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Hydrochar Production from Cigarette Butts and Tobacco for Dye Adsorption

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ABSTRACT

In this study, tobacco waste generated from cigarette and cigar production, as well as discarded cigarette butts resulting from cigarette use, were recycled and their use as adsorbent materials was investigated. For this purpose, both products underwent various thermal and chemical treatments and were converted into hydrochar form. Chemically activated hydrochar was used in dye removal experiments due to its high surface area and adsorbent properties. Malachite green was selected as the dye material for the project. Adsorption experiments were conducted at different initial concentrations, adsorbent doses, and contact times. In experiments on the removal of malachite green with butts and tobacco waste hydrochars, removal rates as high as 99% were obtained. As a result of adsorption experiments carried out with both hydrochars, it was observed that the adsorption fits the Langmuir isotherm model and the Pseudo-Second-Order kinetic model. Tobacco waste and discarded cigarette butts, which are cheap, readily available, and abundant, were found to be effective alternative adsorbents for malachite green removal.

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1. Introduction

Water pollution occurs from anthropogenic and industrial (batteries, metallurgy, mining, nuclear, tannery, textiles, etc.) effluents and domestic sewage, mining, and recreational activities [1]. Therefore, protecting water sources and quality is highly significant because of poses a severe risk to human or environmental health. The concurrent removal of these coexisting highly hazardous pollutants is difficult due to their toxicity and persistent environmental nature [2, 3]. Adsorption, chemical precipitation, coagulation, electrodialysis, exchange, ion exchange, phytoextraction, precipitation, reverse osmosis, solvent extraction, and ultrafiltration are used to remove wastewater contaminated by dye ions. Adsorption is the most effective, low-cost, eco-friendly, and highly selective method [4].

In recent years, hydrochar has been produced from waste biomass, mainly by thermochemical conversion methods, and used as a biosorbent for organic and inorganic pollutants removal in wastewater [5].

Biomass can be used as an energy source by burning directly, or it can be converted into electrical energy, solid material (hydrochar), liquid fuel, or valuable chemicals by applying various thermo-chemical processes [6]. One of the best methods to convert biomass into different fuels is pyrolysis.

Pyrolysis is the thermal decomposition of materials at high temperatures in a limited or completely oxygen-free environment. It is usually used in the processing of organic substances [7]. Pyrolysis is a process that occurs at lower temperatures compared to gasification. Gasification is carried out at 800-1100°C, while pyrolysis is carried out at 400-700°C [8]. The inert gas used during pyrolysis is important for the removal of products from the environment. As a result of the pyrolysis process, three different forms of products are obtained; solid (biochar), liquid (biooil) and gas (syngas). Pyrolysis temperature and speed change the yield of the product obtained in different forms. Slow

pyrolysis is generally preferred for obtaining solid products, it is carried out at low temperatures and long reaction times. In obtaining solid products, 35% product efficiency is obtained with slow pyrolysis, 12% with fast pyrolysis and 5% with flash pyrolysis [9, 10]. In this study, the activation process was carried out with slow pyrolysis.

The activation process is used to increase the usability of hydrochars or biochars produced by various methods as adsorbents [11]. With chemical activation, the physico-chemical properties of the raw material are improved with acid, base and oxidation processes and their transformation into active carbon is ensured. Chemical activation processes are generally carried out by pyrolysis using HCI, HNO_3, H_2SO_4 and H_3PO_4, KOH, NaOH, K_2CO_3, H_2O_2 and $KMnO_4$ chemicals [5, 12]. With chemical activation, generally cross-linked surface functional groups and activated carbons with high specific surface area and pores are obtained. It is known that the surface porosity and functional groups of activated carbons produced with alkaline chemical activation agents such as KOH, NaOH and K_2CO_3 increase [13]. In this study, activation was carried out with KOH agent.

Cigarettes represent one of the most popular tobacco products being consumed worldwide. Tobacco and tobacco smoke contains more than 7,000 chemicals, over 70 of which are carcinogenic. Some carry health risks such as heart and/or lung disease. Globally, more than five trillion discarded cigarette butter year have been produced by smokers [14]. They include ash, unburned tobacco, and smoked filter. 95% of cigarette filters are made of cellulose acetate. Smoked filters contain no tobacco. It is packed tightly together to create a filter and is a plastic slow to degrade. The cigarette butt reduces risks associated with smoking by decreasing smoke toxicants. Cigarette butts may threaten humans and wildlife, including toxic chemicals [9].

