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Methylene Blue Removal by Activated Carbon from *Platanus orientalis* Leaves

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Abstract

In this study, activated carbon was produced from *Platanus orientalis* leaves and the adsorption of methylene blue onto activated carbon was investigated. Facile chemical activation route was conducted for activation process of lignocellulosic leaves. $ZnCl_2$ was used as an activating agent at different ratios. Morphological, structural properties and adsorption characteristics of samples were determined by using field emission scanning microscopy, Fourier transform infrared spectroscopy, Braunauer-Emmet-Teller spectroscopy and UV-vis spectroscopy. Surface characterization showed that activation with $ZnCl_2$ resulted high surface area ($\sim 1024 \text{ m}^2 \text{ g}^{-1}$) and total pore volume ($\sim 0.431 \text{ cm}^3 \text{ g}^{-1}$) compared with crude carbonized leaves samples.

Keywords: Methylene blue, *Platanus Orientalis* Leaves, Activated Carbon, Dye Removal

Introduction

Dyes and pigments are highly used chemicals for several industries such as textiles, leather, paper, etc. Global annual production of dyes and pigments in 2021 is estimated over 10 000 ktons (Wong, et al., 2004). Around 10-15% of dye products are wasted during these industrial processes and this creates huge amount of pollution (Dahiya and Nigam, 2020). Among chemical and biological dyes, methylene blue (MB) is a widely used cationic dye which especially used in cotton or silk dyeing. Its wastewater suffers from poor biodegradability, high chromaticity and high toxicity which adversely affect health in the water environment (Kuang, et al., 2008). Therefore, removal of MB from effluent is of great importance in order to protect environment. Also, dimensions of MB molecule are $\sim 13.82 - 14.47 \text{ \AA}$ in length and $\sim 9.5 \text{ \AA}$ in width (Dotto et al., 2015; Macedo et al., 2006). These dimensions are making MB a suitable material for characterization of meso- or microporous surfaces.

Platanus orientalis is one of the common trees in the world from southeast Europe to India and China. When it is mature enough, it can easily be noticed from widely spreading branches and characteristic leaves with multiple lobes. Since it is a large tree and can grow up to 30-40 m tall, it is cultivated in order to obtain timber esp. for furniture production. It is also a cultural symbol in Iran, Turkey and India (Abadian, et al., 2015; Rinaldi et al., 2019; Sevik, et al., 2019). Even in Turkey there is a district called Cinarcik (means little *Platanus orientalis*) in Yalova. This name was given because of dominant *Platanus orientalis* forests in the vicinity. *Platanus orientalis* is a deciduous tree and depending on the maturity of the tree, it sheds

thousands of leaves during autumn. Since *Platanus orientalis* leaves (POL) are highly abundant, sustainable source and free of charge, it can be used for various applications including adsorbents (Abadian et al., 2015; Mahvi, et al., 2007; Peydayesh and Rahbar-Kelishami, 2015; Sert et al., 2008), and bioindicators (Norouzi and Khademi, 2015). Among these, wastewater treatments are the most common application. In these studies, generally leaves were collected, washed, dried and ground. Then they were used as adsorbents for predetermined chemicals. Since they are biological structures, their components have various functional active sides, they can be used as biosorbents for cadmium (Mahvi et al., 2007), copper(II) (Abadian et al., 2015), lanthanum and cerium(III) adsorption (Sert et al., 2008). In addition to these, leaves of *Platanus orientalis* was also used for hydro char fabrication. Gu et al., prepared hydro char from *Platanus orientalis* leaves by combination of persulfate oxidation and hydrothermal treatment. As reported in the study, energy yield increased noticeably (Gu et al., 2017). Active carbon fabrication was reported as another application of POL. Liu et al., fabricated active carbon by a microwave assisted process using H_3PO_4 for activation. The surface area of the active carbon was around $1090 \text{ m}^2 \text{ g}^{-1}$ with the pore volume of 1.468 cm^3 . Cr(VI) adsorption was investigated and results well-fitted with pseudo-second-order model (Liu et al., 2018). One recent study about POL based active carbon was carried out by Jiang et al. Microspheres were synthesized by combination of hydrothermal carbonization and calcination. H_2SO_4 and KOH were used as the catalyst and activator, respectively. The specific surface area was reported as $1355.53 \text{ m}^2 \text{ g}^{-1}$. Samples showed good adsorption behavior for rhodamine B and methyl

