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Original Article

Synthesis Eco-friendly Biochar from Spent Coffee for Using to Remove Acidic and Basic Dyes from Aqueous Media

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Abstract

This study focuses on reuse of biowaste material locally available as spend coffee to synthesis high surface area biochar and evaluate its potential to remove dyes with acidic and basic in nature, as Acid Fuchsin and Methylene Blue dyes. The biochar was fabricated using chemical activation with various concentrating (20, 30, 50) vol. % of phosphoric acid (H3PO4). The pyrolysis process carried out in a tubular furnace under flow of nitrogen (N2) at 600°C for 3 hours. The final prepped biochar was analyzed using various techniques such as XRD, SEM, FTIR, TGA, EDX, N2-adsorption, BET, and pore volume (Vpore). Furthermore, batch adsorption experiments were conducted to remove Acid Fuchsin and Methylene Blue dyes from aqueous solutions under different variables of pH of solution (2-8), contact time (0-240) min, initial dye concentration (20-200) ppm, and weight of dosage (0.1-0.5) g/L. The results show that the best surface area and pore volume has been archived for the prepared biochar at 30% H3PO4 concentration were (1385.269 m2/g and 1.12 cm3/g) respectively. As well as, the maximum removal percentage was 92.5% for Fuchsin dye and 90% for Methylene Blue dye achieved at 180 minutes of adsorbing time, with maximum removal achieved with initial dye concentration of 20 ppm, 6.5 pH solution, and 0.3 g/L adsorbent. The study found that Freundlich isotherm and kinetic adsorption models fit well with batch the experimental data which indicates homogenous distribution and limiting active sites with adsorption capacity (384.61) mg/g and (357.14) mg/g for Acid Fuchsin and Methylene Blue dyes respectively. While, the pseudo-second-order kinetic model indicates a physic-sorption is the limiting step.

Keywords: *Acid Fuchsin, Batch Adsorption, Biochar, Isotherm Kinetics, Methylene Blue, Spent Coffee*

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1. Introduction

The quality of water resources is a fundamental determinant of environmental sustainability and public health worldwide. Contamination of water by industrial effluents has become a critical challenge, especially in textile-producing regions, due to the discharge of dye-laden wastewater that negatively affects aquatic ecosystems and reduces water usability for human and ecological needs. The presence of color compounds in water bodies can impair light penetration, inhibit photosynthesis, and pose chronic toxicity risks to biota and humans like [1]. Among the various pollutants, synthetic dyes are particularly persistent, resistant to biodegradation, and often toxic even at low concentrations. Conventional wastewater treatment processes such as coagulation, flocculation, oxidation, and membrane filtration have been applied to dye removal; however, they often suffer from high operational costs or limited efficiency for complex dye mixtures. Adsorption has emerged as one of the most promising treatment techniques due to its operational simplicity, high efficiency, and adaptability to diverse contaminant types [2]. Activated carbon (AC) stands out as a widely accepted adsorbent for removing organic pollutants from water owing to its extensive internal porosity and high specific surface area. Commercial activated carbons are typically derived from carbonaceous precursors such as wood, coal, or agricultural residues through physical or chemical activation processes that generate a network of micropores and mesopores, enhancing their adsorption performance. However, commercial AC remains relatively expensive, motivating research into low-cost, sustainable precursor materials [3]. In this context, activated carbon produced from waste biomass, especially spent coffee grounds (SCGs), has attracted considerable interest as a cost-effective and eco-friendly adsorbent for dye removal from wastewater. Recent studies have demonstrated that activated carbon derived from SCGs can achieve high adsorption capacities and removal efficiencies for various dyes, while offering the added benefit of valorizing abundant organic wastes that would otherwise contribute to environmental burdens. These materials often exhibit desirable pore structures and surface functionalities that facilitate effective physical adsorption of dye molecules, making them viable alternatives to conventional adsorbents [4]. Chemical activation

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using agents such as phosphoric acid (H_3PO_4) has been shown to enhance porosity development and functional group formation, contributing to improved adsorption performance. The use of H_3PO_4 not only aids in pore formation but also acts to stabilize the carbon structure, reducing excessive burn-off during activation and preserving high surface area, which is a crucial parameter for adsorption processes [5]. Overall, the development of low-cost AC from industrial and agricultural wastes, such as spent coffee, represents a sustainable approach to wastewater treatment that addresses both pollution mitigation and resource recovery. Continued research on optimizing production conditions and characterizing adsorption mechanisms will further support the practical implementation of these materials in water treatment systems [6]. Recent investigations have extensively explored the potential of spent coffee grounds (SCG) as a precursor for biochar, however achieving a balance between high porosity and useful adsorption remains a challenge. For instance, Kouotou et al. [7] synthesized activated carbon from coffee waste using H_3PO_4 activation. The researches attained a surface area of only 945 m^2/g; this modest porosity constrained the maximum adsorption capacity for organic pollutants. Similarly, Tran et al. [8] demonstrated the effectiveness of SCG-derived biochar for the removal of Methylene Blue; however, their study focused exclusively on cationic dyes, leaving the performance against complex anionic dyes unaddressed. In a more recent study, Al-Zahrani et al. [9] utilized modified biomass-based adsorbents for dye remediation but reported lower structural stability and surface areas not exceeding 850 m^2/g. Furthermore, Nguyen et al. [10] highlighted that while biochar can be eco-friendly, most chemical activation processes for SCG result in moderate micro-porosity that struggles with the high molecular weight of dyes like Acid Fuchsin. In contrast, the present study bridges these gaps by achieving an exceptionally high surface area of 1385.269 m^2/g through optimized H_3PO_4 concentration. This significantly outperforms recent literature values and demonstrates a dual-efficiency in removing both acidic (Acid Fuchsin) and basic (Methylene Blue) dyes, providing a more comprehensive and high-performance solution for industrial wastewater treatment. This study aims to valorize spent coffee biowaste through its conversion into high-performance biochar with enhanced surface area and pore volume, designed as an environmentally benign, cost-effective, and economically viable adsorbent for the removal of Acid Fuchsin (AF) and Methylene Blue (MB) dyes from aqueous media. Biochar was synthesized via chemical activation using different concentrations of phosphoric acid (H_3PO_4), followed by pyrolysis under a nitrogen atmosphere to promote the development of porous structures and surface functionalities. The prepared materials were comprehensively characterized to elucidate their surface morphology, functional groups, thermal stability, and key physicochemical properties. Batch adsorption experiments were systematically conducted to evaluate and optimize dye removal efficiency (R %) by investigating the effects of solution pH, adsorbent dosage, initial dye concentration, and contact time. Furthermore, the adsorption behavior of AF and MB onto the synthesized biochar was analyzed using Langmuir and Freundlich isotherm models, along with monolayer adsorption assumptions, in order to identify the most appropriate isotherm and kinetic models governing single-component dye adsorption. Overall, this work seeks to demonstrate the potential of coffee-derived biochar as a sustainable and effective alternative to conventional adsorbents for dye-contaminated wastewater treatment.

