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Removal of Methylene Blue from Wastewater Using AL Haji Plant as a Low-Cost, Eco-Friendly Adsorbent

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Abstract

This study investigates the use of agricultural waste, specifically the Al Haji plant, for wastewater treatment. The term 'Al Haji plant' refers to species from the Al Haji genus, particularly "Al Haji maurorum " and "Al Haji canescens ", which are drought-resistant plants of the Fabaceae family. Traditionally, these plants are known for their medicinal and antimicrobial properties. In this research, waste from the Al Haji plant was converted into activated carbon (AC) as a low-cost and techno-economically viable biosorbent for the removal of methylene blue (MB) dye from wastewater. The production of AC involved two key steps: chemical activation using sodium hydroxide (NaOH) and carbonisation at 300 °C. The properties and adsorption potential of the resulting AC were investigated. Various parameters were studied, including pH, contact time, stirring rate, initial dye concentration, adsorbent dosage, and temperature. Characterisation of the biosorbents was performed using Scanning Electron Microscopy (SEM) and Brunauer–Emmett–Teller (BET) surface area analysis. The SEM investigation showed notable pores in the Al Haji plant sample that could enhance the absorption of the MB dye. Results indicated that the BET surface area for physical AC is 25.5796 m^2/g. The surface area of chemical AC was reported as 16.2767 m^2/g. The dye exhibited pseudofirst-order adsorption kinetics, with average rate constants of 0.020082 and 0.001589 for physical and chemical activation approaches, respectively. An adsorption isotherm study was performed using the Langmuir model, which showed an R^2 value of 0.827 and a maximum adsorption capacity of 5.24 mg/g." The study of adsorption thermodynamics revealed that MB adsorption on Al Haji activated carbon is spontaneous and endothermic.

Keywords: *Al Haji plant; Batch sorption; Kinetic models; Methylene blue; Thermodynamic Parameter.*

1. Introduction

Recent years have witnessed a significant increase in freshwater use due to technological advancements and rapid population growth, leading to substantial quantities of wastewater that must be immediately discharged into the natural environment[1]. Accelerated mechanical advancement has led to a significantly increased production capacity, an improved quality of life for humanity, and other repercussions, including the emergence of dangerous synthetic substances and dyes[2, 3]. Dyes constitute a significant category of chemical industrial effluents in aquatic environments. They are among the numerous organic compounds that contaminate water systems, owing to their critical role and extensive application across various industrial sectors, including paper, textiles, leather, and food industries[4, 5]. Methylene blue (MB) is among the most harmful metal contaminants released into water due to rapid industrialization and urbanization[6]. These contaminants are persistent, non-biodegradable, accumulate in essential organisms, and can cause significant health impacts in humans above specific concentrations[7, 8]. The ability to eliminate these colors from wastewater is essential for safeguarding human health and the environment[9]. To mitigate the escalating pollution from toxic metals, many physical and chemical remediation strategies, including photo degradation, have been developed[10, 11]. Various methods such as precipitation, membrane filtration, ion exchange, reverse osmosis, electro-coagulation and flocculation have been developed; nevertheless, they exhibit significant drawbacks, including high costs, hazardous sludge production, and selective contamination[12]. Nonetheless, adsorption remains the preeminent approach due to its cost-effectiveness, absence of toxic by-products, and ease of execution Adsorption has emerged as the predominant method for wastewater treatment due to its simplicity, scalability, and efficacy in eliminating trace water pollutants[13, 14] .A range of materials has been examined as adsorbents for MB removal, including resin, silica, clay, and activated carbon. [15].Activated carbon serves as a very efficient adsorbent owing to its plentiful useful groupings, permeability, substantial surface area, and a complex internal structure. It has been widely utilized to eradicate diverse organic and inorganic pollutants from biologically resistant contaminated waterways [16, 17]. Commercial activated carbon is a commonly employed industrial adsorbent for processes of separation, purification, and recovery. However, it necessitates regeneration following each adsorption process and entails significant production costs. Employing biomass waste, including peanuts husks, sugarcane bagasse, coconut shells, bamboo, rice husks, mango kernels, and teas residues, as a precursor for activated carbon is an effective method for decreasing the overall production costs associated with its creation [18], [19]. The two essential stages in the production of activated carbons are carbonation and reactivation. The aim of carbonizing is to reduce the amount of volatile compounds in the original material. The precursor undergoes pyrolysis at temperatures between 300 and 850 °C, leading to its degradation transforming into gases and tars primarily composed of hydrogen, oxygen, and nitrogen. The char generated by this process results in a high fixed carbon content but exhibits low surface area and porosity. Thus, an activation phase is crucial for improving porosity and structural organization, leading to the formation of the highly porous solid referred to as AC. The two techniques for treating adsorbents are physical and chemical approaches. The physical approach for treating an adsorbent entails activation through heating in an oven. The chemical treatment of adsorbents entails activation through the addition of acids or alkalis, including H_3PO_4, NaOH, K_2CO_3, and KOH [20, 21]. The aim of the study is to employ the adsorption technique for effective removal of dyes (methylene blue) from industrial wastewater, with the goal of enhancing efficiency, reducing treatment duration from the aqueous solution.