Cigarette butts are non-biodegradable waste. But we know very little knowledge about their impacts on both human health and the environment. Discarded cigarette butts contain arsenic, benzene, cadmium, ethylene oxide, formaldehyde, nicotine, and dozens of other chemicals caused by tobacco and its residues [9]. 4.5 trillion cigarette butts are not disposed of correctly every year globally, generating 1.69 billion pounds of toxic waste and releasing thousands of chemicals into the air, water, and soil [15]. In recent years, various studies have come to the fore to reduce the toxic effect of cigarette butts and re-evaluate these wastes [16].

For this study, tobacco waste and discarded cigarette butt hydrochars were synthesized. The effects of dosage, initial concentration, and contact time in removing malachite green (MG) from aqueous solution were also assessed while evaluating the solution adsorption isotherms and kinetics. Relevant adsorption results obtained from the prepared hydrochars after chemical activation indicate the utility of the low-cost recycled material.

2. Materials and Methods

2.1 Materials

Tobacco wastes were obtained from British American Tobacco, Samsun, Turkey. The waste cigarette butts were collected from a range of residential areas by our means. The filters separated from waste discarded cigarette butts by peeling off the paper on the outside. A stock aqueous solution was prepared with powdered malachite green dye. It was diluted with pure water at calculated rates to be used in experiments.

2.2. Methods

Hydrochar production

There is no standard method for hydrochar production. It is basically the carbonization process of organic matter using temperature and water pressure in a closed reactor. In this study, average experimental conditions of similar studies in the literature were used for hydrochar production, activation process and adsorption conditions [8, 13]. 10 g tobacco and filter sample with 80mL pure water (Nuve ND4 Pure Water System) (1:8 ratio) were placed in a completely closed hydroreactor with a volume of 250mL, consisting of a separate steel outer and titanium-coated inner chamber. The reactor was kept in a muffle furnace at 250 °C for 5 hours. The reactor was cooled in a bucket full of cold water, and the obtained slurry was butted. Hydrochars were dried in a drying oven (Nuve EN400 Model) at 50 – 60°C for 24 hours.

Hydrochar activation

If the solid part is to be used as a result of the pyrolysis process, slow pyrolysis conditions are applied because the solid product yield in slow pyrolysis conditions is much higher than in fast pyrolysis [13, 17]. The produced hydrochars were activated by slow pyrolysis system using Potassium hydroxide (KOH) at a ratio of 1:4 (10g hydrochars:40g KOH). Pyrolysis was carried out at 5 °C/min in a 100 mL/min nitrogen gas flow at 600°C with a holding time of 1 hour. After pyrolysis, the samples were washed with 2M hydrochloric acid (HCl) in a vacuum filtration (Rocker 400 Vacuum Pump System) and then washed with distilled water until neutral. Pyrolysis conditions were determined by taking the average values of our previous experimental study results and similar studies in the literature [17, 18].

Characterization Analysis

Some characterization analyses were carried out to determine the suitability of natural and hydrochar butts and tobacco for use as an adsorbent. For this purpose, elemental analysis (LECO-Truespec Model) was performed to determine the elements it contained, Scanning Electron Microscope (SEM) (Jeol-JSM7001F Model) to determine microstructure and morphology, and Brunauer–Emmett–

Teller (BET) (Oantachrome-Autosorb IQ2 Model) analysis to measuring the specific surface area.

Batch adsorption experiments

In order to determine the optimum adsorption conditions in the removal of MG dye with waste cigarette butts hydrochar and tobacco hydrochar adsorbents, adsorption experiments are first carried out under different conditions (at various minimum and maximum values). Batch adsorption experiments; were carried out to understand the effects of dosage (0.33 - 16.66 g/L), initial concentration (30 - 800 mg/L), and adsorption time (1-120 min). All adsorption experiments were conducted at room temperature. The samples separated from the adsorbent after adsorption were measured to determine their final concentrations in a spectrophotometer (Thermo Aquamate Model) at a wavelength of 617nm. The spectrophotometer was calibrated at a wavelength of 617 nm using standard MG aqueous solutions with concentrations of 2, 4, 8, 16, 32, and 64 mg/L. The calibration curve equation obtained was ($y = 0.1082x + 0.0408$), with a R^2 value of 0,99. The absorbance values recorded during the sample measurements were substituted for "y" in the equation, and the corresponding "x" values were calculated to determine the MG concentrations of the samples in mg/L.
All experiments were conducted in duplicate, and the average values of the results were used.