2. Materials and Experimental Methods

2.1. Materials and Biochar Synthesis

The primary precursor used in this study, spent coffee grounds (SCG), was sourced from local cafeterias in Baghdad, Iraq. To ensure the removal of all impurities and water-soluble contaminants, the SCG were subjected to rigorous washing with boiling distilled water until the resulting filtrate was completely clear. The cleaned biomass was then dried in a thermal oven at 105°C for a duration of 24 hours to reach a constant weight. Subsequently, the dried material was ground and sieved to obtain a uniform particle size for the activation process. All chemical reagents utilized, including phosphoric acid (H_3PO_4, 85% concentration), Acid Fuchsin (AF), and Methylene Blue (MB), were of analytical grade (sourced from Sigma-Aldrich and BDH) and were used as received without further modification.

The synthesis of the biochar was carried out via a chemical activation-carbonization route. The prepared SCG samples were impregnated with various concentrations of H_3PO_4 (20%, 30%, and 50% v/v) for 24 hours to facilitate deep chemical interaction. Following this, the impregnated samples were carbonized in a horizontal tubular furnace. The thermal treatment was performed at 600°C for 3 hours, maintaining an inert atmosphere through a continuous nitrogen (N_2) flow at a rate of 150 mL/min. Upon cooling to room temperature, the resulting biochar was thoroughly washed with 0.1 M HCl and warm distilled water to neutralize the pH and eliminate any residual acidity or by-products. The final biochar products were dried again at 105°C and stored in airtight containers for subsequent characterization and adsorption studies.

2.2. Physicochemical Characterization of biochar

The physical and chemical characteristics of the prepared biochar from spent coffee were examined using several analytical techniques. Surface morphology was analyzed using a scanning electron microscope (SEM; HRSEM FEI Inspect F50). Surface functional groups were identified by Fourier Transform Infrared Spectroscopy (FTIR; Shimadzu, Japan) over the range of 400–3500 cm^{-1}.

Surface area analysis (BET) was performed using a surface area analyzer (SA-9600 Qsurf Series, Thermo Electron Corporation). Thermal properties of the precursor were evaluated using thermo-gravimetric analysis (TGA; Shimadzu TGA-50). Energy-dispersive X-ray spectroscopy (EDS; HRSEM FEI Inspect F50) was used to determine the elemental composition of the precursor and prepared activated carbon (AC).

The crystalline structure of the prepared AC was examined using X-ray diffraction (XDM300 SSC) equipped with Cu Kα radiation (λ = 0.154 nm), a step size of 0.05°, a 2θ range of 10–80° (high angle), and a fixed power source of 40 kV and 30 mA. Surface textural properties were further characterized using a

Micromeritics TriStar II plus (USA) instrument via N_2 adsorption–desorption measurements at 77 K.

2.3. Dyes

Dyes are a class of colorants that impart color to substances. They are synthetic organic compounds that are generally water-soluble. Various organic dyes are widely used in the textile industry for coloring different products [11]. Textile dyes, along with many industrial pollutants, are highly toxic and potentially carcinogenic, and are associated with environmental degradation and various diseases in animals and humans [12].

Acid Fuchsin (AF) is a prominent acidic dye extensively used for several applications, including textile dyeing and as a laboratory reagent [13]. It is widely used for dyeing textile fabrics, silk, nylon, wool, and leather. Acid Fuchsin is considered a dye pollutant [14] due to its slow degradation rate and its mutagenic and carcinogenic properties. Despite its toxic and persistent nature - attributed to nitrogen-containing bonds in its molecular structure - very few studies have reported adsorption investigations of Acid Fuchsin to date [15]. Figure 1 shows the molecular structure of AF [16].

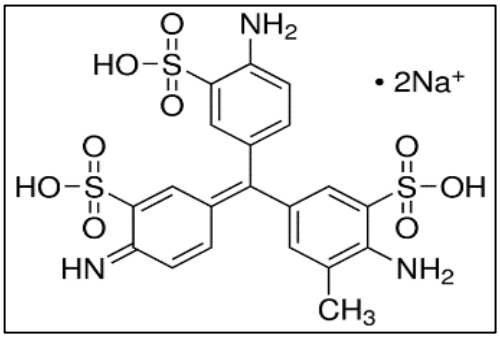

Figure 1. Molecular Structure of AF [16].

Methylene blue (MB) is a monovalent cationic dye classified as a basic dye. It is widely used in the dyeing of silk, leather, paper, and cotton, as well as in ink production. The compound derives its name from the intense deep blue color it exhibits when dissolved in water or alcohol. Methylene blue is a crystalline solid with a faint odor and belongs to the class of basic synthetic dyes [17]. The molecular structure of MB is illustrated in Figure 2 [18].

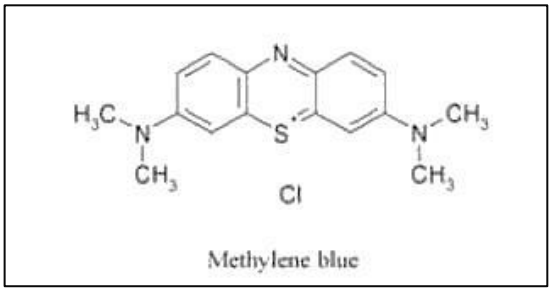

Figure 2. Chemical structure of methylene blue [18].

