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A Novel Biochar Approach: Valorization of Wildfire Residual for The Removal of Cationic Dye from Textile Wastewater

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Abstract

Objective: The main objective of this study was to reveal the cationic dye (methylene blue) removal performance of activated biochar materials produced from the pine tree (*Pinus brutia*) wildfire remnants from 2024 Çanakkale Wildfire area.

Materials and Methods: The pine tree (*Pinus brutia*) wildfire remnant samples were collected from the 2024 Çanakkale Wildfire area. Boric acid and Sodium Hydroxide were used as activation agents on the samples. Then, methylene blue adsorption equilibrium and kinetic studies were conducted to reveal the adsorption performance of the produced biochar adsorbent. The adsorption capacity, adsorption isotherms, and kinetic study experiments were conducted.

Results: The results showed that the Langmuir adsorption isotherm fitted very well with the Sodium Hydroxide-activated biochar samples for the methylene blue adsorption equilibrium experiments. The Methylene Blue Removal percentage of % 96 was reached. The kinetic studies revealed that the pseudo-second-order kinetic model fitted the experimental data well.

Conclusion: The adsorption capacity and percent methylene blue removal performances indicated that the produced activated carbons have competitive adsorption potentials in relation to the literature data.

Keywords Wildfire · biochar · methylene blue · activated carbon · adsorption

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Introduction

Wildfires are natural events that play a complex role in shaping ecosystems worldwide. Despite some known benefits, such as contributing to the ecological balance by supporting species diversity and nutrient cycling, wildfires pose significant environmental concerns due to their increasing frequency and intensity, which are intensified by climate change. The negative effects of wildfires include the release of significant amounts of carbon dioxide, black carbon, and other greenhouse gases into the atmosphere, contributing to global warming. For example, wildfires in Canada in 2023 were estimated to have emitted approximately 2 billion tonnes of carbon dioxide, accounting for almost a quarter of the global wildfire emissions that year (Byrne *et al.*, 2024; Gomez Isaza *et al.*, 2022). Beyond atmospheric effects, wildfires are also known to cause soil degradation, loss of biodiversity and pollution of water resources. The burning of vegetation, especially organic matter, changes the soil structure, reduces fertility, and increases the potential for erosion. Ash and debris from forest fires can leak into water bodies and threaten aquatic life (Gomez Isaza *et al.*, 2022). There has been a significant increase in the number and severity of forest fires in Turkey in the last decade. It has been reported that 16,003 forest fires occurred in Türkiye between 2015 and 2020, destroying approximately 62,315 hectares of forestland. In particular, in 2021, more than 200 forest fires were reported to have burned approximately 1,600 square kilometers of forest in the Mediterranean region, which was stated as the worst forest fire season in Turkish history (Orman Mühendisleri Odası Yönetim Kurulu, 2024). The severity of the situation continued in 2024, with approximately 120,000 hectares of land lost in forest fires, more than four times the figure recorded in the previous year. These fires not only destroyed large areas of forests but also led to the loss of biodiversity and significant economic consequences.

The textile industry is one of the largest consumers of dyes and pigments, using a wide array of chemical compounds to achieve the desired colors and patterns in fabrics. While essential for esthetic and functional purposes, these substances pose significant environmental challenges when released into wastewater without proper treatment. Textile wastewater often contains high concentrations of synthetic dyes with complex aromatic structures resistant to biodegradation. The presence of these dyes in aquatic ecosystems inhibits photosynthesis as they form a barrier to sunlight and cause decreased oxygen levels in the water. Some dyes have been identified as toxic, carcinogenic or mutagenic, posing a risk to aquatic life and potentially entering the human food chain through bioaccumulation (Tkaczyk *et al.*, 2020). Therefore, wastewater containing textile dyes must be effectively treated.

The dye methylene blue is commonly used in the textile industry to color cotton and other fabrics (Khan *et al.*, 2022). However, the use of methylene blue without treatment can lead to environmental and health issues (Oladoye *et al.*, 2022). Even low concentrations of methylene blue can change the color of water, noticeably affecting its appearance and clarity. This change can negatively affect the growth of plants by reducing sunlight penetration. Furthermore, methylene blue has been linked to human health problems such as increased heart rate, vomiting, and blueness of the skin with contact. Hence, it is crucial to eliminate methylene blue from textile wastewater to protect both the environment and public health (Khan *et al.*, 2022; Oladoye *et al.*, 2022).

