



Preparation of activated carbon from waste tires, and its use for dye removal

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ABSTRACT

In this study, activated carbon derivation from end-of-life tires (ELT), and color removal from wastewater with produced activated carbon were investigated. In activated carbon production stage, granulated waste tires were first washed in KOH solution at 1:1 (1-K) and 1:2 (2-K) impregnation ratios prior to thermal activation. Chemically activated waste tires were then subjected to pyrolysis. Following pyrolysis, solids (char) were used as adsorbent material. Surface area of 2-K adsorbent was measured to be 2.945 m²/g by Brunauer-Emmett-Teller analysis. In the second stage of the study, adsorptive removal of methylene blue dye from synthetically prepared wastewater was investigated using 1-K and 2-K activated carbons. Results have shown that activated carbon produced in the study was an appropriate adsorbent for removal of dye. The optimum conditions for 2-K adsorbent, which yielded 83% dye removal, were pH 6.5, 30°C temperature, 7.5 g/L adsorbed dose, and 30 min contact time. Isotherm, kinetic and thermodynamic studies were carried out in order to elucidate the adsorption mechanism. Since ELT derived activated carbon provided good efficiency in adsorption of dye, whole operation may be scaled-up considering the necessity of aligning rates of ELT production and its upcycling.

Keywords: End-of-life tires (ELT); Methylene blue; Adsorption; Activated carbon; Pyrolysis

1. Introduction

Production of low-cost activated carbon alternatives has emerged from the fact that despite being a widely used adsorbent due to its characteristics such as large surface area, high adsorption capacity and porous structure, commercial activated carbon is costly [1]. Therefore, producing activated carbon from upcycled materials is a viable option to investigate. Approximately 17 million tons of waste rubber is produced every year worldwide with an annually increasing trend [2]. After completion of their service time, disposal of end-of-life tires (ELT) in solid waste disposal areas without making a comprehensive evaluation is not only costly but also hazardous in terms of environment and human health [1,3]. Since storage of ELT in solid waste disposal sites is

against the legislation, valorization options must be considered. Pyrolytic activated char, which is the end product of ELT pyrolysis with high carbon content, presents good adsorbent characteristics. ELT derived activated carbon has been used to remove various pollutants such as pesticides, dyestuff and phenol from aqueous solutions [4].

The widespread use of dyestuffs and their discharge to environment cause various environmental problems in the ecosystem. Reverse osmosis, ion exchange and adsorption methods are used for removal of dyes from aqueous solutions [5,6]. However, especially membrane technologies have limitations such as low efficiency, high maintenance, and high cost. Adsorption process is one of the most effective and efficient methods that can be used in dye removal when a suitable adsorbent is used [7–9]. Commercial activated

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carbon performs with high removal efficiencies due to its high porosity and subsequent large surface area. However, its cost has directed researchers to look for more economical alternatives [1].

In this study, effects of adsorbent concentration, concentration of dye, contact time, pH and temperature on removal of synthetic Methylene Blue (MB) via ELT derived activated carbon adsorption was investigated.

2. Material and methods

2.1. Dyestuff

In order to determine adsorption capacities of activated carbon, MB was used as adsorbate. MB ($C_{16}H_{18}ClN_3S_3H_2O$) is a dark blue dyestuff with strong adhesion to water, and thus it is able to dissolve easily in water.

2.2. Activated carbon production

In the study, activated carbon used in adsorption experiments was obtained by chemical and thermal activation of ELT. In activated carbon production from granular waste tires, the first stage of activation process was chemical activation. KOH (Merck, Germany) was used at this stage. Ratio of ELT to chemical (impregnation rate) was 1:1 and 1:2. In chemical activation, 30 g KOH was dissolved in 200 ml of aqueous solution. 30 g of waste rubber was stirred in KOH solution at 40°C for 6 h. Solution was then filtered to remove excess chemicals. The residue was then dried in oven at 105°C for 24 h [10–15].

After chemical activation, ELT samples were subjected to thermal activation by pyrolysis. Nitrogen gas (N_2) with a flow rate of 1 L/min was introduced into the reactor throughout the process, and an inert, oxygen-free environment was provided in the reactor. Chemically activated 50 g of ELT was fed to the reactor, and pyrolysis was performed at 700°C. Heating rate of the reactor was 23°C/min. Pyrolysis was carried out for 2 h in order to achieve a homogenous carbonization. After 2 h, the residue obtained was washed with distilled water, and then washed with 0.1 N HCl solutions until pH reached 6–7. Obtained product was filtered and weighed after drying at 105°C for 2 h. Activated carbon samples were coded as 1-K (1:1) and 2-K (1:2) according to their chemical activation rate.