Isotherm and kinetic models

In order to understand the adsorption mechanism, adsorption experimental results are generally examined in terms of their fitting with adsorption kinetic and isotherm models. In this study, the results were applied to Langmuir, Freundlich and Dubinin-Radushkevich (D-R) isotherm models and Pseudo-first-order, Pseudo-second-order and Intraparticle kinetic models.

3. Results and Discussion

3.1 Characterization Analysis

Elemental analysis

The elemental analysis results of tobacco and butt hydrochar produced in the study are given in Table 1. Elemental analysis indicates that both samples have high carbon content. The elemental analysis results of hydrochars have values close to similar studies in the literature. 44.20-62.73% carbon, 1.49-6.96% hydrogen, 6.85-4.13% nitrogen [16, 19]. Similar results were found in different studies on the raw and hydrochar of tobacco leaf residues [18, 19].
The variation in results for the same materials may result from different conditions of the pyrolysis processes. The elemental analysis indicated the lower nitrogen compounds because the bio-oil part produced during pyrolysis has the most nitrogen compounds. Moreover, there are also losses due to volatile compounds. In the literature, studies conducted with tobacco and tobacco products indicate that the main gases released are water, CH4, hydrocarbons, CO2, CO, carbonyl and hydroxyl [14, 20].

Table 1. *Elemental analysis of tobacco and butt hydrochar*

Sample	Carbon (%)	Hydrogen (%)	Nitrogen (%)
Waste cigarette butts hydrochar	49.45	2.71	0.65
Tobacco hydrochar	33.63	2.54	1.82

Scanning electron microscope (SEM)

SEM images at x500 were used to analyze the surface morphologies of tobacco and cigarette butt hydrochar. (Fig. 1a and b). The SEM image of the butt hydrochar shows a rigid amorphous and porous structure with smaller microstructures (Fig. 1a). It was observed that the pore level

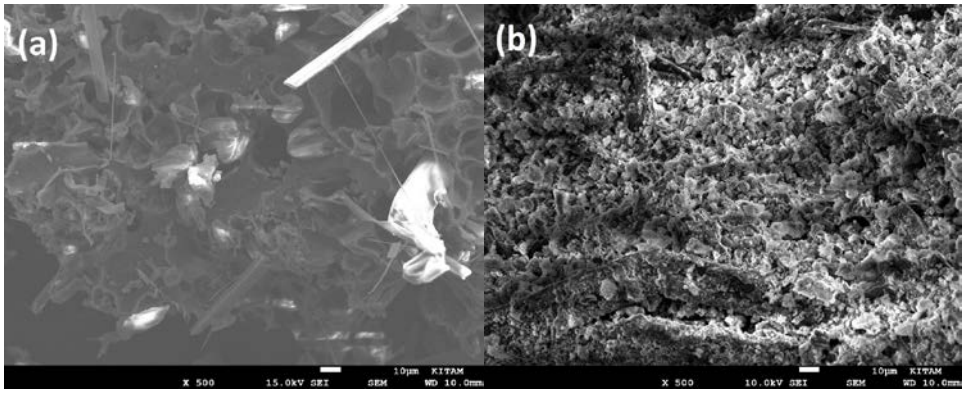


Figure 1. *SEM image of butt hydrochar (a) and tobacco hydrochar (b).*

was high in the sem images of tobacco hydrochar (Fig. 1b). The larger pores and voids are observed in the butt hydrochar compared to the tobacco hydrochar. This may be due to the collection of particles found in the smoke passing through the butt [21]. The microsphere structure of both samples given in Figure 1 shows condensation, decarboxylation, decomposition, dehydration or polymerization, and aromatization of cellulosic components during hydrothermal carbonization, as indicated in the study by Sevilla et al. [21]