Adsorption is a well-known and efficient technique for eliminating charged dyes from wastewater systems by binding dye molecules onto solid material surfaces (adsorbents) to separate them from the water phase. Activated carbon is also one of the most commonly used adsorbents due to its high surface area and porous structure, which attract organic molecules with a strong affinity. Nevertheless, the production of activated carbon can pose environmental challenges. Researchers have been focusing on finding environmentally friendly and cheap adsorbents. For this purpose, agricultural waste products and natural minerals are being studied to produce adsorbents for cationic dye removal from wastewater. While these alternative starting materials offer cost benefits, they also contribute to waste valorization and environmental sustainability.

For the production of activated carbon, raw materials containing high carbon (40-80%) and low inorganic matter are preferred. These raw materials are subjected to some preliminary processes such as washing, grinding, and purification. After these pre-treatments, the carbonization process is carried out in an oxygen-free environment at 500 to 950°C temperatures (Tan *et al.*, 2017). In the carbonization step, the moisture content in the raw mate-

rial decreases and the volatile components are separated while increasing the carbon rate. The cross-links are connected and a porous hard structure can be formed due to the high temperature. The activation process, on the other hand, is a chemical change of the activated carbon surface depending on the area of use (Supanchaiyamat *et al.*, 2019). Two types of activation are applied to activated carbon: physical and chemical. In the physical activation process, oxidizers such as water vapor and carbon dioxide are mostly used. Acid, alkali, and oxidation treatments were applied to activate the biochar, which showed significant improvement in the physicochemical properties of the biochar. The commonly used acids are HCl, HNO_3, H_2SO_4, and H_3PO_4, while the bases are KOH, NaOH, and K_2CO_3 for the chemical activation process. The acid treatment process could improve the pore properties of the biochar, including the surface area and porosity, by removing the impurities of the biochar surface. The volatile substances formed because of the carbon combustion process interact with oxygen and move away from the area where the combustion process takes place (Suárez-Ruiz & Crelling, 2008). According to the structure of the chemicals used in the activation process, the cleaning process is carried out using deionized water to remove impurities, followed by the drying. The choice of precursor material and activation method significantly influences the properties of the resulting activated carbon, including pore size distribution, surface area, and adsorption capacity (Suárez-Ruiz & Crelling, 2008). Optimizing these parameters is crucial for tailoring activated carbon for specific applications, such as the removal of cationic dyes from wastewater.

After the wildfires, significant amounts of partially charred biomass are generated, including carbonized but incompletely burned wood residues. Traditionally, these residues have been considered waste. However, these wildfire residues may be valuable precursors for biochar or activated carbon production. This activated biochar might be used for soil amendment, carbon sequestration, and wastewater treatment applications (Ge *et al.*, 2023).

The Carbonization step is the most energy-intense and CO_2-emitting step of the activated carbon production. The intense heat from the wildfire and the high concentrations of CO_2 in the burning area cause the organic material to carbonize by itself, which is the first step of biochar production. Hence, the required heat and oxygen-free atmosphere for the regular carbonization step can be supplied by the wildfires. The resulting self-carbonized biochar also helps to capture carbon that would otherwise be emitted to the atmosphere and reduces the risk of future wildfires by minimizing the available fuel for new fires. Hence, producing biochar from wildfire residuals is a win-win strategy that supports wildfire recovery efforts by lowering the amount of flammable material and creating a high-carbon product. Biochar made from leftover materials after wildfires have lots of potential beyond that. Because of its unique porous structure and high surface area, it can be a powerful tool for cleaning up various pollutants, including cationic dyes like methylene blue, which are often found in textile wastewater.

Studies have shown that biochar created from lignocellulosic biomass is particularly good at the adsorption of methylene blue, but its success depends on factors like its surface area, pore volume, and surface chemistry (Ying *et al.*, 2021). In short, transforming wildfire leftovers into biochar offers an exciting way to promote environmental sustainability. This method not only helps in managing post-wildfire debris and reducing the risk of future fires but also provides an economical solution for cleaning up wastewater polluted with dyes. Future research focused on fine-tuning the pyrolysis process and gaining deeper insights into how biochar interacts with pollutants will be key to unlocking its full potential in environmental clean-up efforts.