2.3. Analytical methods

In order to determine the physical, chemical and morphological properties of the activated carbon and raw waste tires (ELTs), following analyses were performed. Methods SM 2540B [16], SM 2540E [17] and SM 2540B [16] were used to determine moisture content of raw waste tires, ash content, and solid matter, respectively [13,14]. Scanning electron microscope (SEM) analysis was carried out to determine changes in the surface structure of ELT, solid matter obtained after chemical activation of ELT and activated carbons obtained after carbonization process. JEOL JSM-6610 (Japan) device was used in SEM analysis. For the CHNS-O elemental analysis, a Thermo-Flash 2000 CHN-S elemental (China) analyzer was used, and the IKA C200 Bomb Calorimeter was

used for calorific value analysis. Surface area measurement Brunauer-Emmett-Teller (BET) analysis was performed with Quantachrome Autosorb IQ₂ device (USA).

2.4. Adsorption experiments

In the batch adsorption studies, 0.5, 0.75, 1, 1.5, 2, and 3 g of adsorbent were added to solutions with initial MB concentrations of 5, 10, 15, 20, 30, 50 mg/L. Effects of initial concentration of solution, dose of adsorbent, contact time, temperature and solution's pH were investigated. Color was monitored at 665 nm using a UV spectrophotometer (Shimadzu UV1201, Japan). Then, a calibration curve was plotted from dye solutions prepared in the concentration range of 2–300 mg/L ($R^2 = 0.998$) to determine dye concentrations.

3. Results

3.1. Activated carbon analysis

For pyrolysis, materials with high carbon and hydrogen content are the most suitable substances. For this reason, elemental analysis and thermal value analyses were performed primarily to determine the suitability of ELT samples for thermal activation. Comparative results of elemental analysis and thermal value of both raw ELT and activated carbon samples are presented in Table 1. When the values shown in Table 1 were examined, it was observed that ELT sample contained 85.35% C and 7.70% H. These values indicated that ELT samples were suitable raw materials for pyrolysis methods [15]. In addition, 99.48% solid content was another indicator that waste tire samples were suitable for pyrolysis.

In order to observe the changes in pore structure after activation, SEM images of both raw samples and post-activation samples are presented in Fig. 1. SEM images revealed a very smooth and non-porous structure for raw ELT that was not exposed to any treatment. Presence of a porous structure in 1-K and 2-K activated carbon samples was observed. When SEM images of 1-K and 2-K activated carbons were compared structurally, 2-K activated carbon had a more porous structure, and its surface was rougher than that of 1-K's (Fig. 1).

At high temperatures (700°C), pyrolysis process changes the polymeric structure of raw material. During this change, porous structure is formed by removal of volatile substances in the structure of ELT. Following the thermal activation by pyrolysis, chemicals are removed from the activated carbon surface, and this rough form is observed.

Surface area of the adsorbent is important for determination of adsorption capacity. Structural properties of raw product, chosen activation method, temperature and exposure time are the factors that can change surface area and pore properties [19–21]. Surface area of raw ELT was measured to be 1.349 m²/g. The 1-K and 2-K activated carbons had surface area measurements of 2.867 and 2.945 m²/g, respectively. As a result of chemical activation, not only a mere duplication in surface area was observed when compared to raw ELT but also similar surface area values were obtained despite different ELT/KOH ratios. However following thermal activation (pyrolysis), surface areas of 1-K and 2-K adsorbents were measured as 36.268 and 42.61 m²/g, respectively (Table 2).

Table 1
Ultimate and proximate analysis of waste tire and thermally activated tire

	Ultimate Analysis (% w/w)					HHV kcal/kg
	C	H	N	S	O ^a	
Raw Waste tire						
Martinez et al. [16]	86.30	0.30	0.30	2.80	10.30	–
Aslan et al. [17]	84.20	7.1	0.4	2.1	6.2	7,772
Present study	85.35	7.7	–	–	6.95	9,169
Activated Carbon						
Lopez et al. [18]	84.98	0.83	0.69	–	13.5	–
Present Study	66.93	–	–	–	33.07	6,010
	Proximate Analysis ^b (%w/w)					
	Moisture	Ash	Solid matter			
Present study	0.52	4.72	99.48			