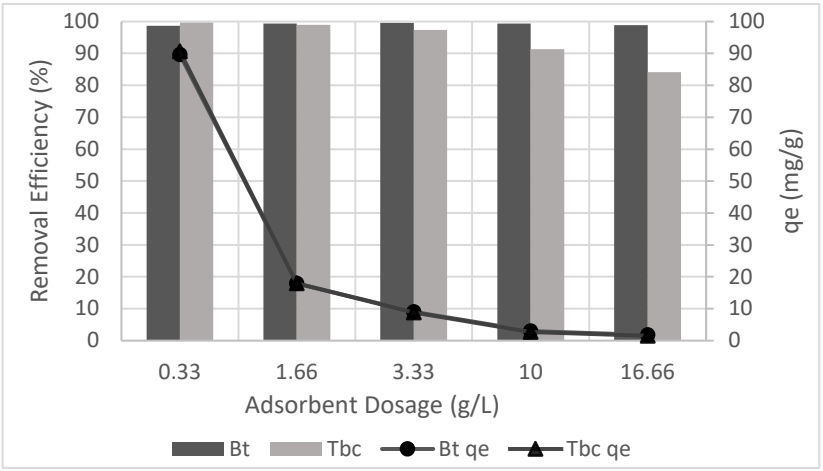
Brunauer-Emmett-Teller (BET)

The surface area analysis by BET was also performed. BET surface area measurements were performed under nitrogen gas flow at 150°C and vacuum application for 14 hours. As a result, the BET surface area for butt and tobacco hydrochar is 8.4m^2/g and 1.8m^2/g respectively. Although these results seem low for natural adsorbents, they are similar to studies conducted with the same materials in the literature [20, 22].

3.2 Batch adsorption experiments

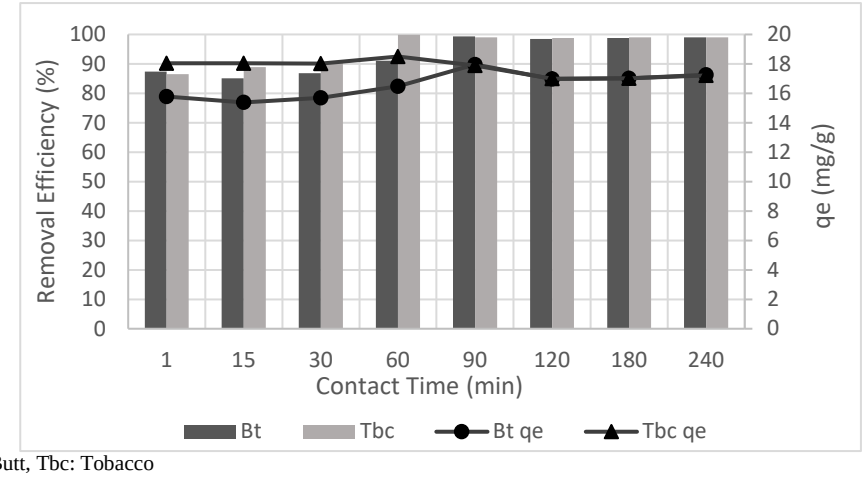
Effect of adsorbent dosage

The effect of adsorbent dose on adsorption efficiency was studied using a different amount of adsorbent (0.33, 1.66, 3.33, 9.99, 16.66 g/L). Other batch adsorption experiment conditions are 30 mL volume, 30 mg/L initial concentration, 200 rpm, 90 min. contact time. In Figure 2, malachite green