In this study, the pine tree (*Pinus brutia*) wildfire remnants from 2024 Çanakkale Wildfire area were collected and used as activated carbon precursors such as biochar. Then, the cationic dye (methylene blue) removal performance of the activated biochar materials was revealed.

Materials and Methods

Material Collection and Pretreatment

The carbonized biochar samples were collected from the 2024 Çanakkale Wildfire area. They were first ground with a mortar and pestle until they became a fine powder. The samples were placed on filter paper and left to dry at 100℃ for 24 h. The dried samples were then used as the starting material for the production of activated carbon. Both acid activation and base activation methods were applied to the samples.

Acid Activation

For the acid activation process, 15 g of the samples were taken and mixed with 5 g of boric acid [(H_3BO_3) 99.8% purity] solution in a 100 ml volumetric flask at room temperature with a heated magnetic stirrer until a homogeneous mixture was obtained. The amount of activation agent was decided on the basis of the solubility information of the H_3BO_3 in water at room temperature (Muñoz *et al.*, 2003; Searles Valley Minerals, 2020). The molarity of the boric acid solution was calculated as 0.8 M. The samples were kept in an oven at 110°C for 20 h to dry. To remove the acidity of the samples, the samples taken from the oven were washed with 0.01 M NaOH until the pH of the washing water reached 6.5-7.

Base Activation

For the base activation, 15 g of the dried samples were taken and activated with 2.25 M NaOH solution by soaking the samples in the base solution and stirring for 30 min until a homogeneous mixture was obtained. The ratio of the sodium hydroxide concentration to the sample amount used for the base activation was decided using the preliminary study results and literature information (Cazetta *et al.*, 2011). The samples were kept in an oven at 110°C for 20 h to dry. In order to remove the alkalinity of the samples, the samples taken from the oven were washed with 0.01 M HCl until the pH of the washing water reached 6.5-7.

After the activation process, all samples were left to dry in an oven at 110 C overnight. The surface morphology of the wildfire remnant materials and activated biochar was analyzed using Fourier Transform Infrared (FTIR) spectroscopy. FTIR spectra were acquired with a Cary 630 FTIR Spectrometer (Agilent Technologies), which was equipped with an attenuated total reflectance (ATR) module. The spectral data were collected over a wavenumber range of 4000–650 cm^{-1}. To improve the signal-to-noise ratio, measurements were performed at a resolution of 4 cm^{-1}, with 16 scans applied to each sample.

Adsorption experiments

10 mg of the activated samples were added to 100 mL of methylene blue solutions of different concentrations (2 to 10 ppm) and mixed in a magnetic stirrer at approximately 150 rpm. Samples taken from the methylene blue solutions every hour were analyzed using a UV-Vis spectrophotometer at a wavelength of 668 nm. All absorbance data were measured three times and averaged to eliminate possible measurement errors. The adsorption equilibrium capacity Q_{eq} (mg/g) and removal efficiency (% R) of MB on the adsorbent were calculated via the following equations:

$$Q_{eq}\left(\frac{mg}{g}\right) = \frac{(C_0 - C)V}{M} \quad (1)$$

$$\% R = \frac{C_0 - C}{C_0} \times 100 \quad (2)$$

where Q_{eq} (mg/g) is the amount of Methylene Blue adsorbed on the adsorbent, C_o (mg/L) and C (mg/L) are the initial concentration and the equilibrium concentration of Methylene Blue, respectively, V (L) is the volume of the solution, M (g) is the mass of the adsorbent used, and % R is the removal efficiency of the Methylene Blue.

The adsorption kinetics of the methylene blue/boric acid-activated system were calculated using Equation 3 for the pseudo-first-order kinetic model and Equation 4 for the pseudo-second-order kinetic model.

$$\ln(q_e - q_t) = lnq_e - k_1 t \quad (3)$$

$$\frac{t}{qt} = \frac{1}{qe}t + \frac{1}{k_2 q_e^2} \quad (4)$$

Where q_e is the amount of adsorbed dye per unit adsorbent at equilibrium (mg/g), q_t is the amount of adsorbed dye per unit adsorbent at any time (mg/g), t is the time (min), k_1 is the rate of the pseudo-1st order kinetic model (min^{-1}).