2.4. Batch adsorption study

Adsorption of (AF) and (MB) dyes by the prepared biochar was carried out by batch mode process. A certain amount of prepared activated carbon was added to 200 mL of the dye solution which is known as initial concentration. This concentration has been achieved by dissolving 1 g from dyes in 1-liter de-ionized distilled water. The solution of stock has been diluted to achieve the desired concentration of 20, 50, 100, 150, and 200 mg/L of dyes pollutants. The sorption equilibrium has been achieved by shaking 0.3 g/L from the prepared AC into 200 mL simulated solutions in a shaker at ambient temperature (25 ± 2) ◦C and pH 6.5 for 180 min. The adsorbents are removed from the solution after the adsorption by utilizing a micro pipette the solution was filtered at different intervals of time like (10, 20, 30, 60, 120, and 180) min. The concentrations of dyes were measured using UV spectrophotometer equipment (Shimadzu UV/Vis 1601 Spectrophotometer, Japan) .The maximum wavelength of (AF) is (540-549) nm and for (MB) is (658-668) nm.

The percentage dye removal and amount of dye adsorbed per unit adsorbent (mg dye/g of adsorbent) were calculated using Equations 1 and 2 respectively:

$$R\% = \frac{(Co - Ce)}{Ce} * 100\% \quad \text{--------- (1)}$$

$$q_e = (Co - Ce).\frac{V}{M} \quad \text{---------- (2)}$$

where, q_e is the adsorption capacity (mg/ g), Co is the initial dye concentration (mg/ L), Ce is the equilibrium dye concentration in solution (mg/ L), V is the volume of the solution (L), m is the weight of the modified spent coffee in (grams) [19].

2.5. Adsorption isotherms

Different models have been used in the literature to describe the adsorption isotherms. The Langmuir and Freundlich models are the most frequently used models for the adsorption process. In this work, we used these isotherm models to fit the experimental data for the adsorption of dyes from aqueous solution by the prepared activated carbon at ambient conditions.

The Langmuir isotherm model assumes that the adsorption occurs at homogeneous sites at adsorbent surface, and saturation takes place when the dye molecules fill the sites where no more adsorption can occur at that site. The constant (KL and qmax) values can be evaluated from the intercept and the slope of the linear plot of experimental data [20].

The Langmuir sorption isotherm form by Equation [21]:

$$\frac{Ce}{qe} = \frac{1}{q_{max}.KL} + \frac{Ce}{q_{max}} \quad \text{--------- (3)}$$

where, Ce (mg/L) is the equilibrium concentration, qe (mg/g) is the amount of adsorbed per unit mass of adsorbent, q max (mg/g) and KL (L/mg) are Langmuir constants related to adsorption capacity and rate of adsorption.

The Freundlich isotherm can be used for describing adsorption on a heterogeneous surface energy system where the binding sites are not equivalent. The constants KF and n can be evaluated from the intercept and the slope of the linear plot of experimental data of log qe versus log Ce.

The Freundlich sorption isotherm using the log-transformed by [21]:

$$\text{Log qe} = \log kf + \frac{1}{n} \log Ce \quad \text{---------- (4)}$$

qe (mg/g) is the amount of adsorbed per unit mass of adsorbent, Ce (mg/L), 1/ n (mg/g), and Kf (L/mg) 1/ n = the concentrations in equilibrium, the adsorption rate, Freundlich constants related to the favorability of the adsorption process, and the adsorption capacity of the adsorbate, respectively.

2.6. Kinetic models

The adsorption kinetic of the prepared biochar was investigated using the pseudo-first order and pseudo-second order.

The pseudo-first order equation based on the capacity of the solid adsorbent mainly affects the adsorption rate during the adsorption process. The kinetic model equation is given by Eq. [22]:

$$\log (q_e - qt) = \log q_e - \frac{k_1}{2.303} t \quad \text{--------- (5)}$$

where, qe and qt denote the adsorption capacity (mg/g) at equilibrium and at a given time, respectively, and K_1 is the rate constant of the pseudo-first order model.

The pseudo-second-order model is based on the assumption that the limiting stage of the reaction rate can be the chemisorption involving valence forces by electron exchange between the adsorbent and the adsorbate [23].

The equation of the psudo-second-order model is defined by [23]:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} \quad \text{--------- (6)}$$

Where qe (mg/g) and qt (mg/g) are the capacity of adsorption (mg/g) at a time (min) and K_2 is the adsorption rate constant.

3. Results and discussion

3.1. Biochar characterization

3.1.1. Surface Area and Porosity Analysis

The structural characteristics, including specific surface area and porosity, are fundamental parameters governing the adsorption efficiency of biochar. In this study, the nitrogen (N_2) adsorption-desorption isotherms were employed to evaluate the textural properties of the synthesized biochar. As summarized in Table (1), the chemical activation with phosphoric acid (H_3PO_4) significantly enhanced the development of the pore network.

The results demonstrate that the biochar activated with 30% H_3PO_4 exhibited the most superior textural attributes, achieving a remarkable BET specific surface area of 1385.269 m^2/g and a total pore volume of 1.2 cm^3/g. This substantial increase in surface area can be attributed to the dehydrating effect of H_3PO_4, which promotes the degradation of organic compounds and the simultaneous formation of a complex microporous and mesoporous framework [24].

Furthermore, the pore size distribution analysis indicates that the synthesized biochar possesses a well-developed porosity, which is crucial for the effective entrapment of both Acid Fuchsin and Methylene Blue molecules. The high surface area recorded in this work significantly exceeds values reported in several recent studies for coffee-based adsorbents, thereby providing a greater density of active sites for dye sequestration. This optimization of the pore structure confirms the efficacy of the proposed synthesis method in producing a high-performance, eco-friendly adsorbent suitable for complex wastewater treatment.

3.1.2. Energy Dispersive X-ray Spectroscopy (EDX) Analysis

Energy dispersive X-ray spectroscopy (EDX) was employed to evaluate the elemental composition and surface atomic distribution of both the spent coffee precursor and the synthesized biochar. The elemental profiles, as detailed in Table (2), reveal a significant chemical transformation following the carbonization and chemical activation processes.

Table 1. Surface area and pore volume for activated carbon prepared at carbonization temperature of 600 °C and time 3 h.