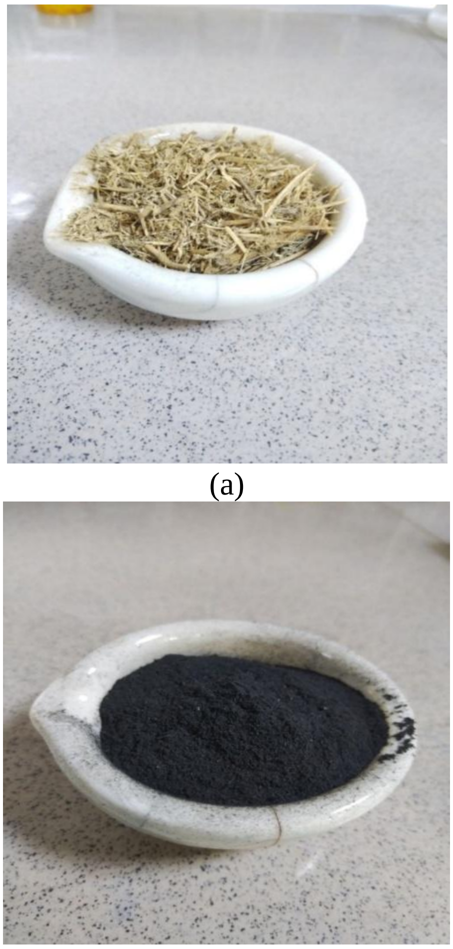
2. Materials and Processes

2.1. Materials

The AL Haji residue was locally sourced from leftovers. The Al Haji plant used in this study was collected from its natural habitat in [Al-Ishaqi area in Salah al-Din Governorate, Iraq]. Methylene blue (MB) was chosen as the target adsorbate . It was supplied from (Merck, Germany). Methylene blue was directly used with distilled water. Sodium hydroxide (NaOH), employed as the activation agent with a minimum assays of 98% was purchased from England. The effect of each variable (e.g. pH, time, dosage) was studied individually using a single variable change approach with the other variables stabilising at their optimal values

2.2. Preparation of Activated Carbon

The AL Haji plant was used as a precursor for synthesizing activated carbon (AC). The sample was washed thoroughly with distilled water and dried at 105°C for 24 hours. It was then crushed into powder using a disk mill. The preparation of activated carbon was carried out by chemical activation. The material was first heated at 300°C for two hours while passing nitrogen gas over it at a rate of 500 cc/min. After that, it was activated with a 0.5 M NaOH solution for 24 hours. Following activation, the sample was thoroughly washed with distilled water multiple times to remove any remaining base, until a pH from 6 to7 was reached.", ensuring the removal of residual alkalinity. The material was then dried again at 105°C for 2 hours before storage in an airtight container to prevent moisture absorption. Physical activation was conducted in a muffle furnace at 300°C for 2 hours under a controlled flow of CO_2 gas at a rate of 500 cc/min for one hour. This step was followed by purging the system with N_2 gas at a flow rate of 500 cc/min for one hour, allowing the sample to cool to ambient temperature. These parameters were chosen to optimize pore structure and surface area based on previous studies[22, 23].



(a)

(b)

Fig. 1. AL Haji plant (a) before burning (b) after burning

2.3. Preparation of Solutions

Methylene blue (MB) ($C_{16}H_{18}N_3SCl.{}_3H_2O$) (Merck, Germany), an analytical grade product, was utilized to prepare artificial wastewater. All of the materials were analytical quality. The stock MB (methylene blue) solution (0.5 g) was produced by using distilled. The stock solution later was diluted to the concentrations (25, 30, 40, 50, 70 ,100 mg/L) using distilled water.

2.4. Batch Adsorption Experiments

Adsorption experiments in batch mode were conducted at room temperature utilizing 100 ml of Methylene blue. The mixture was then vigorously shaken on a platform shaker until equilibrium was reached at different of the initial pH (2–10), adsorbent dosages (0.5–3 g/L), contact time (10–90 min), initial MB concentration (25– 100 mg/L), stirring rate(200-1000 rpm),and temperatures (25, 30, 35, 40,45,and 50 °C)The study also examined the adsorption of MB on AL Haji particles. Following the adsorption trials, the solutions underwent filtration, and the concentrations of MB in the resulting liquid were analysed using a UV/Vis-DR 3000. A wavelength of 668 nm was selected to obtain maximum absorption. [24] The removal percentage of Methylene Blue (MB), denoted as R%, was evaluated based on the initial and equilibrium concentrations as shown in Eq. (1)

$$R\% = ((C_0 - C_e) / C_0) \times 100\%, \qquad ...(1)$$

where, R denotes the removal percentage, while and represent the initial and equilibrium concentrations of MB (mg/L), respectively. The adsorption capacity of activated carbon at equilibrium is given by AL Haji