^aCalculated by difference

^bMeasured on dry based

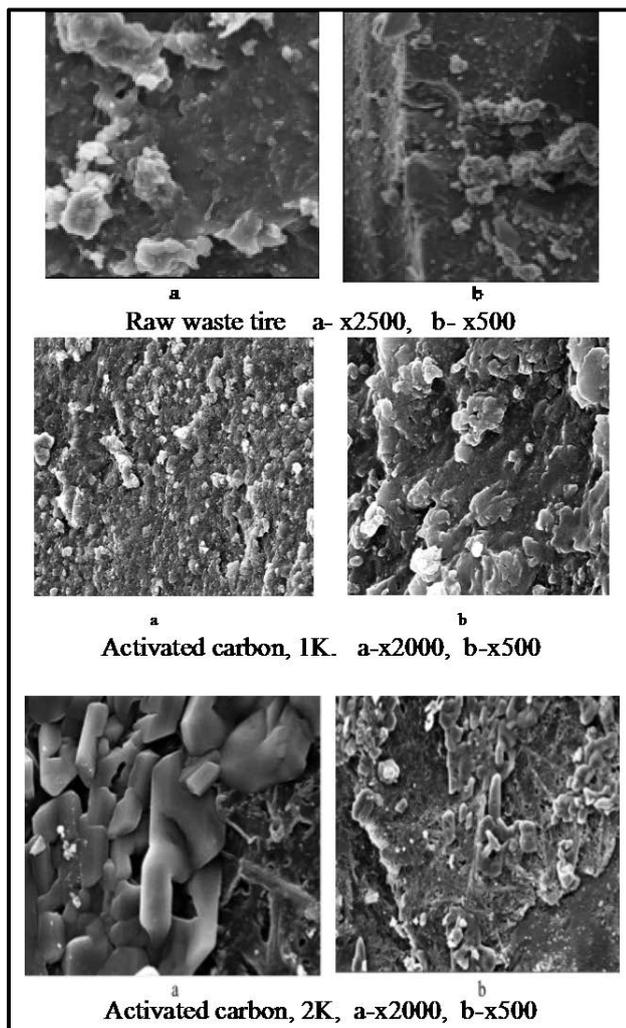


Fig. 1. SEM images of raw waste tire and obtained activated carbon samples.

3.2. Adsorption studies

3.2.1. Effect of pH on MB Adsorption

Adsorption efficiency is significantly affected by initial pH. The effect of pH on color removal was investigated by using 1-K activated carbon at pH values of 4.5, 5, 6, 6.5, 7 and 9. In order to determine the optimum pH, adsorption was carried out with 2 g/200 ml activated carbon, stirring rate of 200 rpm, temperature of 20°C, initial concentration of 20 mg/L, and agitation time of 2 h. The resulting MB removal efficiency and the change of adsorption capacity versus time for different pH values are presented in Fig. 2.

In the adsorption study with 1-K activated carbon, it was observed that the highest adsorption efficiency was obtained at pH 6.5, which was the original pH of the solution. Since change in pH of the solution causes structural changes of the adsorbate molecules, decrease in pH value decreases the efficiency of removal. Similar results are found in the literature [22].

In studies conducted for different pH values, change in adsorption capacity (q_e) values was the highest in pH 6.5. For pH 6.5, the q_e values ranged from 0.563 to 1.856 mg/g (Fig. 2b).

3.2.2. Effect of adsorbent dose

First, 1-K activated carbon and 0.5–2.5 g/200 ml were studied as adsorbent doses. Then, results obtained from this study were evaluated, and the effect of adsorbent dose was investigated using adsorbent doses of 0.5, 1.5 and 2 g/200 ml (pH 6.5°C and 20°C) for 2-K activated carbon. Variation of time-dependent MB removal efficiency for different 1-K adsorbent doses is given in Fig. 3a. The time dependent variation of adsorption capacity is given in Fig. 3b. Changes of removal efficiencies and adsorption capacities for 2-K adsorbents were presented in Figs. 4a and b.

