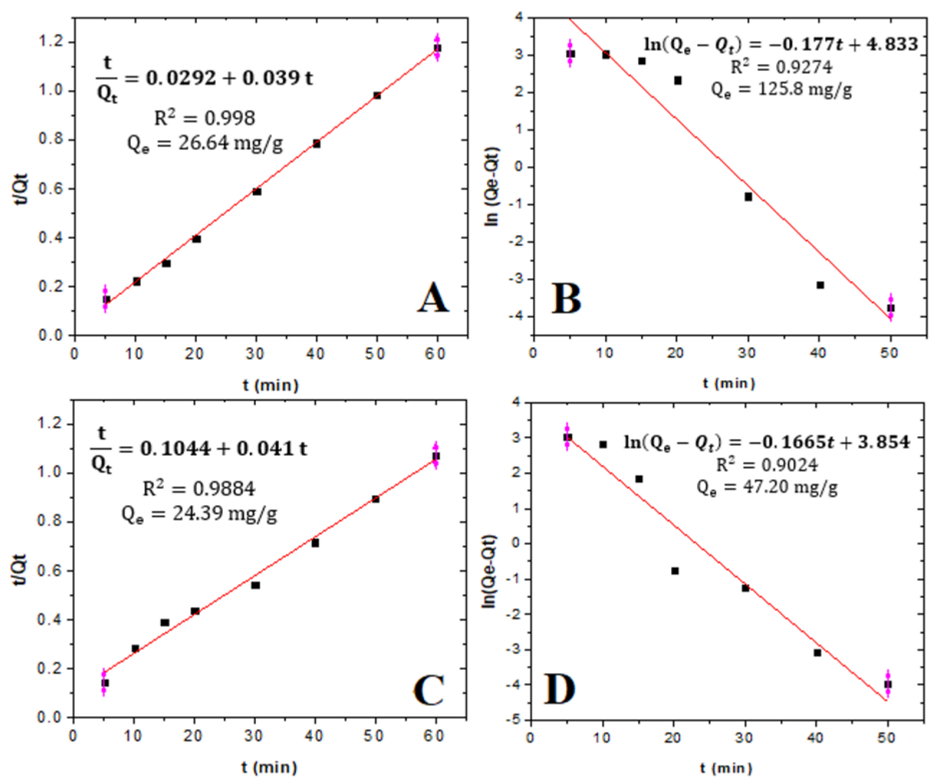

Fig. 6. Pseudo first-order kinetics model (A) and pseudo second-order (B) for ACFF used for first time. Pseudo first-order kinetics model (C) and pseudo second-order (D) for ACFF used for ten times.

It is clear from R^2 values shown that the pseudo-first-order kinetics model best describes the adsorption of MB on ACFF for first or for the tenth time, and it can be supported with the Q_e calculated, which is similar to the experimental Q_e. The pseudo-first-order kinetic model suggests chemical interactions between MB and ACFF by the presence of functional groups present on the surface of ACFF that favored the adsorption process.

3.6 Chemical structure properties

FTIR analysis was performed to identify functional groups from ACFF, which allowed adsorption of MB. Fig. 7 shows the band at 1560 cm^{-1} due to a carbonyl (Norhaniza Yusof *et al*., 2016; Hernández-Botello *et al*., 2020) or lactone groups (Andrade *et al*., 2018) formed by the interaction with atmospheric oxygen during the calcination process, which allows the formation of covalent bonds between the ACFF carbonyl group and the cationic species of MB (Salazar-Rabago *et al*., 2017). After second adsorption process, MB removed is turned to CO and CO_2 which allows covalent binding between ACFF surface and these degradation products, therefore, the only band in the FTIR spectra shows that more carbonaceous material were formed after calcination process due at high temperature used.

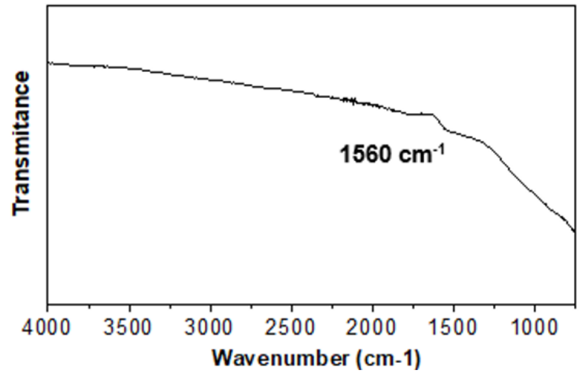

Fig. 7. FTIR spectra of ACFF.

Isotherm analysis shows that the adsorption mechanism has a monolayer adsorption described for the Langmuir model with pseudo-first-order kinetics. According to FTIR results, the proposed adsorption mechanism was chemisorption by covalent bonds between MB and ACFF carbonyl groups. These features make the calcination of ACFF an efficient treatment for reusing for several removal cycles.

Conclusions

The current study suggests reusing of polyacrylonitrile based activated carbon fiber felt (ACFF) for adsorption of methylene blue (MB). The thermal process (calcination) of ACFF under atmosphere air, after every removal process, allows reusing it until for ten times, using the same ACFF sample, with no significant change in adsorption capacity (from 33.55 mg g^{-1} to 30.30 mg g^{-1}) having a high removal percentage (> 99%). From the third reusing process, removal percentage decreased because of the carbon deposits formation by calcination of adsorbed MB and surface area reduction; however, the use of magnetic stirring, in every washing step after removal process, allowed desorption of carbon deposits after 30 minutes and the subsequent increase in adsorption percentage. For tenth reuse, necessary time to achieve a removal greater than 99% were 30 minutes, which means high removal percentages can be obtained when ACFF is reused by increasing removal process time.

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