The equilibrium adsorption capacity, (mg/g), was determined using the following equation. (2) [25, 26]

$$q_e = \frac{(C_0 - C_e)V}{m} \qquad ... (2)$$

where q_e represents the adsorbed amount of MB (mg/g), V denotes the sample volume (mL), and m refers to the mass of the added adsorbent (g)

3. Results and Discussion

3.1. Effect of pH

The pH level plays a crucial role in the adsorption of methylene blue dye onto adsorbents, particularly when physical or chemical activation methods are used because they affect the surface charge of the adsorbent material. For instance, in acidic media, adsorbents may have a positive surface charge, repelling positively charged dye molecules, while in alkaline media, the adsorbent surface may become negatively charged, attracting positively charged dye molecules. Therefore, the optimization of pH is essential to maximize dye removal efficiency. The study found that physical activation led to better removal of methylene blue dye than chemical activation**,** particularly because physical activation improved the adsorbent's surface area, reactivity, and the interaction between the adsorbent and dye molecules. In contrast to physical activation, chemical activation is more sensitive to pH changes and may not achieve the same efficiency across a wide pH range. It shows 95.1704 % removal rate when pH is 4 for physical activated carbon where it has a lower dye removal rate of 84.072 % by chemical activation with pH of 4 [27].

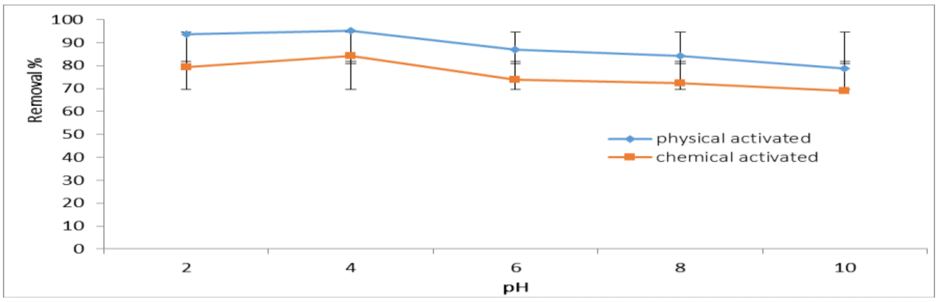


Fig. 2. Effect of pH on the precent of dye removal on AC

3.2. Effect of Contact Time

Through batch studies, the effect of contact duration on the ability of dyes to bind to activated carbon was investigated to reach the equilibrium depicted in Figure ٣. It depicts the movement of the dye molecule from the solvent to the adsorbent particle as it diffuses across the surface, which can be used to explain the mechanism of colour removal. According to the findings, activated carbon achieved equilibrium after operating for 90 minutes. The adsorption capacity remained unchanged After 90 min, the dye removal rate reached 96.484 % for physical activation and 87.52 % for chemical activation. The adsorption rate initially increased significantly before progressively decreasing as a result of saturation. The physical adsorption's passive uptake or the ion exchange of the adsorbent surface may be used to define the fast-phase sorption. Physical activation typically results in enhanced adsorption capacity and accelerated adsorption rates relative to chemical activation, particularly when accounting for the temporal aspect of the experiment. Physical approaches enhance the accessibility and efficacy of adsorption sites, making them preferable for the rapid and efficient removal of pollutants such as methylene blue [28-30].

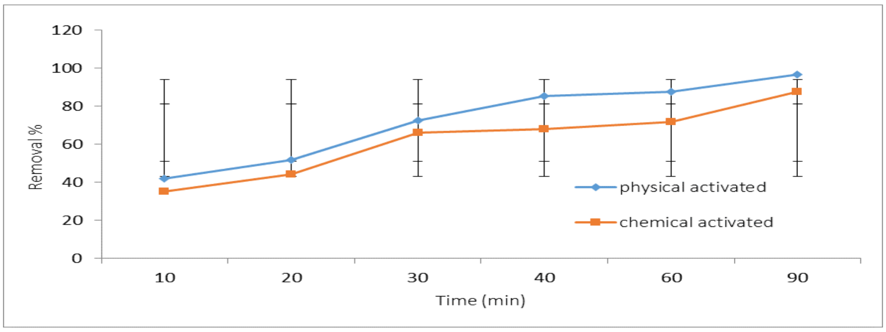


Fig. 3. Effect impact of contact time on AC's dye removal percentage

3.3. Effect of Stirring Rate

The thickness of the adsorbent film formed by the particles that revolve in the adsorption system of a liquid makes the difference. This is mainly due to the rate of solute diffusion to the particle surface, which plays a crucial role in the adsorption process, the accomplment resulted in showing that when the agitation speed raised from 200 rpm to 1000 rpm the augmentation of agitation strength showed an effect on the elimination rate. This might be seen in Figure 4 at a speed of 400 rpm for the physically activated samples where dye removal rate reached 97. 862 %. However the increase in the removal efficiency is no significant. This proves the effectiveness of physical activation in eliminating MB due to its impact on surface properties and adsorption rate.