Material	H_3PO_4 acid concentration (%)	Carbonization temperature (°C)	Surface area (BET) m^2/g	total pore volume (V_{Total}) (cm^3/g)
AC1	20	600	322.98	0.032
AC 2	30	600	1385.269	1.12
AC3	50	600	279.8	0.029

It was observed that the raw spent coffee possessed a higher oxygen (O) content compared to the biochar, reflecting the presence of various oxygenated organic functional groups in the precursor. However, the carbon (C) content increased substantially from 47.1% in the raw material to 69.22% in the final biochar. This carbon enrichment is primarily attributed to the thermal decomposition occurring during the carbonization stage, which facilitates the elimination of volatile matter and non-carbonaceous elements, thereby concentrating the carbon matrix into a more stable, aromatic framework.

Furthermore, the EDX spectra indicated that both the precursor and the biochar contained trace amounts of inorganic constituents, including magnesium (Mg), silicon (Si), and sulfur (S). The low concentration of these elements is consistent with the characteristics of biomass-derived adsorbents and aligns with findings reported in the literature [25].

Table 2. The results of EDX analysis for spent coffee and the prepared biochar.

No.	Elements	Spent coffee	The prepared biochar
1	C	47.1	69.22
2	O	51.3	18.66
3	Mg	0.2	0.6
4	Al	0.5	0.2
5	Si	0.1	1.61
6	S	0.2	0
7	K	0.3	0
8	Ca	0.3	0.1

3.1.3. Thermo Gravimetric Analysis (TGA)

The biowaste precursor was analyzed using thermo-gravimetric analysis (TGA) to evaluate the thermal decomposition of the sample. Low sample masses (3.6190 mg) and small particle sizes (1–1.18 μm) were selected to reduce the effect of intra-particle mass and heat transport limitations and, thus, to avoid the problem of the "thermal lag" between the sample and the controlling (external) thermocouple during the tests. The selected temperature range was 25–900°C at a rate of 50°C/min under nitrogen purge (5 L/min).

The TGA analysis curve of precursor was illustrated in Figure .3. It has clearly presented that the curve can be separated into three parts. First part (up to 200 °C) the weight change is almost (85.3 %). This is mainly attributed to the loss of moisture and other volatile substances extractable from water. Second part from (200 to 425) °C is considered the main stage where the majority of the weight loss occurs, contributing to the depolymerisation of

biomass components, such as hemicellulose, cellulose, and lignin. In this part the spent coffee biomass has lost weight about (26.5 %). The last part of the cure, up to 900 °C, is attributed to solid decomposition, where the weight loss rate is slower, and a slight weight loss could be due to char consolidation. This final stage weight loss was (4.9 %). Where, the overall weight change of biomass sample was 96.87%. Similar results were reported in previous studies for other researchers [26].

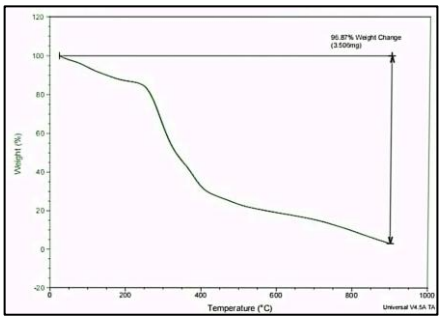

Figure 3. Thermal gravimetric analyses (TGA) for spent coffee.

3.1.4. X-Ray diffraction analysis (XRD)

It is important to investigate and quantify the crystalline nature of adsorbent materials in order to achieve sufficient adsorption. The technique of X-ray diffraction analysis (XRD) is to determine the crystallinity of adsorbent materials, identify the crystalline phases present, determine the spacing between lattice planes, and investigate the preferential ordering and epitaxial growth of crystallites within the material [27]. From the XRD analysis of the activated carbon prepared shown in Figure.4, there exist two broad diffraction peaks located at 2θ = 20°–30° and 40°–50°, revealing the presence of amorphous carbon which is disorderly stacked up by carbon rings.

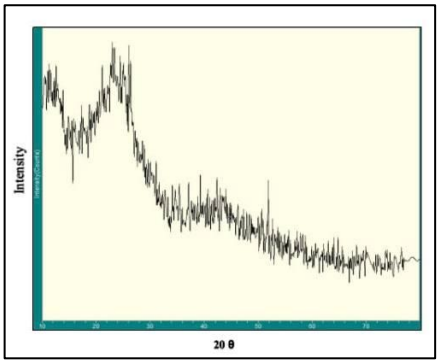

Figure 4. XRD for the prepared activated carbon

3.1.5. Scanning electron microscope (SEM)

The SEM method was used to observe the surface physical morphology of the material depending on the impregnation ratios and the external surfaces of the biochar have the pores which were different sizes and different shapes. It can be seen from the micro-graphs that the external surfaces of the biochar are full of cavities and quite irregular as a result of activation by 30% of phosphoric acid. The pores observed in SEM images as shown in Figure.5 (a, b) are having diameter in (2 to 20) micrometer range. The Figure.5a shows the biochar that have a bulky morphology with smooth surface and no pores. In contrast Figure.5b show irregular shape with macropores and active sites that was observed in the activated spent coffee.These distinguishable macropores might be formed through the releaseof pyrolysis gases during the chemical activation with H3PO4 .The increase in porosity and cavities can lead to enhance the ability of these materials to adsorb dyes with high efficiency [28].

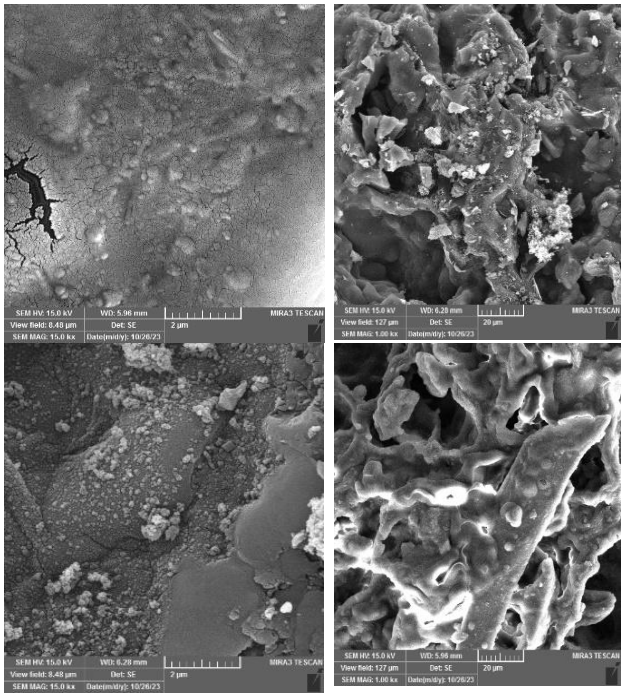

Figure 5. Scanning electron microscopy (SEM) for: (a) spent coffee before activation (b) spent coffee after activation.