Results and Discussion

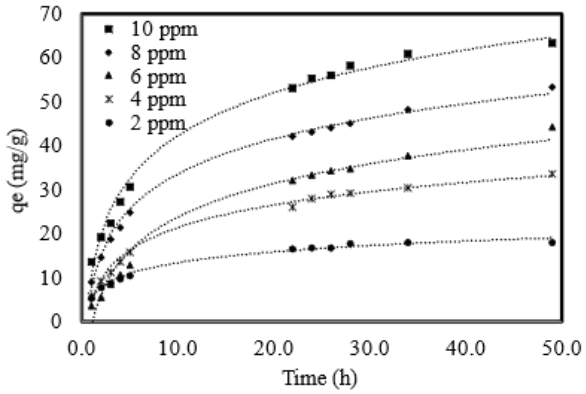
Figure 1 presents the experimental data illustrating the amount of MB adsorbed per gram of boric acid-activated adsorbent over time for five different MB solutions (2-10 ppm). Figure 1 shows that adsorption reaches equilibrium in roughly 48 h for all solutions. These experimental data were also used for the kinetic calculations.

The color removal for all samples can be clearly seen from the picture of the methylene blue solutions before and after the adsorption experiments, illustrated in Figure 2 (top) for the boric acid-activated biochar and (bottom) for the sodium hydroxide-activated biochar.

On the other hand, adsorption isotherms describe the equilibrium distribution of adsorbate molecules between a liquid and a solid phase. Evaluating the experimental isotherm data by applying appropriate models is cru-

Figure 1

The experimental data for the amount of adsorbed Methylene Blue per gram adsorbent (Boric Acid Activated) with respect to time.

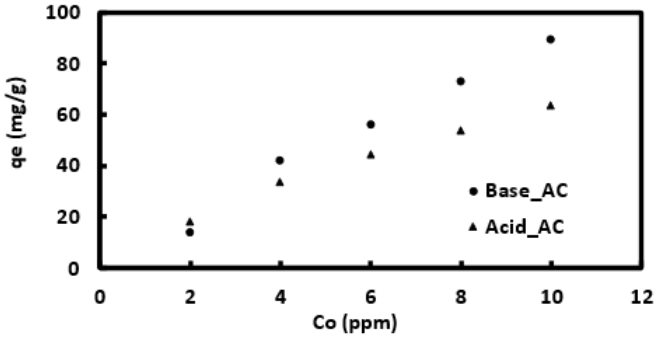


cial for determining a reliable and effective adsorption mechanism. The experimental adsorption equilibrium isotherms for the boric acid-activated and Sodium Hydroxide-activated biochar samples are illustrated in Figure 3. In this study, the linearized forms of the Langmuir and Freundlich isotherm models were used to analyze the equilibrium behavior of methylene blue on boric acid-activated carbon (Figure 4 a-b) and sodium hydroxide-activated carbon (Figure 4 c-d), aiding in optimizing the adsorption system. These models, commonly called two-parameter isotherms, provide key insights into the adsorption capacity and energy-related constants, as illustrated in Figure 4.

The removal efficiency of Methylene Blue adsorption on activated carbon is a critical parameter in evaluating the

Figure 3

The adsorption equilibrium isotherms for boric acid-activated and sodium hydroxide-activated biochar samples



effectiveness of an adsorption process. High removal efficiency indicates strong adsorptive interactions between the dye molecules and the carbon surface, influenced by factors such as pore structure, surface chemistry, and operating conditions. Optimizing this efficiency is essential for wastewater treatment applications because it directly impacts environmental sustainability and economic feasibility. Understanding the adsorption mechanisms and enhancing the removal efficiency contribute to the development of more effective and sustainable adsorbent materials for industrial and environmental applications. The Methylene Blue removal efficiencies of the prepared samples in this study were calculated based on Eqn-2. The methylene blue removal efficiency (% R) of the acid-activated and base-activated adsorbents can be seen in Table 1.

Figure 2

Picture of methylene blue solutions before and after adsorption experiments (top) for Boric Acid activated biochar, (bottom) for Sodium Hydroxide activated biochar.