For the 1-K adsorbent, it was observed that the best removal efficiency was obtained 2 g/200 ml adsorbent dose. The lowest removal efficiency in Fig. 3a was 21% for

Table 2
BET analysis results for 1-K and 2-K adsorbents

Sample	BET 1-K Adsorbent (m ² /g)	BET 2-K Adsorbent (m ² /g)
After chemical activation	2.867	2.945
After chemical + thermal activation	36.268	42.61
Raw waste tire		1.349

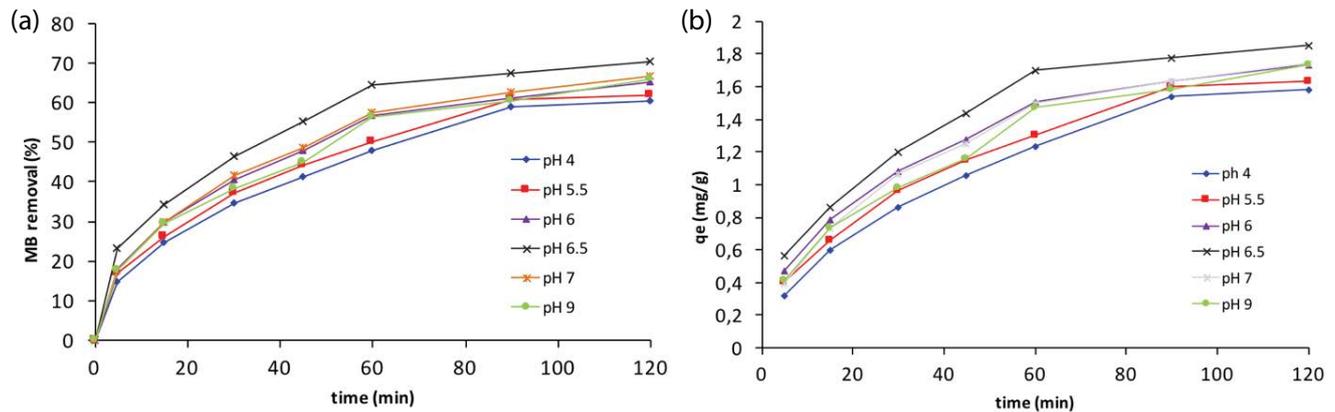


Fig. 2. (a) Change of MM removal over time for different pH values and (b) change of adsorption capacity against time for different pH values. ($T = 20^\circ\text{C}$, $C_0 = 20 \text{ mg/L}$, stirring rate = 200 rpm, 2 gr/200 ml 1-K adsorbent).

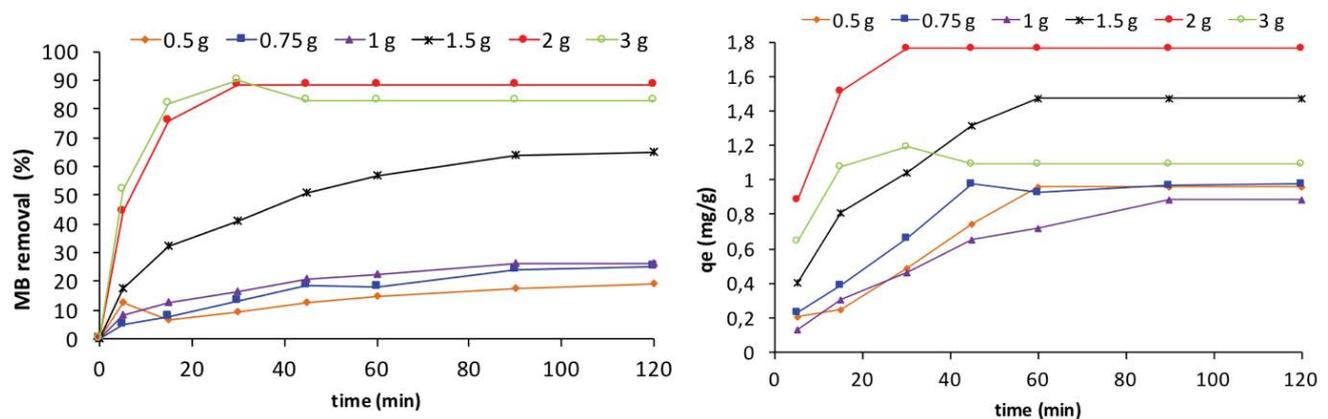


Fig. 3. (a) Change of MM removal over time for different doses of adsorbent with 1-K adsorbent and (b) variation of adsorption capacity versus time for different adsorbent doses with 1-K adsorbent. ($T = 20^\circ\text{C}$, $C_0 = 20 \text{ mg/L}$, 200 rpm).

0.5 g/200 ml. Adsorption capacity change for 2 g/200 ml dose appeared to be 1.8 mg/g (Fig. 3b). For the 2-K adsorbent, while the best removal efficiency was 87% for 2 g/200 ml dose, the lowest removal efficiency was 80% for 0.5 g/200 ml dose (Fig. 4a). The adsorption capacity change for 2 g/200 ml adsorbent was calculated as 1.74 mg/g (Fig. 4b).