orange mainly because of hydroxyl and carboxyl groups on the surface (Jiang et al., 2020). Musah et al. fabricated two types of active carbon from POL. First sample was only thermally treated second sample was both thermally treated and treated with H_3PO_4 . The second sample showed higher performance in terms of methylene blue removal from the solution. However, surface area was not given in the study (Musah, Peng, and Xu, 2020). As can be seen from the recent literature, the number of the studies about POL based active carbon is very limited. In the studies mentioned above, microwave and hydrothermal synthesis routes were used. For the activation H_3PO_4 , KOH were used as activators. In this study another route and chemicals were used for the preparation of active carbon. $ZnCl_2$ activated carbon from POL was synthesized and used as an adsorbent for MB. Ground POL were impregnated into $ZnCl_2$ /water solutions and dried. Carbonization was carried out 700 °C. In order to increase the surface area, samples were treated with HCl solution. $ZnCl_2$ mass ratio on yield, morphology, surface texture and adsorption performance were analyzed, and the most optimum ratio was used in determination of kinetic MB adsorption measurements. As far as known, there is no study in the literature that focused on this type of process for POL based active carbon fabrication and characterization.

Materials and Methods

Activated Carbon Preparation

Schematic illustration of experimental procedure used in this study was shown in Fig. 1. Dry leaves of plane tree (*platanus orientalis*) were collected from Samanlı Mountains (Yalova, Turkey) in October 2020. Dry leaves were collected from the trees that were around 20-30 years old. Firstly, raw material was washed with distilled water in order to remove the dirt for 4 times and dried in oven at 100 °C for 12 h. Dried leaves were ground and sieved by using a stainless-steel sieve (100 mesh <149 μm). 1, 2, 3 g $ZnCl_2$ was dissolved in 100 ml distilled water and 3 different solutions were prepared. Following that, 5 g of sieved leaf particles were impregnated into $ZnCl_2$ solutions. As a result of this 3 different mixtures were obtained with the leaf to activating agent ratios (POL:AA) of 5:1, 5:2 and 5:3, respectively (Table 1). Then, suspensions were mechanically mixed and sonicated (Wisd, Ultrasonic Cleaner, Model#: WUC-A03H, 40 kHz) for 3 h at room temperature and following that, samples were dried in convection oven (Kenton China, KH-35A) at 100 °C for 12 h.