While(MB) is found to have less virustatic than in the stirring case, indicating that the stirring speed has little impact. on the film thickness. 400 rpm was the stimulation rate used in each experiment The dye removal rate is reaching 90.275 % for chemical activationThe physical activation of Haji plant demonstrated higher efficiency in eliminating MB. This can be attributed to physical treatments (such as UV irradiation or thermal treatment), which enhance the surface characteristics of the adsorbent, thereby improving both the adsorption rate and overall efficiency. These approaches enhance the contact between dye molecules and the plant surface without introducing substances that may disrupt the adsorption process. Chemical activation may result in the generation of undesirable by-products or diminish the long-term efficacy of the adsorbent[31, 32].

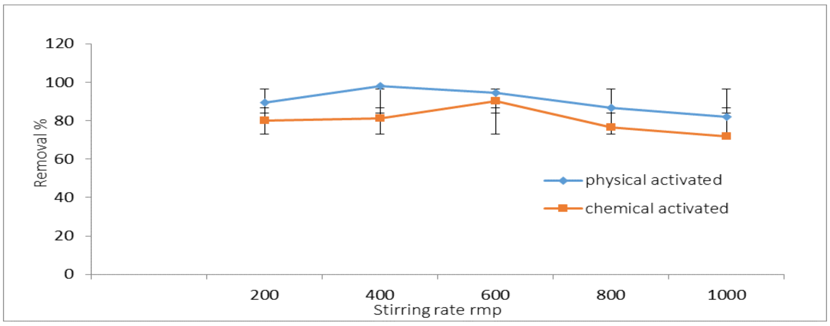


Fig. 4. Effect of stirring rate on the precent of dye removal on AC

3.4. Effect of adsorbent dose

The effects of different AC concentrations (0.5.1, 1.5, 2,2.5, and 3g) on absorption was investigated at pH = 4 , dye concentration of 25 mg/L MB, and shaking duration (90 min) as shown in Figure 5. The rate of adsorption was generally observed to increase with the amount of adsorbent used. When the adsorbent concentration was increased from 0.5 to 3 g, this increase became more apparent where most of the dye is eliminated in 90 minutes. Beyond 3 g, the dye concentration was hardly varied. The dye removal rate for physical activation is 98.552% at a concentration of 3 g of activated carbon, while the dye removal rate for chemical activation is 90.968% at the same concentration. The increased surface area of the adsorbent and the growing number of adsorption sites are responsible for the increase in dye removal percentage as the adsorbent dose rises. The superior efficacy of physically activated carbon in eliminating methylene blue is due to its enhanced porous structure, increased surface area, improved adsorption kinetics, and advantageous surface chemistry. These features enable physical activation to produce an adsorbent that is more efficient in adsorbing big organic compounds, such as methylene blue, than chemically activated carbon[33, 34].

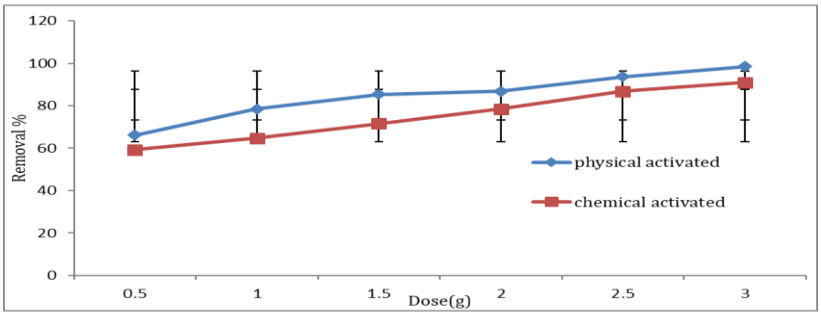


Fig. 5. Effect of adsorbent dose on the percent of dye removal on AC

3.5. Effect of Initial Concentration

Figure 6 illustrates the impact of the initial methylene blue concentration (25–100 mg/l) on the effectiveness of methylene blue removal. The studies, which were carried out at room temperature (25 °C), pH 4.0, and a swirling speed of 400 rpm, used a fixed dose of adsorbent (3 g). The effect with respect to the initial dye concentration is determined by the direct relationship between the initial dye concentration and the available binding sites on the sorbent surface. Figure 6 illustrates the effect of the initial dye concentration. The percentage of dye removal often decreased as the initial dye concentration increased, which may be explained by the saturation of adsorption sites on the adsorbent surface. At low concentrations, the adsorbent surface exhibited open active sites; however, when the initial dye concentration increased, the active sites required to adsorb dye molecules disappeared. Perhaps because to the significant driving force on mass at a high starting dye concentration, increasing the initial dye concentration results in an increase in the adsorbent's loading capacity. Stated otherwise, the residual concentration of dye molecules will be larger at higher initial dye concentrations. At low concentrations, the absorption rate becomes independent of the initial concentration, because the number of dye molecules is smaller than the number of available absorption sites. The removal rate reaches 97.176% for physical activation and 89.588% for chemical activation. Physical activation is more efficient because it improves the surface characteristics of the plant material, hence augmenting its capacity to absorb MB. Chemical activation, conversely, may not enhance adsorption to the same degree because it does not directly influence surface area or surface activity in a comparable manner [35, 36].