*Bt: Butt, Tbc: Tobacco

Figure 2. *Effects of adsorbent dosage for butt and tobacco hydrochars.*



*Bt: Butt, Tbc: Tobacco

Figure 3. *Effects of contact time for butt and tobacco hydrochars.*

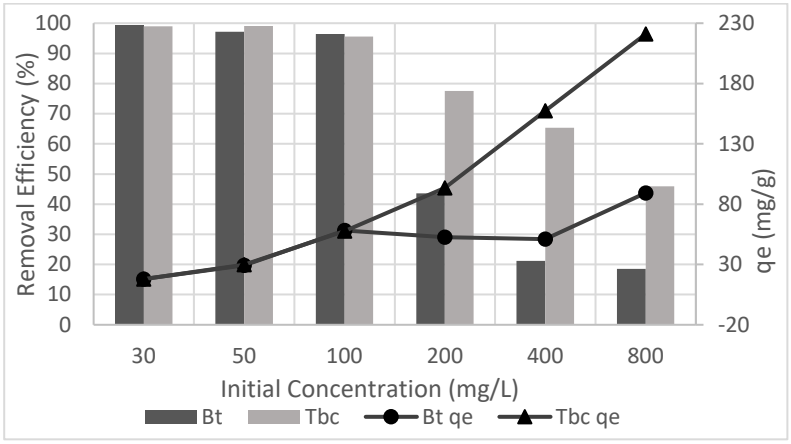
removal efficiencies and adsorption capacity (qe) values versus adsorbent dose are given.
In the Figure 2, it is seen that removal efficiency above 85% was obtained in all doses and materials. No significant change in removal efficiency was observed in the cigarette butt samples depending on the dose. In the tobacco samples, removal efficiency decreased by around 10-14% with the increase in dose. The highest removal efficiency was obtained at around 99% at 1.66g/L dose for both adsorbents.

Effect of contact time

In order to show the effect of contact time on MG removal, adsorption experiments were carried out at different contact times. For this purpose, experiments were carried out at contact times of 1, 15, 30, 60, 90, 120, 180 and 240 min. In Figure 3, malachite green removal efficiencies and adsorption capacity (qe) values versus contact time are given. In both adsorbent samples, MG removal efficiency increased with increasing contact time. It is seen that cigarette butt samples reached equilibrium after 90 minutes and tobacco samples reached equilibrium after 60 minutes. The appropriate contact time for each adsorbent was selected as 90 minutes. After 90 minutes, efficiencies were obtained around 99%.

Effect of initial concentration

To determine the effect of MG initial concentration on adsorption, experiments were carried out at different initial MG concentrations. Initial concentration studies were carried out at 30, 50, 100, 200, 400 and 800 mg/L MG concentrations. In Figure 4, MG removal efficiencies and adsorption capacity (qe) values versus initial concentration are given.
In Figure 4, a gradual decrease in removal efficiency is observed with increasing concentration for both adsorbents. This can be explained by an existing higher driving force for mass transfer from the liquid phase (adsorbate) to the solid phase (adsorbent) at the interface [23]. At high concentrations, the tobacco adsorbent is seen to have much higher removal efficiency and adsorption capacity than the cigarette butt adsorbent. At low concentrations, removal efficiency values above 95% were obtained for both adsorbents.
The K (mol²/kJ²) value calculated in the D-R isotherm is a constant that represents the energy density or free energy change associated with the adsorption process and reflects the heterogeneity of the adsorbent surface. This parameter aids in distinguishing whether the adsorption mechanism is physical or chemical. Using the K value, the average free energy (E) of the adsorption process can be determined. The E (kJ/mol) value quantifies the energy required to transfer a molecule to the adsorption site. Specifically: E <8 kJ/mol: Adsorption is typically physical, dominated by weak forces such as van der Waals interactions. E = 8–16 kJ/mol: Adsorption may involve chemical properties, such as ion exchange. E > 16 kJ/mol: Adsorption is likely driven by chemical bonding, such as covalent interactions [24]. The E values obtained from the study were determined to be 2.96kJ/mol for butt hydrochars and 1.81kJ/mol for tobacco hydrochars. These results indicate that the adsorption mechanisms for both materials can be classified as physical.



*Bt: Butt, Tbc: Tobacco

Figure 4. *Effects of initial concentration for butt and tobacco hydrochars.*

3.3 Adsorption isoterms

Table 2. *Isotherm parameters for MG adsorption onto the butt and tobacco hydrochar*

Isotherms	Parameters	*Malachite Green*	
		Butt Hydrochar	**Tobacco Hydrochar**
Langmuir	R^2	0.90	0.97
	K_L (L/mg)	0.02	0.04
	Q_m (mg/g)	83.33	227.27
Freundlich	R^2	0.73	0.97
	K_F (mg^{1-n} Lng^{-1})	29.11	32.10
	n	6.80	3.16
D-R	R^2	0,66	0,78
	K (mol^2/kJ^2)	0,0571	0,1524
	Lnqm	3,99	4,76