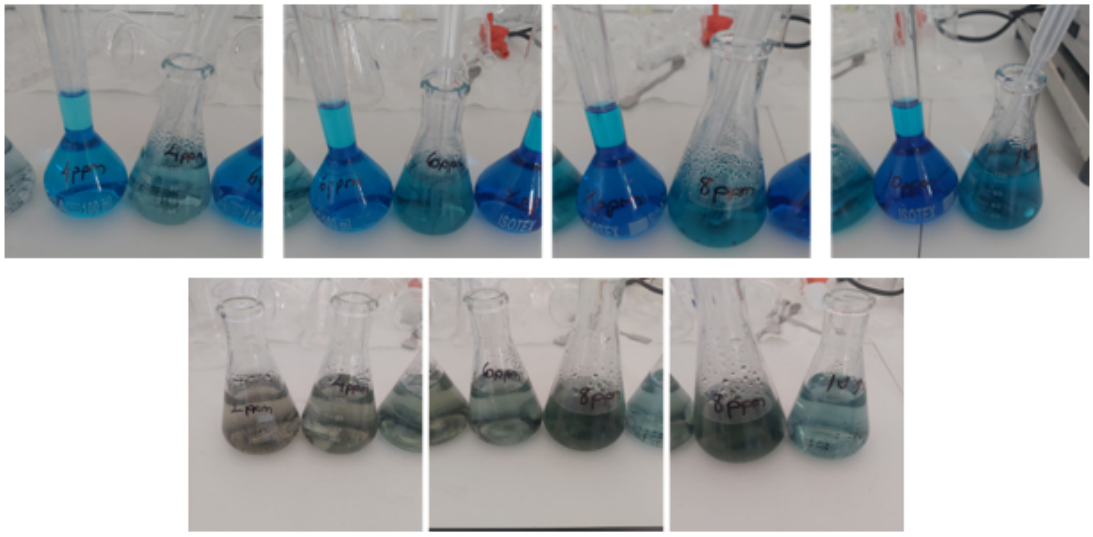


Figure 4

The Methylene Blue adsorption equilibrium isotherm fitting for the biochar samples. (a) Langmuir fitting, (b) Freundlich fitting for the boric acid-activated biochar samples. (c) Langmuir fitting, (d) Freundlich fitting for the sodium hydroxide-activated biochar samples.