3.1.6. N2 adsorption–desorption analysis

The N_2 adsorption–desorption isotherms of the prepared activated carbon were determined using an automatic adsorption analyzer. Nitrogen adsorption is the standard technique employed to evaluate the porosity of carbonaceous adsorbents. The resulting adsorption isotherm provides valuable information regarding the porous structure of the adsorbent, as well as its adsorption heat and relevant physicochemical properties.

As illustrated in Figure 6, the N_2 adsorption–desorption isotherms recorded over a relative pressure (P/P_0) range of 0–1 indicate that the adsorption behavior of the prepared activated carbon corresponds to a type IV isotherm. According to the IUPAC classification, this type of isotherm is characteristic of mesoporous materials according to the IUPAC classification [29].

As shown in Figure 6, the N_2 adsorption–desorption isotherm exhibits a pronounced hysteresis loop at high relative pressures, indicating the coexistence of micro- and mesoporous structures in the prepared activated carbon. This behavior is consistent with the BET results, which reveal a high specific surface area of 1385.269 m2/g, along with a total pore volume of 1.12 cm3/g which confirming the predominance of a mesoporous texture. The development of this well-defined porous structure is mainly attributed to the chemical activation with phosphoric acid, which effectively promotes pore formation and enhances the surface area of the adsorbent.

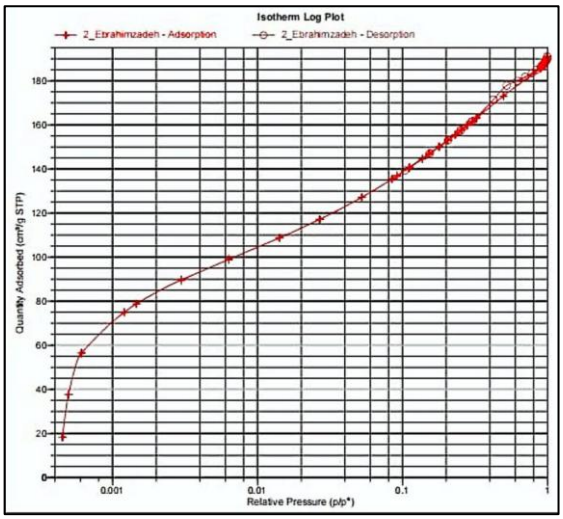

Figure 6. N2 adsorption-desorption isotherm of activated carbon prepared.

3.1.7. Pore size distribution

The N_2 adsorption–desorption isotherm (Figure 6) exhibits a pronounced hysteresis loop at high relative pressures, confirming the coexistence of micro- and mesoporous structures in the prepared activated carbon. This observation is consistent with the BET analysis, which reveals a high specific surface area of 1385.269 m2/g, a total pore volume of 1.12 cm3/g, and an average pore diameter range of 1–5 nm, indicating a predominantly mesoporous texture.

Further insight into the pore structure was obtained from the pore size distribution derived using the Barrett–Joyner–Halenda (BJH) method (Figure 7). The results show that mesopores are mainly distributed within the range of 1–5 nm, while macropores are negligibly present or poorly developed. This well-developed mesoporous network plays a crucial role in enhancing the adsorption performance by facilitating the diffusion of both Acid Fuchsin (AF) and Methylene Blue (MB) molecules into the internal pore structure. Moreover, the combination of high surface area and appropriate pore size distribution increases the accessibility of active adsorption sites, thereby promoting efficient dye uptake through electrostatic interactions and surface adsorption mechanisms.

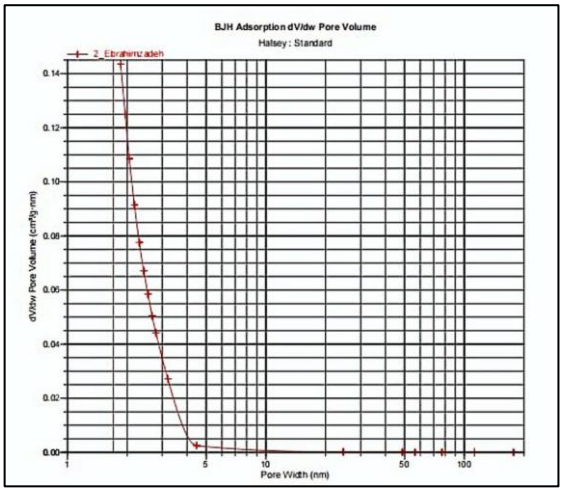

Figure 7. Pore size distribution of optimal Activated carbon prepared.

3.1.8. Fourier transform infrared spectroscopy (FTIR)

The FTIR study of activated carbon may help to classify the type of functionality present in a prepared AC. In order to achieve this, the FTIR spectrum was used with the present materials. It has been shown in Figure 8 and 9. The peak at around 2956.58 - 2916.67 cm^{-1} indicates the presence of C-H bond of strong intensity of carboxylic acid and derivative group [30]. Methylene group (CH2) stretching is obtained at 2214.55 cm^{-1}.Peaks at 1000.90 cm^{-1} and 715.35 cm^{-1} were prominent in organosolv pretreated spent coffee waste indicating the preservation of the hemicellulose and cellulose fractions at 1753.21 cm^{-1} and 1552.13 cm^{-1} for C=O and C–O stretching of the carboxylic group, respectively [32]. There was another peak observed at 1519.04, 1149.77 cm^{-1} corresponding to the Nirto (R-NO2) N=O bonds. The peak at 684.85 cm^{-1} and 479.93 are related to the C=C bending of alkene and C_X fluoride functional group [32].

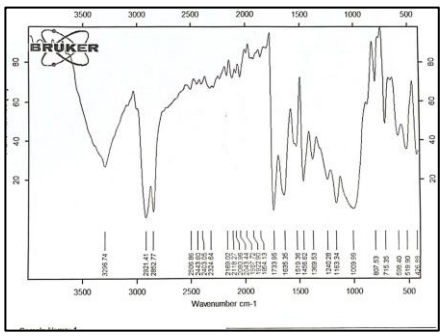

Figure 8. FTIR spectra of raw material.