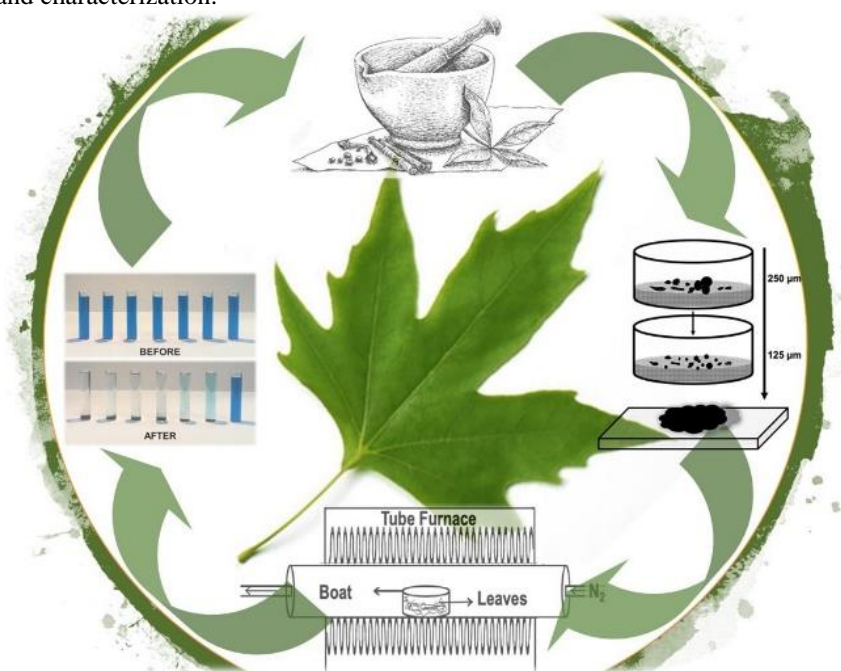


Figure 1. Schematic illustration of experimental procedure

All dried samples were carbonized at 700 °C for 1 h with 5 °C min^{-1} heating rate under N_2 flow of 1 L min^{-1} in the tube furnace (MTI, OTF-1200F). The carbonization yield was calculated according to following equation:

$$\%Yield = \frac{Mass_{output}}{Mass_{input}} \times 100 \quad (1)$$

The carbonized leaves were treated with 1M HCl solution to remove Zn metal or ZnO. Then, samples were purified with deionized water and ethanol until

getting neutral pH, and samples further dried at 80 °C for 24 h. For comparison, ground and sieved leaves were directly carbonized without any extra treatment. Sample code, content, carbonization yield and weight loss values were given in Table 1.

Structural, Morphological and Textural Characterization

The surface morphology of the samples was analyzed by a field emission scanning electron microscope (FESEM, 30 kV). Prior to analysis samples were sputter coated (3 – 6 nm) by Au/Pd alloy. Structural

properties of the samples were characterized by using Fourier transform infrared spectroscopy (FTIR, Perkin Elmer, Spectrum 100 IR) and attenuated total internal reflectance (ATR) spectroscopy was used. Spectra were recorded between 550 and 4000 cm^{-1} in the transmittance mode with spectral resolution of 4 cm^{-1} at a scan rate of 4 scans. Surface texture of activated carbon was characterized by N_2 adsorption at 77 K using a surface area analyzer (Quantachrome Co., USA). All samples were degassed at 120 °C for 3 h.

Table 1. Sample identification and production yield values of activated carbon

Sample Code	Leaf content (g)	ZnCl ₂ content (g)	%Yield
S1	5.00	0.00	28.45
S2	5.00	1.00	34.49
S3	5.00	2.00	39.36
S4	5.00	3.00	41.57

Analysis of Methylene Blue Adsorption

The concentration of MB (Merck) in solution was determined by using a UV-visible spectrophotometer (PG Instruments) at λ_{max} : 668 nm. Adsorption isotherms were determined by using different initial MB concentrations (0.5 – 20 mg L^{-1}) around ~pH 5.5. Equal masses of 0.01 g of adsorbent were added to 100 mL solutions. Samples were agitated for 24 h at 25 °C. The samples were centrifuged at 3000 rpm for 10 min

prior to measurements. The amounts of adsorbed MB at equilibrium (Q_e , mg g^{-1}) and at time (Q_t , mg g^{-1}) were calculated by using following equations:

$$Q_e = (C_0 - C_E) \times V/m \quad (2)$$

$$Q_t = (C_0 - C_t) \times V/m \quad (3)$$

where C_0 , C_E and C_t (mg L^{-1}) are the concentrations of MB at start, at equilibrium and at time t (5 – 120 min), respectively. m (g) is the mass of adsorbent and V (L) is the volume of the solution.

Results and Discussion

Morphological Characterization

In order to understand the effect of activator to biomass ratio on surface morphology, FESEM was used. SEM images of S1 – S4 were shown in Fig. 2. Low magnification and high magnification of the samples were given in order to see the particle and surface morphology. All samples showed similar particle morphology regardless of the content. However, surface morphology showed some differences. Parallel with the BET analysis S3 showed more features on the surface those probably led to higher methylene blue absorption.