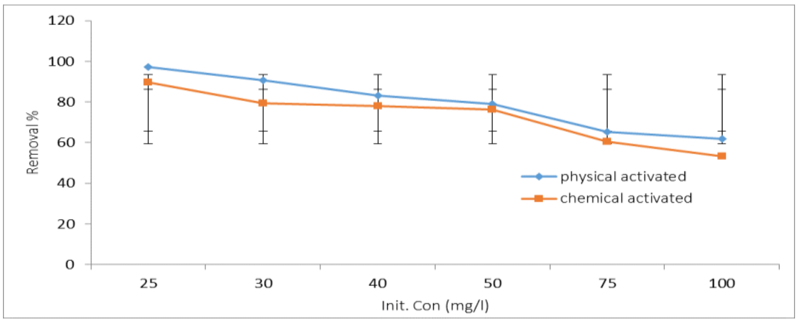


Fig. 6. Relation between initial dye concentration on precent removal for different dyes on Ac

3.6. Effect of Temperature

The effects of temperature on MB adsorption on AL Haji are shown in Figure 7. It was observed from Figure ٧ that the amount of methylene blue removed by AL Haji increased as the temperature increased. Similarly, the MB extraction efficiency of Al Haji increased from 86.828% to 98.552% when the temperature was raised from 25 °C to 50 °C. For both physical and chemical activation, the MB removal performance of Al Haji improved from 66.828% to 86.836% over the same temperature range. The study showed that when the temperature increases, the effectiveness of removing MB also improves. This indicates that the process of removing MB is endothermic[37]. When there is a sufficient amount of energy, such as high temperature, the quantity of molecules that interact with the active site on the surface will increase according to[38]. the adsorption effectiveness increased at high temperatures because of the internal diffusion that controlled the adsorption process. This is because increase in temperature increases the movement of the solute Physical activation is more efficacious at elevated temperatures as it augments membrane permeability and accelerates cellular processes without the hazards of chemical breakdown or toxicity. Chemical activation, conversely, is more susceptible to temperature-induced breakdown, diminishing its total efficacy[39, 40].