3.2.3. Effect of temperature

In the adsorption experiments with 1-K and 2-K adsorbents, 10°C, 20°C, 30°C and 40°C constant temperatures were used to determine the effect of temperature on adsorption capacity. The MB removal efficiencies obtained for different

temperature values are shown in Fig. 5a, while the adsorption capacity change (q_e) values are given in Fig. 5b. Changes in adsorption efficiency on the 2-K adsorbent by time, and the change in adsorption capacity were given in Figs. 6a and b, respectively.

Temperature experiments revealed that for both 1-K and 2-K adsorbents removal efficiency decreased as the temperature decreased. For 1-K adsorbent, the lowest removal efficiency was 88% at 10°C and the highest removal efficiency was calculated as 91% at 30°C (Fig. 5a). Similarly for 2-K adsorbent, the lowest removal efficiency was calculated as 84% at 10°C temperature and the highest removal efficiency was calculated as 90% at 30°C (Fig. 6a). As seen in Figs. 5b and 6b, the highest q_e values were achieved at 30°C.

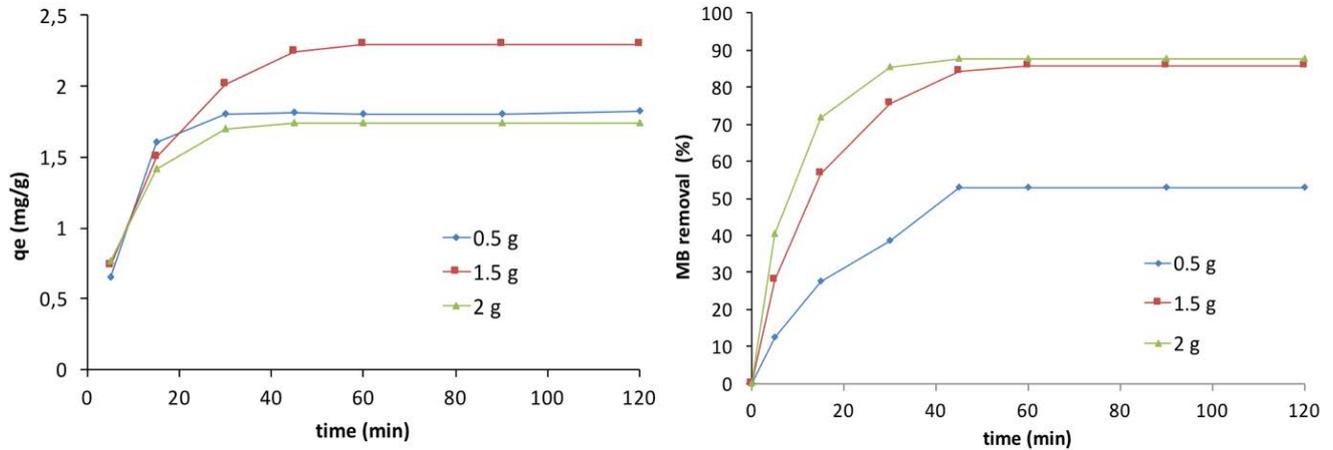


Fig. 4. (a) Change of MM removal over time for different doses of adsorbent with 2-K adsorbent and (b) variation of adsorption capacity versus time for different adsorbent doses with 2-K adsorbent. ($T = 20^\circ\text{C}$, $C_0 = 20 \text{ mg/L}$, 200 rpm).