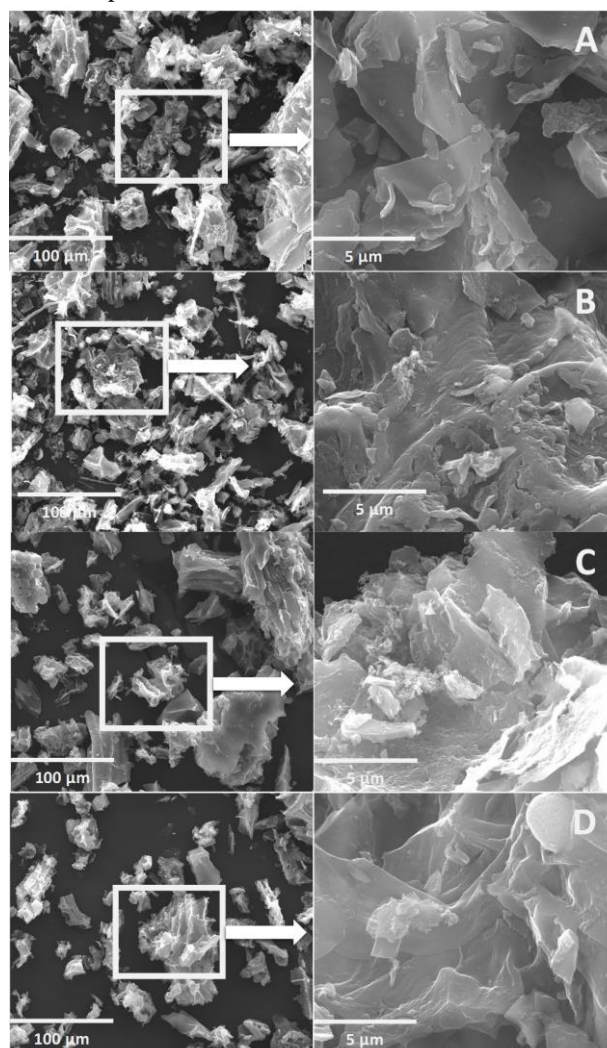


Figure 2. SEM images of carbonized and activated POL samples at different magnifications: a) S1, b) S2, c) S3 and d) S4

BET Isotherms

The isotherms of S2, S3 and S4 seemed to be combination of Type I and Type II of the IUPAC classification which observed with solids of plate-like particles that create slit-shaped pores (Sing et al., 1985).

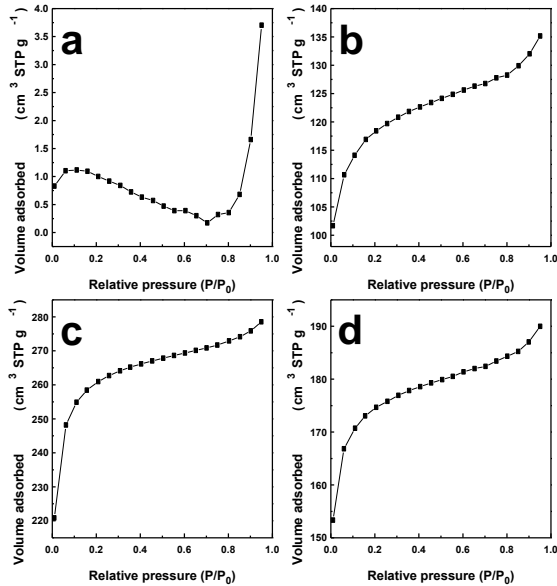


Figure 3. Adsorption isotherms of N₂ at STP for a) S1, b) S2, c) S3 and d) S4

All textural parameters related with samples were summarized in Table 2. As seen from the results, surface area of activated carbon samples was increased from 90 to 200 times by thermochemical activation. According to average pore radius results, mesoporous structure of carbonized *platanus orientalis* leaves was turned into microporous structure by activation. Also, around four times increment was observed in pore volume of the carbonized leaves by activation.