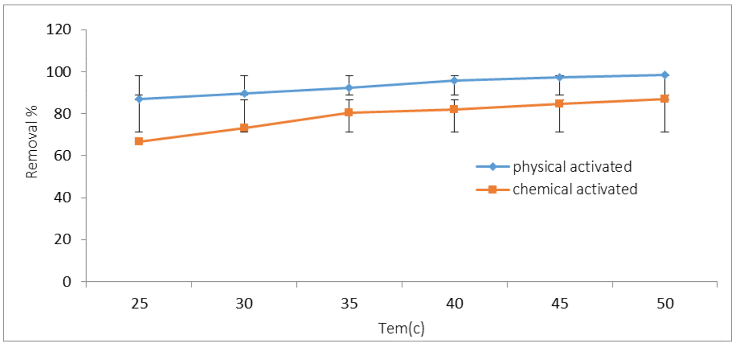


Fig. 7. Effect of Temperature on the precent of dye removal on AAC

3.7. FTIR Analysis of AL Haji Powder and AAC

Untreated Sample (Before Activation) 3400.50 cm^{-1}: This peak represents the O-H stretching vibration, characteristic of hydroxyl groups (alcohols, phenols, or water). The broadness of the peak suggests hydrogen bonding, which is typical in plant cell wall components like cellulose, lignin, and other phenolic compounds. These hydroxyl groups play a key role in adsorption by forming hydrogen bonds with other molecules or metal ions during the adsorption process. 3232.70 cm^{-1}:O-H stretching vibrhydrogen bonding environments in the, 2916.37 and 2848.86 cm^{-1}: These-H stretching vibration methyl (–CH$_3$) and *methylene (–CH$_2$) groadsorption because, 1730.15 cm^{-1}: TC=O stretching vibration, carbonyl groups (found adsorption processes, 1637.56 and 1616.35 cm^{-1}: These=C stretching in aromatic rings (such as those in lπ-π interactions , 1537.27 cm^{-1}: This peak is associated with N-H bending vibrations, which are typical of amine groups. Amine groups, found in alkaloids, proteins, or amino acids, can interact with metals or other species via electrostatic interactions or complexion, 623.01 and 472.56 cm^{-1}: These low-frequency peaks are typically associated with metal-oxygen bonds, suggesting that mineral or inorganic components in the plant material may interact with metals or ions during adsorption. These peaks may reflect the presence of minerals in the plant structure. Physically Activated Sample (e.g., Heating or Grinding) In this case, the physical activation process alters the Sample structure, which influences the FTIR spectrum. Physical activation typically enhances the surface area and modifies the hydrogen bonding, which can facilitate adsorption. 233.70 cm^{-1}**:** Similar to the untreated sample, this peak corresponds to the O-H stretching vibration. A slight shift compared with the untreated sample could indicate changes in the hydrogen bonding network of hydroxyl groups, possibly due to physical treatment that alters the molecular interactions in the sample.2918.30 cm^{-1}This is again the **C-**H stretching vibration from methylene and methyl groups, indicating that the aliphatic components remain unchanged by physical activation. The presence of these groups facilitates hydrophobic interactions in the adsorption of organic contaminants or ions. 1710.80 cm^{-1}**:** This shift in the C=O stretching vibration suggests some reorganization or change in the carbonyl environment, possibly due to the alteration of ester or acid groups during physical activation. This change could enhance the ability of the material to interact with adsorbents, such as metal ions or organic molecules. 1612.49 and 1508.41 cm^{-1}**:** These peaks, linked to C=C stretching vibrations in aromatic rings, indicate that the aromatic components of the plant material are still present, albeit slightly altered due to physical activation. These changes might enhance π-π interactions during the adsorption of organic molecules. 1111.00 cm^{-1}: This peak is likely due to C-O stretching (in alcohols, ethers, or esters) and indicates the presence of polar functional groups that can engage in hydrogen bonding or electrostatic interactions with metal ions or organic contaminants during adsorption. 781.17, 623.01, and 472.58 cm^{-1}: These peaks remain consistent with the untreated sample and suggest the continued presence of mineral or inorganic components, which could be responsible for metal ion adsorption or complexion at the surface of the plant material. Chemical activation such as through acid–base treatments leads to the breaking of chemical bonds, modification of functional groups, and the formation of new reactive sites within the material. This process significantly alters the adsorption properties by increasing the number of functional groups that can interact with adsorbates. 3165.19 cm^{-1}: This peak is associated with O-H stretching, but the shift to a lower wavenumber indicates weaker hydrogen bonding or possible protonation of the hydroxyl groups after chemical treatment. This could make the material more hydrophilic and capable of forming stronger interactions with metal ions or polar molecules. 2926.01 cm^{-1}: This peak is from C-H stretching in methylene and methyl groups, indicating that aliphatic groups remain intact after chemical activation. These groups may still contribute to hydrophobic interactions during adsorption. 1570.06 and 1516.05 cm^{-1}. These peaks correspond to C=C stretching in aromatic rings. The shift in these peaks suggests that aromatic components might be altered by chemical activation, which could increase π-π interactions during adsorption of aromatic pollutants or metal ions. 1382.96 and 1153.43 cm^{-1}: These bands are associated with N-H bending and C-N stretching vibrations, suggesting that amine groups or amide functional groups were modified during chemical activation. These amine groups can form complexes with metal ions or interact with organic pollutants. 1029.29 cm^{-1}: This peak corresponds to C-O stretching, likely from alcohols or ethers. The presence of these groups after chemical treatment suggests an increased surface polarity, which may enhance the material's ability to adsorb polar contaminants. 887.26 cm^{-1}: This peak may be due to C-H bending vibrations in aliphatic or aromatic structures. The shift here could indicate some structural reorganization after chemical activation. 781.17, 626.87, and 474.49 cm^{-1}: These peaks are still associated with metal-oxygen bonds, indicating that chemical activation did not alter the inorganic components of the plant. These may be key in adsorption of Methylene blue [23, 41-44].