The isotherm of adsorption explains the relationship between the adsorbate particles adsorbed on the adsorbent surface and the equilibrium adsorbate concentration in the liquid phase [25]. Adsorption mechanisms can be examined for physical and chemical adsorption. The weak Van der Waals force between the adsorbate molecules and the adsorbent surface is effective in physical adsorption [4, 25]. Strong covalent bonds or ionic interactions are effective in chemical adsorption. So, adsorbate molecules are accumulated on the adsorbent surface by chemical bonds in chemisorption [4]. A multi-molecular layer is formed in physical adsorption, and a mono-molecular layer is formed in chemical adsorption. The Langmuir isotherm indicates that adsorption is commonly monolayer due to adsorbate molecules adhering to particular homogeneous sites on the adsorbent surface [25]. There are unfilled sites on the adsorbent, and is no interaction between the molecules retained on the surface. The multilayer adsorption on the homogeneous sites of the adsorbent surface was clarified with the Freundlich adsorption isotherm. It is reversible (desorption with temperature increase or pressure reduction) [4, 12]. The Dubinin-Radushkevich (D-R) isotherm is a model used to describe adsorption processes, particularly in cases where the adsorption follows a pore-filling mechanism rather than surface adsorption. It is suitable for predicting the adsorption of gases and liquids onto porous materials and considers the energy of adsorption to estimate the heterogeneity of the surface. Unlike the Langmuir isotherm, the D-R model can handle non-uniform systems and is based on the Polanyi potential theory [24].

Table 2 shows the isotherm parameters and their correlation coefficients (R^2) for MG adsorption onto butt and tobacco hydrochar. Figure 5 shows the isotherm models graphs. The Langmuir isotherm model best fits the experimental data for MG onto butt and tobacco hydrochar according to the correlation coefficients (R^2). Maximum adsorption capacity (Qm) 83.33mg/g and 227.27mg/g was derived for the adsorption of MG onto butt and tobacco hydrochars respectively.

Table 3. *Kinetic parameters of MG adsorption onto the butt and tobacco hydrochar*

Kinetics	Parameters	MG	
		Butt Hydrochar	Tobacco Hydrochar
Pseudo First Order	R^2	0.90	0.70
	k_1 (min^{-1})	0.008	0.05
	q_e (mg g^{-1})	2.57	893.90
Pseudo Second Order	R^2	0.99	0.99
	k_2 (g mg^{-1} min^{-1})	0.034	0.05
	q_e (mg g^{-1})	17.45	17.18
Intraparticle	R^2	0.61	0.45
	k_i (m $g^{-1}.min^{-2}$)	0.20	0.08
	C	15.05	18.34