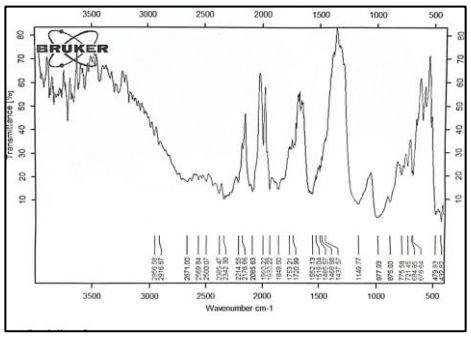

Figure 9. FTIR spectra of activated carbon prepared.

3.2. Adsorption studies

3.2.1. Effect of pH

The effect of solution pH on the adsorption of Acid Fuchsin (AF) and Methylene Blue (MB) onto the prepared activated carbon was investigated at an initial dye concentration of 20 ppm over a pH range of 2–8. As illustrated in Figure 10, the adsorption capacity was minimal at pH 2. A noticeable enhancement in dye adsorption was observed as the pH increased, particularly within the range of 6.5–8, with the maximum adsorption efficiency achieved at pH 8. The observed pH-dependent adsorption behavior can be attributed to changes in the surface charge of the activated carbon and the ionization state of the dye molecules. At lower pH values, the adsorbent surface becomes increasingly protonated, resulting in a reduction in negatively charged active sites and an increase in positively charged sites. This condition is unfavorable for the adsorption of positively charged dye cations due to electrostatic repulsion. In addition, the presence of excess H^+ ions under acidic

conditions leads to competitive adsorption with dye cations for available active sites, further reducing dye uptake [33, 34].

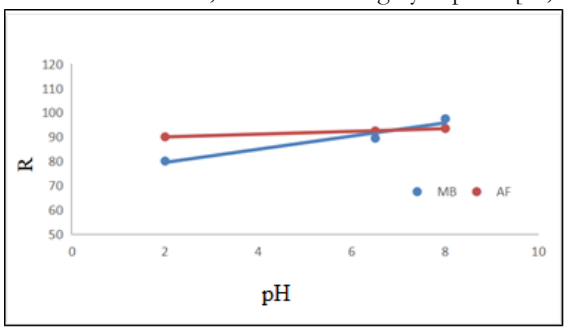

Figure 10. Effect of pH on Removal of AF&MB at (Time 180 min, dosage 0.3 g/L and concentration 20 ppm).

3.2.2 Effect of Initial Concentration

The effect of initial concentration of AF and MB on the adsorption capacity of prepared of activated carbon is shown in Figure.1. From the figure as the initial concentration of AF and MB increases from 20 to 200 ppm, Maximum dyes removal occurred for low initial concentration of AF and MB that showed gradual reduction when initial concentration of dyes was raised. It could be ascribed to fixed concentration of adsorbent dosage. .The amount of adsorption for dye removal is highly dependent on the initial dye concentration. The effect of initial dye concentration depends on the immediate relation between the concentration of the dye and the available sites on an adsorbent surface. In general, the percentage of dye removal decreases with an increase in the initial dye concentration, which may be due to the saturation of adsorption sites on the adsorbent surface. On the other hand the increase in initial dye concentration will cause an increase in the capacity of the adsorbent and this may be due to the high driving force for mass transfer at a high initial dye concentration [35], so with increase in initial dye concentration the adsorption sites were fixed and achieved saturation. Hence with increase in dye concentration no further adsorption could be achieved and resulted in reduced removal of dye with increase in dye concentration

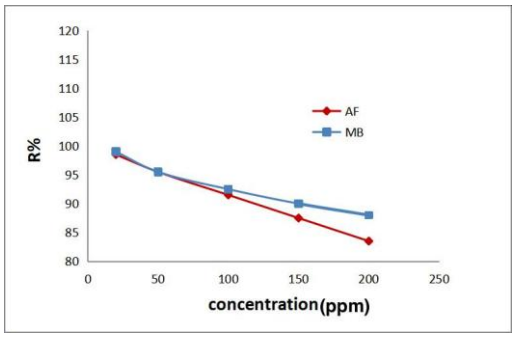

Figure 11. Effect of Initial Concentration of dyes on Removal at (dosage 0.3 g/L, PH 6.5 and Time 180 min).

3.2.3 Effect of Adsorbent Dose

The effect of adsorbent dosage on the removal efficiency of Acid Fuchsin (AF) and Methylene Blue (MB) was further interpreted in relation to the adsorption isotherm behavior. As shown in Figure 12, increasing the activated carbon dose from 0.1 to 0.5 g/L at an initial dye concentration of 200 ppm resulted in a notable increase in removal efficiency, followed by a tendency toward saturation at higher dosages. This behavior can be attributed to the progressive increase in available surface area and active adsorption sites with increasing adsorbent mass, which enhances dye uptake until equilibrium conditions are approached [36].

At higher adsorbent dosages, the stabilization of removal efficiency suggests a reduction in the effective utilization of adsorption sites, likely due to overlapping or aggregation of adsorbent particles. This observation is consistent with the assumptions of the Langmuir and Freundlich isotherm models, where adsorption reaches equilibrium as the number of accessible sites becomes sufficient to accommodate the dye molecules present in solution. The good agreement of the experimental data with the selected isotherm models confirms that adsorption of AF and MB onto the prepared activated carbon is governed by surface site availability and adsorption capacity, as described by monolayer and heterogeneous surface interactions.

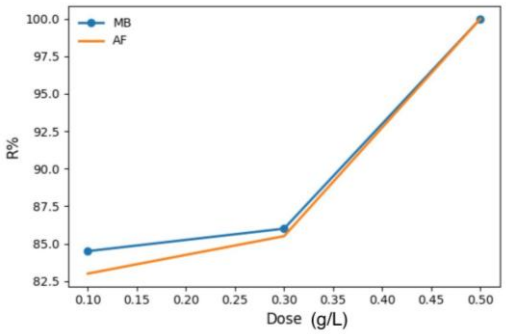

Figure 12. Effect of adsorbent dosage on Removal of AF and MB at (Time 180 min, Initial pH 6.5 and Concentration 200 ppm).