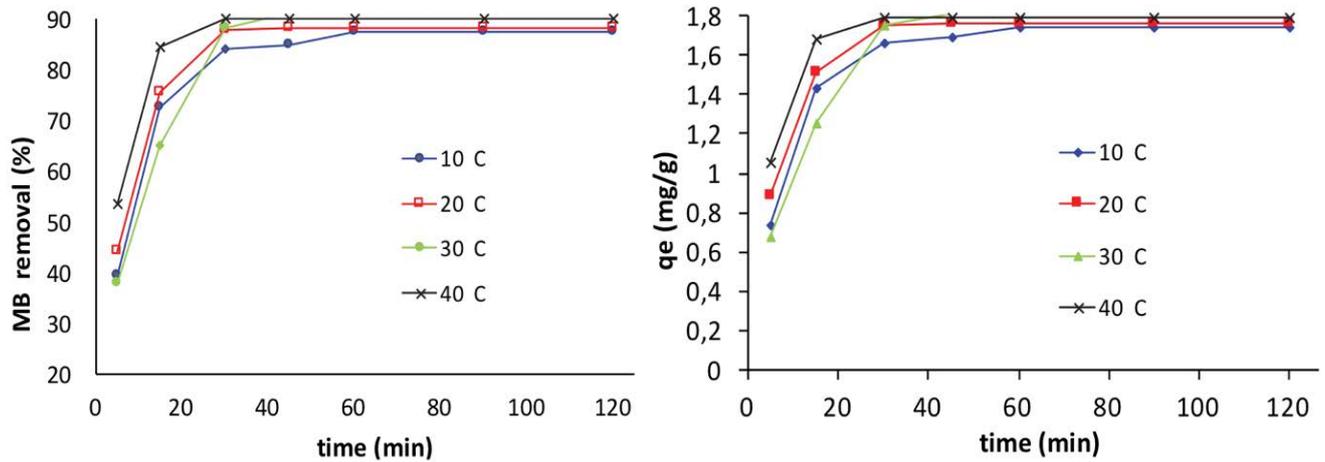


Fig. 5. (a) MM removal efficiencies obtained for different temperature values when 1-K adsorbent is used and (b) change of adsorption capacity against time for different temperature values ($\text{pH} = 6.5$, $C_0 = 20 \text{ mg/L}$, 200 rpm).

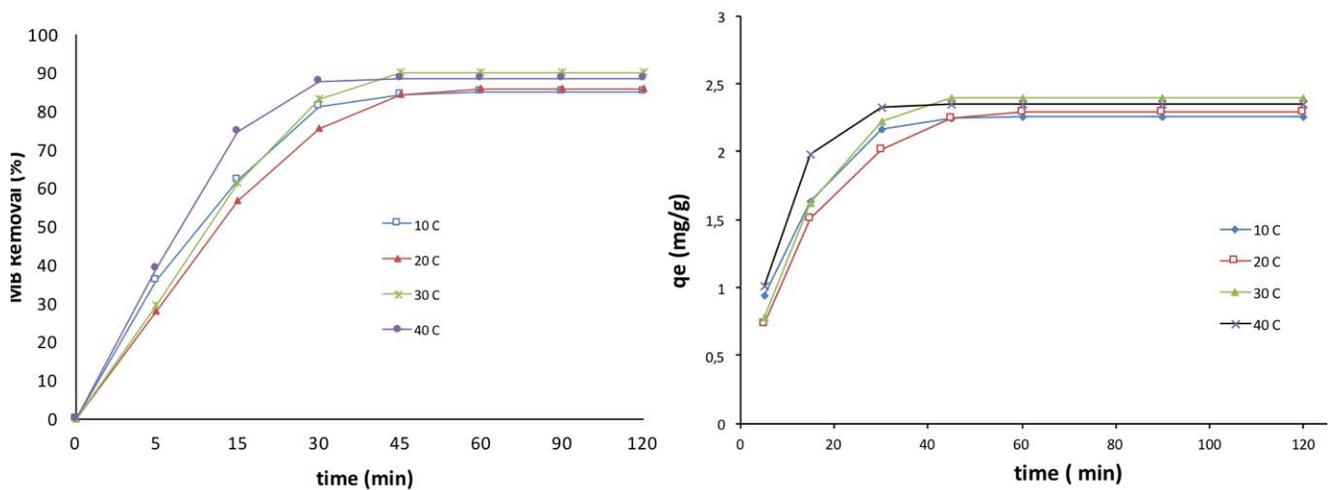


Fig. 6. (a) MM removal efficiencies obtained for different temperature values when 2-K adsorbent is used and (b) change of adsorption capacity against time for different temperature values ($\text{pH} = 6.5$, $C_0 = 20 \text{ mg/L}$, 200 rpm).

For the 1-K adsorbent, as shown in Fig. 5a, MB was adsorbed to a large extent in the first 20 min. No significant change was observed in the MB concentration after 30 min. In Fig. 6a, it is observed that in the first 30 min for 2-K adsorbent, the MB dyestuff was substantially removed from the solution. No significant change was observed in the removal of MB after 45 min, especially for all temperature values.

3.2.4. Adsorption isotherms

The data obtained from adsorption experiments were applied to Freundlich, Langmuir and Temkin isotherms. Calculated isotherm constants are given in Table 3.