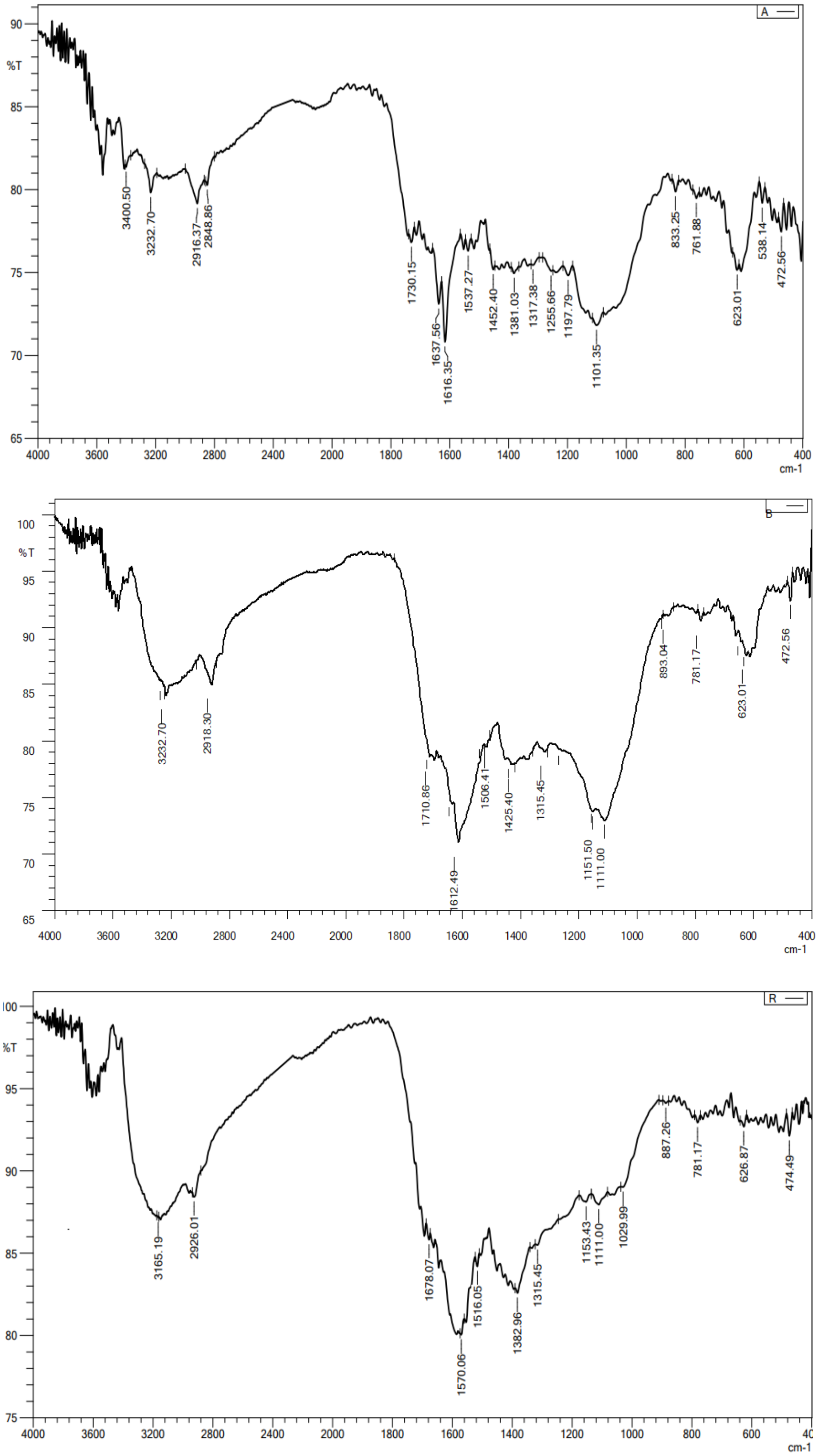
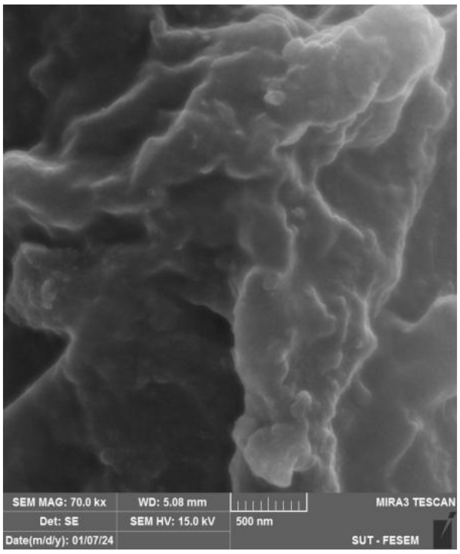


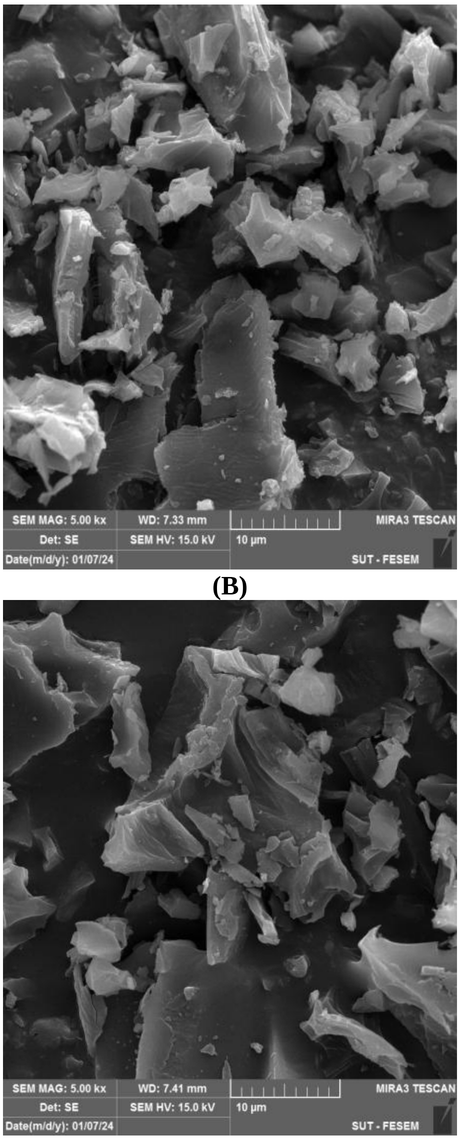
Fig. 8. FTIR (A) AL Haji powder (B) AL Haji physical activated carbon (C) AL Haji chemical activated carbon