3.2.4 Effect of Contact Time

The adsorption kinetics of Acid Fuchsin (AF) and Methylene Blue (MB) onto the prepared activated carbon were investigated by varying the contact time at initial dye concentrations of 20–200 ppm. As shown in Figure 13, the removal efficiency of both dyes increased rapidly within the first 45 minutes, followed by a slower approach to equilibrium at approximately 180 minutes. The maximum removal percentages of AF were 50.1, 77.5, 82.5, 90, 92.5, 93, and 93.1%, and for MB were 48.5, 72.5, 79.95, 83.5, 90, 90.1, and 90.2% for initial concentrations of 20, 50, 100, 150, and 200 ppm, respectively.

The rapid initial adsorption can be attributed to the high concentration gradient (driving force) and the abundant availability of unoccupied surface area and active sites on the mesoporous structure of the activated carbon, as confirmed by BET and BJH analyses. As equilibrium is approached, the remaining vacant sites decrease, slowing the adsorption rate.

The adsorption process is further influenced by solution pH, where higher pH values favor the adsorption of cationic dyes like MB due to increased negatively charged sites on the adsorbent surface, whereas acidic conditions suppress adsorption due to electrostatic repulsion and competition with H^+ ions. The combined effects of high surface area, well-developed mesopores, optimal adsorbent dose, and favorable pH collectively enhance dye removal, in agreement with Langmuir and Freundlich isotherm

models, which describe monolayer adsorption on heterogeneous surfaces and the distribution of adsorption energies.

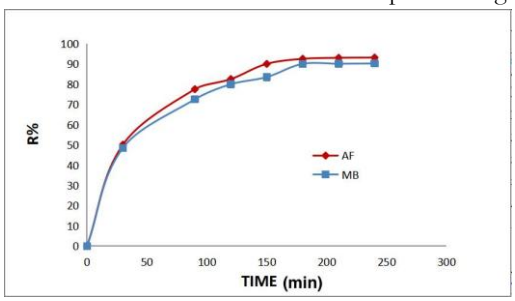

Figure 13. Effect of Contact Time on Removal of AF and MB at (dosage 0.3 g/L, PH 6.5 and Conc. 20 ppm).

3.3 Adsorption Isotherm

The adsorption isotherms is very important to know how adsorbents interact with absorbent substances. Adsorption data is installed on different isotherms to find a suitable model that can be used in the design in this project the Langmuir and Frendlich Eqs where used to the experimental data of the adsorption of dyes solution. The Langmui and Freundlich isothermal adsorption models were applied for dyes adsorption utilized by prepared activated carbon which having particle size (1-1.18 mm) at pH equals (6.5) and constant temperature (25 oC). The results are summarized in Table (3, 4) and shown in Figures (14, 15) for Langmuir and Frendlich respectively. The tables show the calculated constants of the tow equations for Langmuir and Frendlich, with the correlation coefficient R^2 values. The R^2 value is 0.9912 % for the Langmuir isotherm for AF solution with maximum adsorption capacity of 384.61 mg/g and 0.9981% for MB solution with maximum adsorption capacity of 357.14 mg/g .This may be because of the surface area of prepared activated carbon has a homogeneous distribution active sites .Several studies and research have shown the successful application of isotherm to link experimental adsorption data to adsorption to activated carbon [37, 38]. For Freundlich isothermal the values of n and Kf as tabulated in Tables (3,4) were determined to be at 1.030 and 39.29 L/mg for AF and at 0.9244 and 51.38 L/mg for MB , respectively as shown in Figure (16,17). In general, it has been found that for the biosorption of dyes onto prepared AC, the model of Freundlich isotherm shows a best fit to the adsorption data than the Langmuir isotherm model.

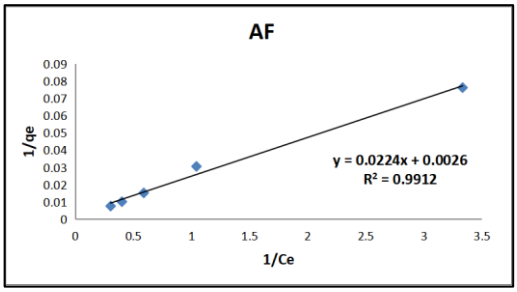

Figure 14. Langmuir isotherm for the adsorption of AF (adsorbent dosage 0.3 g/L; initial pH 6.5; time 180 min)

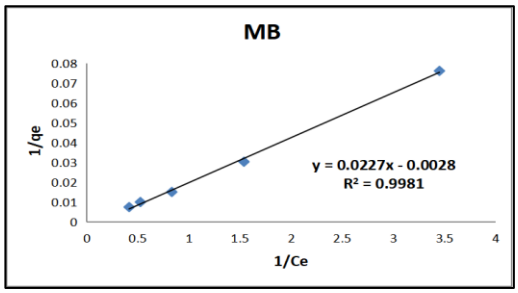

Figure 15. Langmuir isotherm for the adsorption of MB (adsorbent dosage 0.3 g/L; initial pH 6.5; time 180 min).

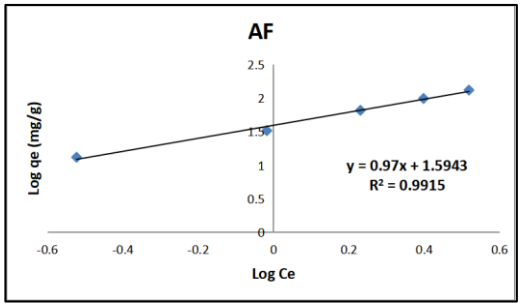

Figure 16. Freundlich isotherm for the adsorption of AF (adsorbent dosage 0.3 g/L; initial pH 6.5; time 180 min).

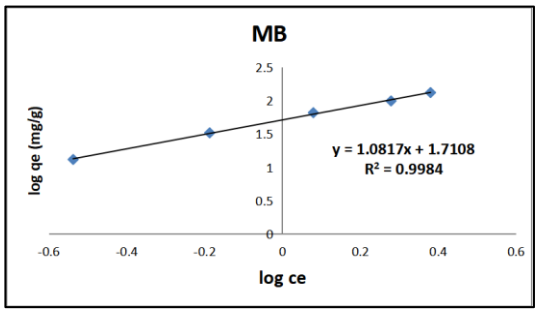

Figure 17. Freundlich isotherm for the adsorption of MB (adsorbent dosage 0.3 g/L; initial pH 6.5; time 180 min).