Table 2. Textural parameters of inactivated and activated carbon from *platanus orientalis* leaves

Sample No	Surface Area (m ² g ⁻¹)	Average Pore Radius (nm)	Pore Volume (cm ³ g ⁻¹)	Total Pore Volume (cm ³ g ⁻¹)
S1	4.740	2.41680	0.007	0.005728
S2	454.795	0.91940	0.028	0.2091
S3	1023.92	0.84161	0.029	0.4309
S4	687.074	0.85542	0.026	0.2939

FTIR Spectroscopy

As shown in Fig. 4, FTIR measurements were performed for all inactivated (S1) and activated carbon samples. All activated carbon samples showed identical patterns. There exhibited a broad absorption band at 3200 – 3500 cm⁻¹, which corresponds to the stretching vibration of –OH group in all samples (ElShafei, ElSherbiny, Darwish, and Philip, 2017). There were two characteristic peaks appeared at 2920 and 2850 cm⁻¹ corresponding to sp³–CH stretching (Bhadusha and Ananthabaskaran, 2011). Also, additional aromatic C=C stretching peak at 1660 cm⁻¹ was observed for all samples (Fanning and Vannice, 1993; Mopoung, Moonsri, Palas, and Khumpai, 2015). It was also observed broad aromatic sp²–CH bend at 800 – 950 cm⁻¹ for activated carbon samples (Guo and Lua, 2002). The characteristic transmission % intensity was gradually increased with the increase of ZnCl₂ addition during carbonization.

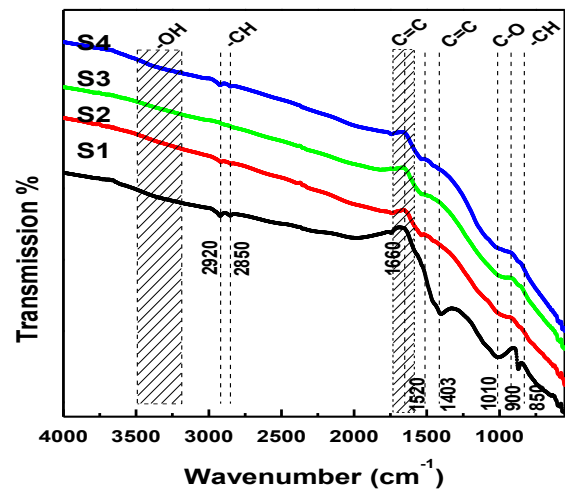


Figure 4. FTIR spectra of carbonized (S1) and activated (S2, S3, S4) *platanus orientalis* leaves

Adsorption isotherms

The effect of carbon source on MB adsorption at 12 ppm initial dye concentration is shown in Fig. 5b. It is obvious that the percentage of dye removal of S3 is the highest value (73.45%). The dye removal efficiencies of S1, S2 and S4 are 23.36%, 20.18% and 48.84%, respectively.

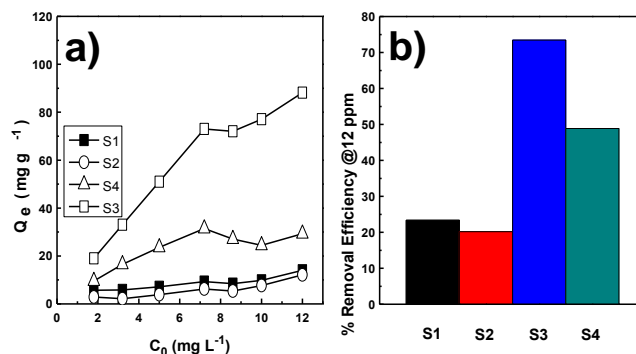


Figure 5. a) MB removal by using different carbon sources and different initial MB concentrations, b) MB removal efficiencies of different carbon sources at 12 ppm MB concentration

The characteristics of Langmuir isotherm can be expressed by using separation factor (R_L) which is defined by:

$$R_L = \frac{1}{1 + k_1 C_0} \quad (4)$$

where k_1 and C_0 are Langmuir constant and the highest dye concentration, respectively. According to adsorption isotherm shown in Fig. 6a, R_L value was found as 0.074 which indicates type of the isotherm to be favorable (Kadirvelu, Thamaraiselvi, and Namasivayam, 2001).