When the R^2 values obtained from the isotherms were evaluated, it was observed that the adsorption for 1-K adsorbent could be expressed by Langmuir isotherm ($R^2 = 0.95$). This is thought to be due to homogeneous distribution of active parts on the adsorbent surface. The q_m (maximum adsorption capacity) value calculated according to the Langmuir isotherm was found to be 3.65 mg/g for 1-K adsorbent. The constant adsorption coefficient (R_L) value was calculated using the adsorption constant, and K values are given in

Table 3. The R_L constant had a value between 0 and 1, indicating the availability of adsorption [11]. At the end of the calculations, the R_L constant was calculated as 0.15, hence within the expected range. This indicated that adsorption was favorable.

As seen from Table 3, the adsorption mechanism of MB on 2-K adsorbent was compatible with Freundlich ($R^2 = 0.96$) and Temkin ($R^2 = 0.97$) isotherms. In the Freundlich isotherm for the 2-K adsorbent, the n -value was found to be 1.04. This indicated that physical adsorption was more effective than chemical adsorption. It refers to weak adsorption bonds due to Van Der Waals forces [23].

3.2.5. Adsorption kinetics

Pseudo-first-order and pseudo-second-order kinetic models have been applied to understand the kinetics of MB adsorption by using ELT derived activated carbons. Kinetic parameters are given in Tables 4 and 5.

The correlation coefficient (R^2) values calculated for the pseudo-second-order kinetic model were found to be between 0.98 and 0.998 for the 1-K adsorbent, and between

Table 3
Freundlich, Langmuir, Temkin Isotherm constants for 1-K and 2-K adsorbents

	Freundlich Isotherm			Langmuir Isotherm			Temkin Isotherm		
	K_f (mg/g)	n (1/n)	R^2	q_m (mg/g)	K (L/g)	R^2	K_t (L/g)	B	R^2
1-K	0.98	2.29 (0.43)	0.64	3.65	0.28	0.95	1.52	2.10	0.83
2-K	2.01	1.04 (0.95)	0.96	34.24	0.01	0.13	1.33	1.97	0.97

Table 4
Kinetic constants obtained for 1-K adsorbent

C_0 (mg/L)	Pseudo-first-order				Pseudo-second-order			
	R^2	k_1 (1/min)	q_e (mg/g)	q_{theo} (mg/g)	R^2	k_2 (g/mg min)	q_e (mg/g)	q_{theo} (mg/g)
5	0.858	0.07	0.219	0.219	0.998	4.02	0.219	0.216
10	0.785	0.03	0.722	0.153	0.999	1.16	0.722	0.732
15	0.715	0.03	1.224	0.672	0.998	0.48	1.22	1.243
20	0.812	0.06	1.771	0.221	0.998	0.27	1.77	1.809
30	0.937	0.07	2.723	0.530	0.998	0.18	2.72	2.782
50	0.841	0.05	4.521	5.514	0.98	0.01	4.52	4.868

Table 5
Kinetic constants obtained for 2-K adsorbent

C_0 (mg/L)	Pseudo-first-order				Pseudo-second-order			
	R^2	k_1 (1/min)	q_e (mg/g)	q_{theo} (mg/g)	R^2	k_2 (g/mg min)	q_e (mg/g)	q_{theo} (mg/g)
5	0.730	0.066	0.306	0.07	0.997	1.026	0.306	0.462
10	0.575	0.04	0.7	0.159	0.999	1.129	0.7	0.966
15	0.569	0.037	1.25	0.211	0.999	1.150	1.25	1.491
20	0.712	0.045	1.82	0.404	0.999	1.069	1.82	2.058
30	0.747	0.055	2.72	0.66	0.999	0.939	2.72	3.106
50	0.383	0.037	4.31	0.76	1	1.604	4.31	4.992

Table 6
Thermodynamic parameters

Temperature (K)	K_c	ΔG° (kJ/mol)	ΔH° (kJ/mol)	ΔS° (kJ/molK)
283	3.78	-30.88		
293	3.82	-32.19	-7.0176	0.0853
303	3.85	-33.54		
313	3.65	-33.21		

0.997 and 1 for the 2-K adsorbent (Tables 4 and 5). Since the R^2 values were close to 1, and the q_e and q_{theo} values were closer to each other, the second order kinetic model was suitable for both 1-K and 2-K adsorbents. It could be said that chemisorption was the rate limiting step for this study. When the literature was examined; the kinetic results obtained from this study were similar to studies of dye removal from wastewater [5,23–27].

3.2.6. Adsorption thermodynamics

In terms of applicability of the process, it was an important stage to examine the adsorption process from a thermodynamic point of view [28]. As the best removal efficiency was obtained in tests with 2-K activated carbon, thermodynamic studies were carried out using the results obtained from those. Calculated Gibbs free energy (ΔG°), enthalpy (ΔH°) and entropy (ΔS°) values are given in Table 6.