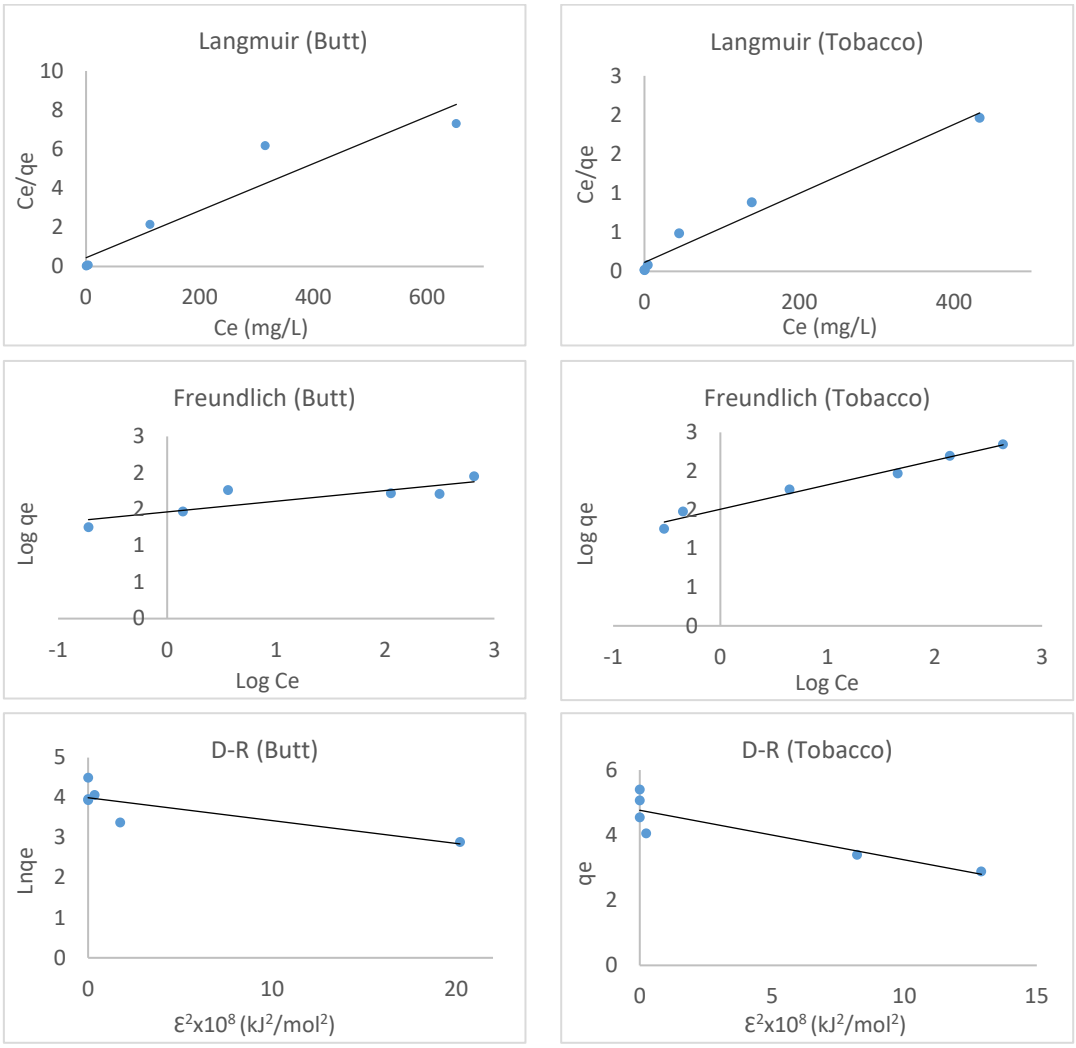


Figure 5. *Adsorption isotherm graphs.*

The Langmuir model describes monolayer sorption in that the adsorbate molecule is adsorbed on particular localized adsorption sites [25]. It indicates no transmigration of the adsorbate in the plane of the surfaces and gives uniform energies of monolayer sorption on the adsorbent surface [12].

The Freundlich isotherm model best fits the experimental data for MG onto tobacco hydrochar. K_F (mg/g) represents the adsorption capacity at equilibrium and n describes the adsorbent surface's heterogeneity and the adsorbed molecules' distribution on the adsorbent surface. The n was 3.16 (Table 2), showing the intensity of MG adsorption to tobacco hydrochar. n > 1 indicates suitable adsorption of the adsorbate molecules onto the adsorbent surface [25].

3.4 Adsorption kinetics

The kinetics of adsorption gives information about the reaction rate concerning the time at constant pressure or concentration [12]. The first step of the sorption mechanism is diffusion adsorption from the adsorbate to the adsorbent surface. The adsorbate molecules coming to the liquid-solid interface pass through the stagnant part on the surface and move towards the pores of the adsorbent in the second stage. This stage is realized by film mass transfer or boundary layer diffusion theory. The third stage is known as pore diffusion. At this stage, the species to be adsorbed is transported in pores of different sizes subject. In the fourth stage, the adsorbate molecules are attached to the pores of the appropriate size and take place [25, 26].

Figure 6 shows the kinetic models graphs. The pseudo-first-order, pseudo-second-order, and intraparticle diffusion models studied the MG adsorption kinetics on butt and tobacco hydrochar. The pseudo-second-order kinetic model