Table 3. Fitted isotherm models for the adsorption of AF on optimal AC.

Langmuir isotherm		Freundlich isotherm	
$\frac{Ce}{qe} = \frac{1}{q_{max}.KL} + \frac{Ce}{q_{max}}$		$Log\ qe = log\ kf + \frac{1}{n}\ log\ Ce$	
qmax (mg/g)	384.61	n	1.030
KL (L/mg)	0.1160	Kf	39.29
R^2	0.9912	R^2	0.9915

Table 4. Fitted isotherm models for the adsorption of MB on optimal AC.

Langmuir isotherm		Freundlich isotherm	
$\frac{Ce}{qe} = \frac{1}{q_{max}.KL} + \frac{Ce}{q_{max}}$		$Log\ qe = log\ kf + \frac{1}{n}\ log\ Ce$	
qmax (mg/g)	357.14	n	0.9244
KL (L/mg)	0.1233	Kf	51.38
R^2	0.9981	R^2	0.9984

3.4 adsorption kinetics

To investigate the adsorption mechanism, the pseudo-first-order, pseudo-second-order, and intraparticle diffusion kinetic models

were applied to analyze the experimental data for dye adsorption onto the prepared activated carbon. The suitability of each kinetic model was evaluated by comparing the experimental data with the values predicted by the models using the correlation coefficient (R^2).

The pseudo-first-order kinetic model is illustrated in Figures (18) and (19). The obtained R^2 values were 0.8124 for Acid Fuchsin (AF) and 0.8052 for Methylene Blue (MB), indicating a relatively weak agreement between the experimental data and this model.

In contrast, the pseudo-second-order kinetic model, shown in Figures (20) and (21), exhibited significantly higher correlation coefficients, with R^2 values of 0.9987 for AF and 0.9979 for MB. These high R^2 values demonstrate an excellent fit between the experimental data and the pseudo-second-order model.

Based on these results, it can be concluded that the adsorption kinetics of both dyes are better described by the pseudo-second-order model rather than the pseudo-first-order model.

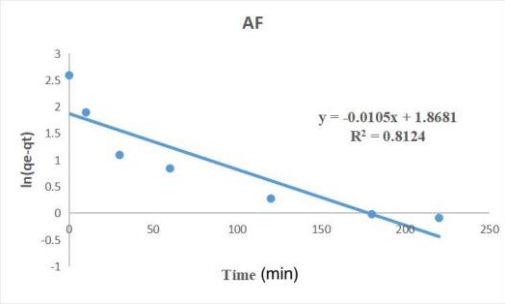

Figure 18. Pseudo-first-order for the adsorption of AF (adsorbent dosage 0.3 g/L; initial pH 6.5; Concentration 20 ppm).

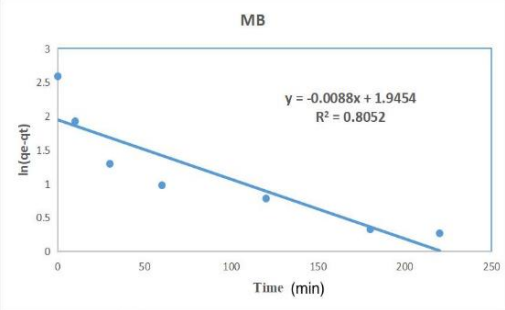

Figure 19. Pseudo-first-order for the adsorption of MB (adsorbent dosage 0.3 g/L; initial pH 6.5; Concentration 20 ppm).

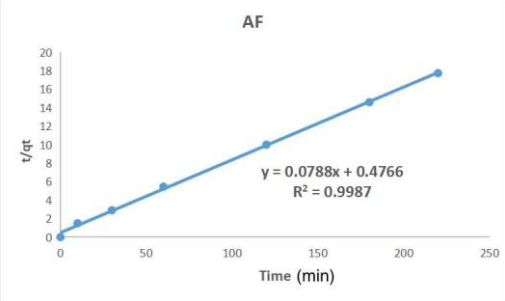

Figure 20. Pseudo-second-order for the adsorption of AF (adsorbent dosage 0.3 g/L; initial pH 6.5; Concentration 20 ppm).

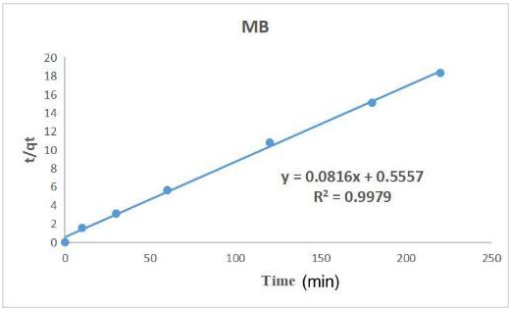

Figure 21. Pseudo-second-order for the adsorption of MB (adsorbent dosage 0.3 g/L; initial pH 6.5; Concentration 20 ppm).

4. Conclusions

Activated carbon derived from spent coffee waste was successfully prepared and demonstrated high efficiency for the removal of Acid Fuchsin (AF) and Methylene Blue (MB) from aqueous solutions. Chemical activation with phosphoric acid followed by thermal treatment at 600 °C for 3 h produced a highly porous material, with the highest BET surface area (≈ 1385 $m^2\ g^{-1}$) obtained at an impregnation ratio of 30 wt.%.

Adsorption performance was significantly influenced by solution pH, initial dye concentration, contact time, and adsorbent dosage. Removal efficiency increased with increasing contact time and adsorbent dose, reaching equilibrium after approximately 180 min, while higher initial dye concentrations resulted in lower removal efficiency. Neutral to slightly alkaline conditions (pH 6.5–8) favored adsorption, whereas acidic conditions markedly reduced removal efficiency.

The optimal adsorption conditions were identified as an initial dye concentration of 20 ppm, solution pH of 6.5, and an adsorbent dosage of 0.3 g L^{-1}. Equilibrium data were better described by the Freundlich isotherm model, indicating heterogeneous surface adsorption, and the adsorption kinetics followed a pseudo-second-order model, suggesting that physisorption is the dominant mechanism.

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