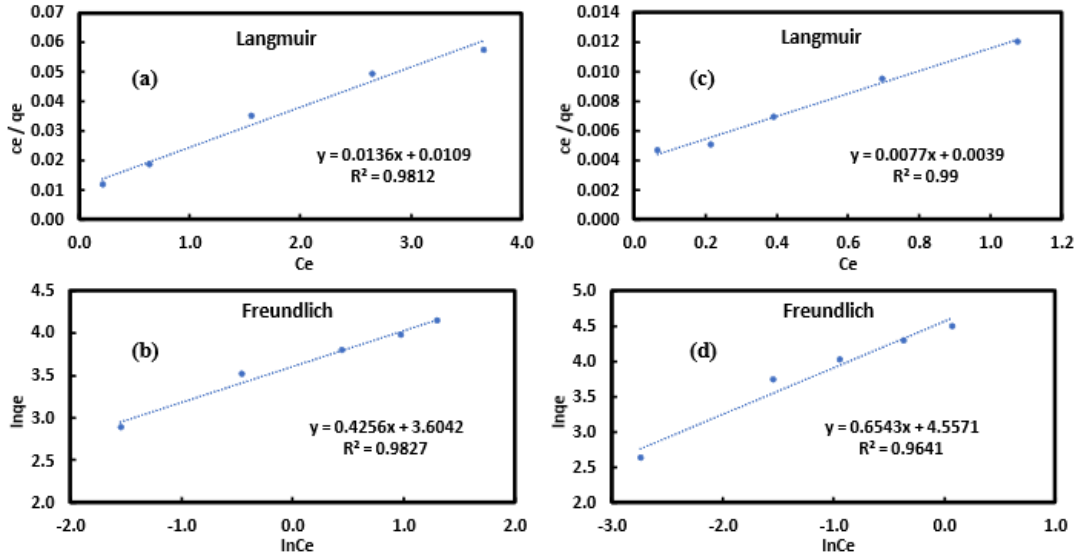


Table 1

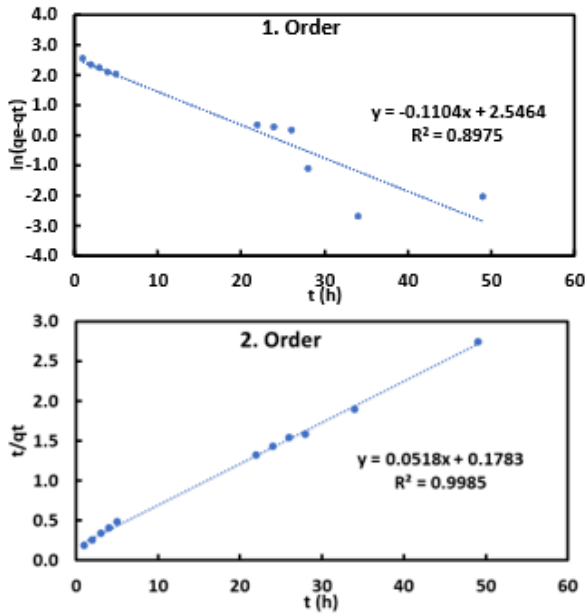
% Removal Efficiency of Boric Acid-Activated and Sodium Hydroxide activated biochar samples.

Acid Activated		Base Activated	
Co	% R	Co	% R
2	89.659	2	96.774
4	76.319	4	94.673
6	62.836	6	93.477
8	60.249	8	91.319
10	60.970	10	89.252

Kinetic studies play a crucial role in understanding the reaction dynamics, particularly in determining the order of the rate constant. Kinetic parameters provide essential insights for the design and modeling of adsorption processes. In this study, the adsorption kinetics of the boric acid–methylene blue (MB) system were evaluated using the linear fitting of two kinetic models: pseudo-first-order and pseudo-second-order, as depicted in Figure 5. The data indicate that equilibrium was achieved within 10 h for the boric acid–MB (2 ppm) system, suggesting favorable interactions between the adsorbent and adsorbate. Among the models assessed, the pseudo-second-order kinetic model exhibited the highest correlation coefficient (R^2). Furthermore, the calculated equilibrium adsorbed amount from the pseudo-second-order kinetic model ($q_{e,model}$) values (19.31 mg/g) closely matched the experimentally obtained $q_{e,experimental}$ values (17.9 mg/g).

Figure 5

Kinetic evaluation of the Methylene Blue (2 ppm) adsorption on the Boric Acid Activated biochar adsorbent.



The Methylene blue removal efficiency percentage was calculated according to the initial concentration in the activation process. Very high Methylene Blue removal efficiency percentages were achieved in both boric acid-activated and NaOH-activated biochar samples (Table 1). This result is an important indicator that a highly competitive performance was achieved compared to the examples in the literature, as shown in Table 2.

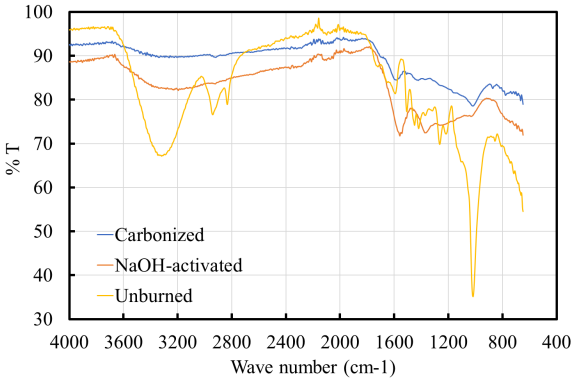
Table 2

Comparison Table for percent Removal Efficiencies and Equilibrium Adsorption Capacities

Activated Carbons	% Methylene Blue Removal Efficiency	Adsorption Capacity (mg/g)	References
NaOH-activated biochar	96.77	130.25	This study
KOH-activated *Acacia mangium*	36.47	59.92	Danish *et al.*, 2018
H_3PO_4- activated *Acacia mangium*	97.31	159.89	Danish *et al.*, 2018
HCl-activated peanut stick	Not calculated	2.54	Ghaedi *et al.*, 2014
peanut husk	Not calculated	72.13	Song *et al.*, 2011
Commercial ACs - Filtrasorb 400 - Norit - Picacarb	64 56 56	315 280 270	Raposo *et al.*, 2009

Figure 6

FT-IR spectra of unburned, carbonized, and NaOH-activated biochar samples.