For thermodynamic calculations, K_c equilibrium constants were calculated for temperatures of 10°C, 20°C, 30°C and 40°C (283, 293, 303, 313 K). According to results in Table 6, Gibbs free energy (ΔG°) values obtained for all tested temperatures were negative. The negative value of ΔG° indicated that adsorption of MB dye on activated carbon was thermodynamically favorable and proceeded without the need for any external energy [27,29]. The increase of ΔG° value with temperature increase indicated that applicability for high temperatures would increase, and that the adsorption process was endothermic. In the literature, similar results were observed in the studies on removal of MB adsorption, Astrazon Blue FGRL with rice husk [30,31].

Enthalpy value (ΔH°) was calculated as -7.0176 kJ/mol (Table 6). Negative value of standard enthalpy change showed that the adsorption was exothermic and stable [32,33]. Entropy value (ΔS°) was calculated as 0.0853 kJ/mol (Table 6). The positive entropy indicated an increase in irregularity at the solution-adsorbent interface during adsorption. It could be argued that positive entropy change was the result of the adsorbent's tendency to adsorb pollutant. Similar results were observed in the literature [34,35].

4. Discussion and conclusions

Considering the high production rate of ELT, one billion per year approximately, efforts have been put in to valorizing this high energy, high carbon resource. However, around 30% of ELT is still known to end up at landfills at best [36]. In this study, we focused on the material recovery option for ELT and upcycled it to produce an affordable and efficient adsorbent.

Prior to any treatment, surface area of raw ELT was small (1.349 m²/g). At the end of chemical activation with KOH, when the BET values were examined, it was observed that there was no significant change in surface area. Surface area measurement (BET) and SEM images showed that pores of 1-K and 2-K activated carbons increased after thermochemical treatment. Results of elemental analysis revealed that carbon content of produced activated carbons decreased compared to that of raw ELT sample. It was thought to happen due to increase in the pore structure of activated carbons.

MB removal adsorption studies were conducted with different adsorbent amounts between 2.5–15 g/L for 1-K and 2-K adsorbents. The original pH of the solution was 6.5, and the temperature was 20°C; and when the adsorbent dose of 10 g/L was used for the 1-K adsorbent, the adsorption capacity was found to be the maximum. Under same conditions, maximum adsorption capacity was achieved with adsorbent dose of 7.5 g/L for the 2-K adsorbent.

When pH-related MB removal efficiencies were examined, pH 6.5 was found to be the optimum pH. As the electrostatic force between the cationic MB and the solid surface increased, the rise in pH increased MB adsorption. It is thought that over pH 6.5 the structure of dye has started to degrade and therefore a slight decrease in yield was observed.

To determine the effect of temperature in removal of MB, 10°C–40°C temperature values were tested. Regardless of the adsorbent dose, removal of MB increased with temperature. The highest removal efficiency was observed at 30°C. After 30°C, as the temperature value increased, there was no significant increase in removal efficiency. Temperature dependent increase in adsorption efficiency indicated that adsorption mechanism was an endothermic reaction. And also the calculated negative values for Gibbs free energy change (ΔG°) supported this finding.

Data obtained from batch studies were applied to Langmuir, Freundlich and Temkin isotherms. Results for 1-K adsorbent was observed to be compatible with the Langmuir isotherm ($R^2 = 0.95$), and the studies with 2-K adsorbent were compatible with Freundlich ($R^2 = 0.9654$) and Temkin isotherm models ($R^2 = 0.97$).

When the adsorption thermodynamics were analyzed, Gibbs free energy change (ΔG°) was negative. The positive value of entropy change ($\Delta S^\circ = 0.0853$ kJ/mol K) indicated that there was an interaction between adsorbent and adsorbate. It showed that the irregularity between adsorbent-adsorbed interface increased, and that there might be a structural change between adsorbent and dyestuff. At the end of thermodynamic studies, the negative value of enthalpy ($\Delta H^\circ = -7.0176$ kJ/mol) indicated that the adsorption system was exothermic.

The procurement of a material, which can be produced with different properties, increased the importance of the tires that had completed their life. Since the liquid and gas products, obtained after thermal activation, were products containing high energy, each product could be utilized. In the adsorption tests with activated carbon, MB dye removal was achieved with high efficiency. When the results of the study were evaluated, it was observed that activated carbon, obtained by thermochemical processes from waste tires,

is a valuable raw material which might be used at industrial levels as an alternative to commercial activated carbon.

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