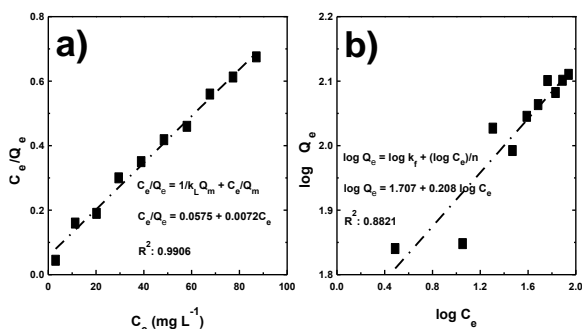


Figure 6. a) Langmuir and b) Freundlich adsorption isotherms of MB dye onto S3

Table 3. Adsorption isotherms and their constants for MB adsorption on S3

Model	Equation	Constants	Values
Langmuir isotherm	C_e/Q_e	k_1	0.1252
	$= C_e/Q_{max}$	Q_{max}	138.888
	$+ 1/k_1 Q_{max}$	R^2	0.9906
		R_L	0.074
Freundlich isotherm	$\log Q_e = \log k_f - (\log C_e)/n$	k_f	50.933
		N	4.7892
		R^2	0.8821

Adsorption kinetics

In this study, adsorption kinetics of MB on S3 were analyzed by using 3 different kinetic models (Fig. 7). The rate of removal % of dye molecules from solution and time dependent dye concentration by using S3 were seen in the Fig. 7a. All the MB molecules were removed from solution in 5 h and corresponding equivalent concentration of MB was found 41.0298 mg g⁻¹. The most effective model was determined according to consistency between theoretical and experimental results (R^2 values). All kinetic model equations and corresponding model parameters were given in Table 4. In the first model (Lagergren-Svenska model), it is assumed that adsorption rate is proportional to the difference between equivalent (Q_e) and time dependent concentration (Q_t) (Rudzinski and Plazinski, 2006). As seen from Fig. 7b, there was a linear relationship between $\log(Q_e - Q_t)$ and time. k_1 and Q_e values were calculated from the slope and intercept of the plot, respectively (Table 4). Like pseudo first-order kinetic model, the linear relationship between t/Q_t and time was observed for pseudo-second-order kinetic model. Q_e and k_2 values were calculated from

the slope and intercept of the plot, respectively (Table 4).