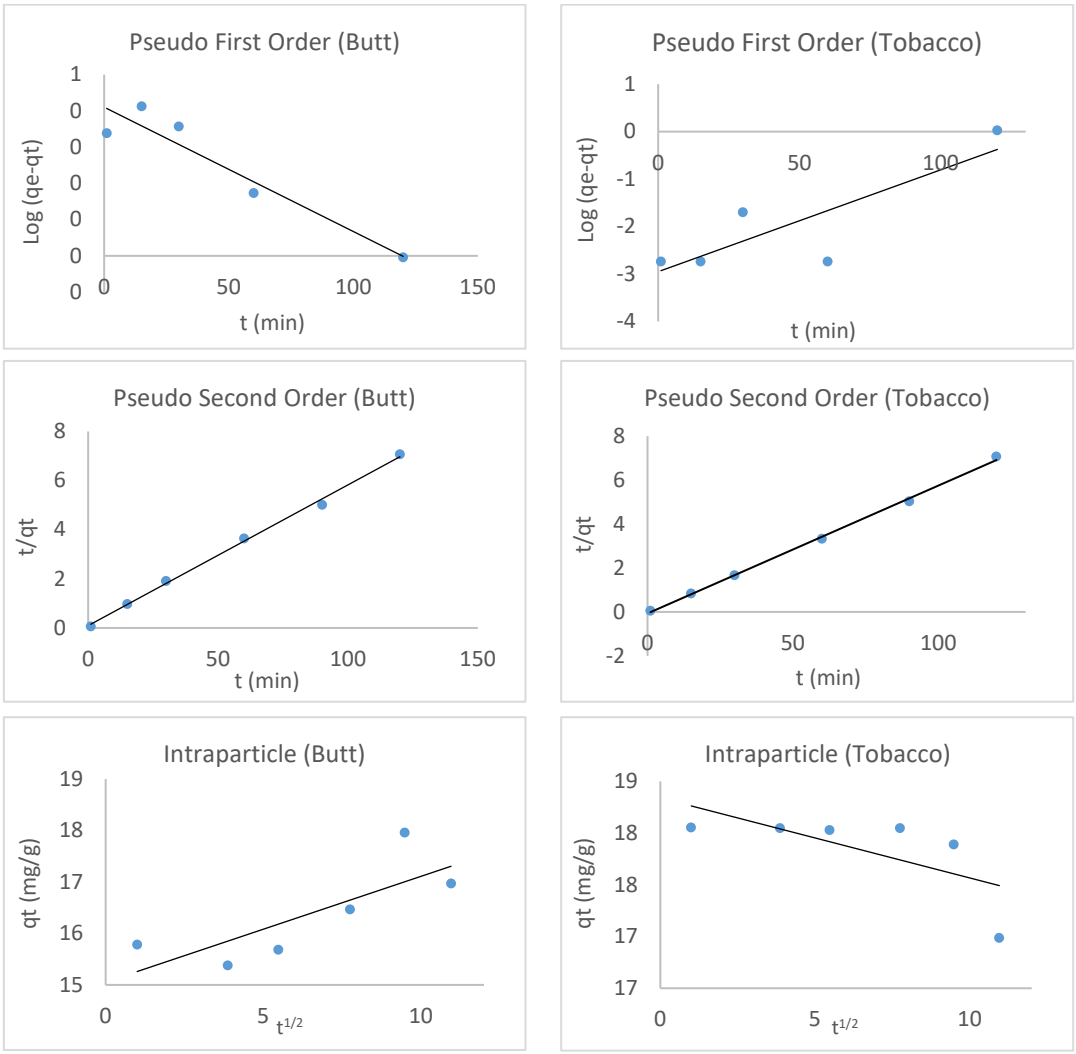


Figure 6. *Adsorption kinetic graphs.*

best fits the experimental data (R^2 = 0.99) for the adsorption of MG onto butt and tobacco hydrochar (Table 3).
The pseudo-second-order kinetic model's fitting showed the chemisorption mechanism's involvement in the rate-determining step [26].

4. Conclusion

In this study, chemically activated hydrochars were produced from cigarette butts and tobacco wastes using potassium hydroxide (KOH) through a two-step pyrolysis method. Elemental composition, morphological structure, and surface area analyses of the produced hydrochars indicated that they are suitable for use as adsorbents. The hydrochars were then applied to the removal of Malachite Green, a chemical dye widely used in various industrial applications that poses toxic risks to both the environment and human health, from aqueous solutions. The adsorption method was employed in the removal studies, yielding exceptionally high removal efficiencies of up to 99%. However, a rapid decline in removal efficiency was observed at higher Malachite Green concentrations (200 mg/L and above). Isotherm and kinetic modeling studies were conducted to elucidate the adsorption mechanism. The results revealed that the adsorption process was single-layered, homogeneous, and chemical in nature. The findings suggest that tobacco and cigarette butt wastes can serve as effective and low-cost alternatives for dye removal. Future studies could further support these results through desorption and regeneration experiments.

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