The chemical stability of the biochar samples was assessed using Fourier Transform Infrared (FTIR) spectroscopy. FTIR spectra facilitated the identification of the functional groups present on the carbon surface. When the infrared radiation interacted with the adsorbent material, it induced various vibrational modes, including stretching, contraction, and bending of the chemical bonds. The resulting FTIR spectra curves are presented in Figure 6. A broad absorption band observed around 3400 cm^{-1} was associated with surface hydroxyl groups, adsorbed water, and amine groups. The O–H stretching vibrations were detected over a wide frequency range, indicating the presence of both free hydroxyl groups and hydrogen-bonded O–H bands of the carboxylic acids. The presence of moisture on the sample surfaces was confirmed by these O–H vibrations. For the unburned biochar sample, the lactonic functional groups contributed to the FTIR vibrations in the 1350–1550 cm^{-1} range. However, these vibrations disappeared in the carbonized and activated samples, suggesting structural modifications. The peak at 2930 cm^{-1} was attributed to the asymmetrical stretching of the C–H bonds in the methyl groups, which are characteristic of the lignin structure. In addition, a medium-intensity peak at 2380 cm^{-1} was associated with aldehyde functional groups, likely due to H–C=O or C–H stretching vibrations. In the NaOH-activated biochar sample, two prominent peaks at 1360 cm^{-1} and 1560 cm^{-1} were identified. These were attributed to C–H vibrations in alkanes and C–C stretching in aromatic structures, respectively, highlighting the structural changes induced by activation.

Conclusion

In this study, activated biochar was produced from wildfire residuals and its potential usage in dye removal from textile wastewater was investigated. The main findings can be listed as; 1) Innovative Recycling: Wildfire remnants were successfully used as a precursor material that reduces environmental pollution and offers economic value, 2) Effect of Chemical Activation: The biochar sample that activated with sodium hydroxide reached the highest capacity in methylene blue removal. Samples activated with boric acid also showed strong adsorption properties. 3) Isotherm models: The Langmuir and Freundlich models were examined from the adsorption isotherm models. The suitability of the Langmuir model was observed from R^2 analyzes. The Langmuir constants q_{max} and K_L were calculated as 130.25 mg/g and 1.96, respectively. 4) Kinetic Analysis: The adsorption kinetic study calculations were carried out only for the 2 ppm methylene blue solution. Because of the studies, it was revealed that the adsorption process obeyed the Pseudo-2nd Order kinetic model from R^2 analysis and that can be attributed to the dominant chemical bonding on the adsorption. The constants of the Pseudo-2nd Order kinetic model, q_e (model) and k_2 (rate constant), were calculated as 19.31 mg/g and 0.015, respectively, and it was observed that they were quite close to the experimental q_e value (17.9 mg/g). 5) % Removal Efficiency: When % Methylene blue removal was calculated according to the initial concentration in the acid activation process, it was seen that 96.77% Methylene Blue removal results were achieved in NaOH-activated samples. This result is an important indicator that a highly competitive performance was achieved compared to the examples in the literature.

As a result, activated carbon produced from forest fire waste has been proven to be an effective adsorbent for dye removal from textile wastewater. This method offers both an environmentally friendly solution and stands out as an innovative recycling approach.

Ethics Approval	No specific ethical approval was necessary, and no ethical contraventions occurred in this report.
Peer Review	Externally peer-reviewed.
Author Contributions	Conception/Design of Study- L.E., H.E.; Data Acquisition- L.E., H.E., C.A.; Data Analysis/Interpretation- L.E., H.E., C.A.; Drafting Manuscript- L.E., H.E., C.A.; Critical Revision of Manuscript- L.E., H.E.; Final Approval and Accountability- L.E., H.E., C.A.; Technical or Material Support- L.E., H.E., C.A.; Supervision- L.E., H.E.
Conflict of Interest	The authors have no conflict of interest to declare.
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