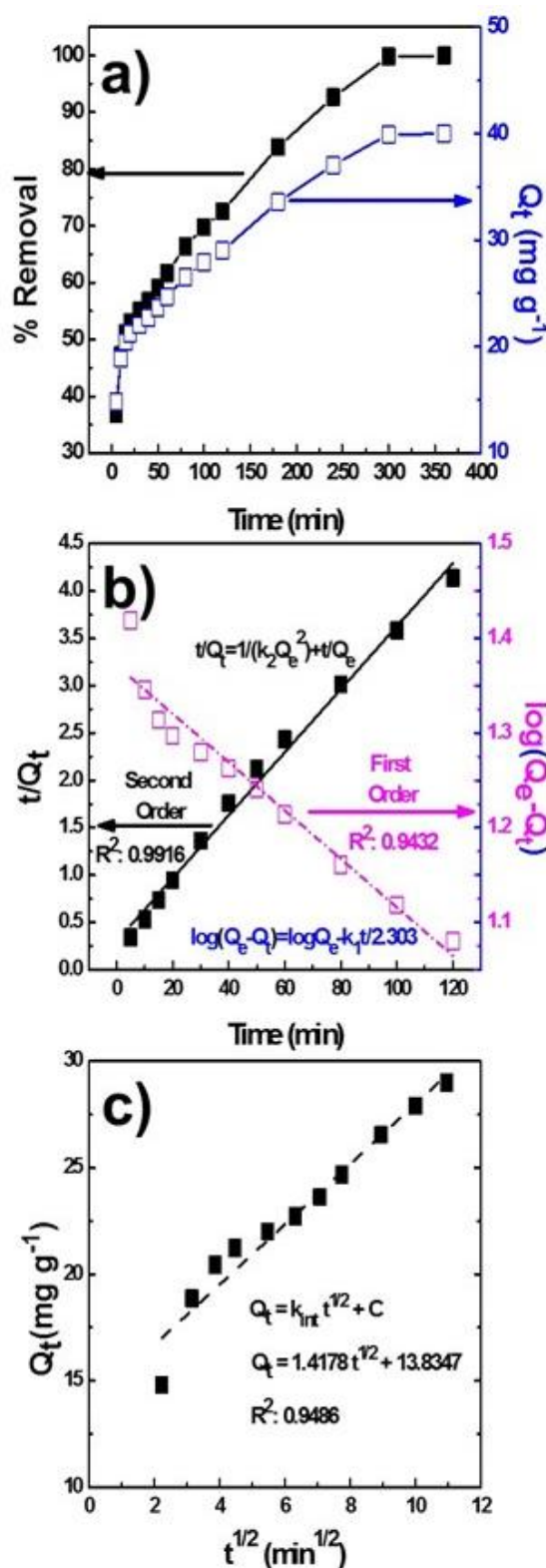


Figure 7. a) Time dependency of MB removal/adsorption by using S3, b) corresponding pseudo-first and -second order model plots, and c) Weber-Morris intra-particle diffusion model

In addition to first- and second-order models, diffusion mechanism of MB was analyzed by using intra-particle diffusion model (Fig. 7c). According to this model,

adsorption of dye molecules on adsorbent occurs in two steps. Dye molecules firstly migrate from solution to the surface of adsorbent and then diffuse into interior pores of adsorbent (Doke and Khan, 2017). There was a linear relationship between Q_t and $t^{1/2}$. k_{int} and C values were calculated from the slope and intercept of the plot, respectively (Table 4). As seen from Fig. 7c, fitted line was not passing through the origin. This indicates that adsorption process has not only been controlled by intra-particle diffusion. Among these models, pseudo-second order kinetic model gave the highest R^2 value which was 0.9916. So, pseudo-second-order model was the best model to explain MB adsorption process onto S3.

Table 4. Kinetic model parameters of MB adsorption on S3

Model	Equation	Constants	Values
Lagergren-Svenska model	$\log(Q_e - Q_t)$	k_1	$5.9878 \cdot 10^{-3}$
	$= \log Q_e - k_1 t / 2.303$	Q_e R^2	23.5179 0.9432
Pseudo second-order model	t/Q_t	k_2	0.00368
	$= 1/k_2 Q_e^2 + t/Q_e$	Q_e R^2	30.03003 0.9916
Intra-particle diffusion model	Q_t	k_{int}	1.4178
	$= k_{int} t^{1/2} + C$	C R^2	13.8347 0.9486

It was known that activated carbon production from lignocellulosic materials strongly depends on activation method. For future studies, it is recommended to use different activation chemicals, different activation and carbonization conditions to improve surface texture or adsorption characteristics. Also, stability, scalability, and recycling of adsorbent under different conditions should be studied for a sustainable filtration process afterwards. In addition to adsorption or filtration applications, this material might be a good alternative for energy storage and conversion applications such as electrode materials for capacitors or secondary batteries or can be used as additives for separators. Future research will be focused on these applications.

Conclusions

The present study showed that *platanus orientalis* leaves can be used as an alternative raw material for activated carbon production for the removal of MB dye from aqueous media. Surface area of POL was increased around 200 times by the thermochemical activation ($\sim 1024 \text{ m}^2 \text{ g}^{-1}$). Adsorption behavior of activated POL is described by a monolayer Langmuir type isotherm. Kinetic data of activated POL follow second order kinetic model. The value of the maximum adsorption capacity, Q_e (30.03 mg g^{-1}) is better than commercial activated carbon reported in earlier studies. Since POLs have high cellulosic content and micropores on their surfaces, they might be promising materials for active carbon fabrication.

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