3.8. Scanning Electron Microscopy (SEM)

A scanning electron microscopy technique was utilized to examine the surface morphology of both the AL Haji powder and the prepared AL Haji activated carbon (AAC) . Unprocessed Sample (500 nanometres): The particle size is extremely small (500 nanometres), which gives the material high structural stability. No cracking or defects are observed, as the material has not been subjected to any external forces, maintaining its original form. The pore distribution in this sample is relatively uniform, with smaller pores dominating the structure. Physically Activated Sample (10 micrometres): This activation leads to greater cracking and an increase in brittleness due to the mechanical stress applied during processing, making the particles more susceptible to cracking and defects. The material exhibits a non-homogeneous structure, with a broader distribution of pore sizes, including larger pores, which result from the physical activation process. The increased brittleness and cracking make the structure less stable and more prone to irregularities. Chemically Activated Sample (10 micrometres): On the other hand, chemical activation enhances the structural integrity of the material by improving molecular bonding, making it more stable and resilient compared to physical activation. The chemical treatment leads to a more homogeneous pore structure with improved molecular bonding, which reduces brittleness. This treatment not only strengthens the material but also promotes the formation of more uniform pores, with a more controlled distribution of pore sizes compared to the physically activated sample"[45-47].



(A)



(C)

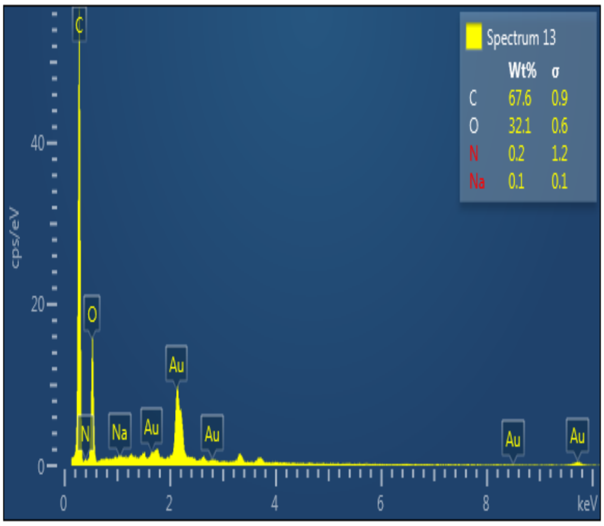
Fig. 9. SEM images of (A) AL Haji powder (B) AL Haji physical activated carbon (C) AL Haji chemical activated carbon

The elemental compositions of the raw material (AL Haji) and AAC were examined using an energy dispersive spectroscope (EDX). High Carbon Content (67.7%) The untreated sample Figure (a) has a very high carbon content, indicating that it is primarily composed of organic materials such as carbon-rich compounds (e.g., hydrocarbons, polymers). This could be a natural or synthetic material, potentially a polymer or organic compound with little to no processing Oxygen (32.1%)Oxygen is present as part of oxygen-containing functional groups (e.g., hydroxyl, carbonyl, or ether groups), typical in organic materials or biological compounds Low Nitrogen and Sodium (0.2% and 0.1%, respectively) The presence of nitrogen and sodium in trace amounts suggests minimal contamination or the presence of minor inorganic species. Physically Activated Sample Figure (b) Reduced

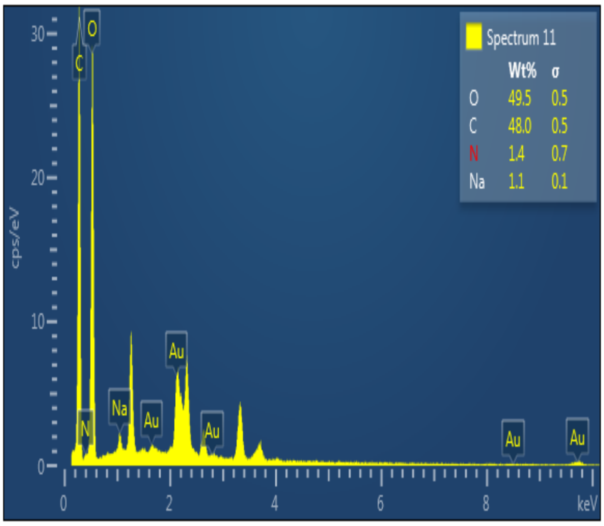
Carbon Content (49.5%) The significant reduction in carbon content (from 67.7% to 49.5%) after physical activation suggests that physical processes such as heating, compression, or grinding led to the breakdown or removal of some carbonaceous material. This could result in a material with a less carbon-rich structure, potentially altering its chemical composition or surface characteristics. Increase in Oxygen Content (48.0%) The increased oxygen content could indicate oxidation during physical activation, or the introduction of more oxygenated functional groups to the material's surface, which can improve its reactivity or interaction with other chemicals Nitrogen (1.4%) and Sodium (1.1%) The increase in nitrogen and sodium suggests that the activation process could have introduced nitrogenous species (such as amines or nitrates) and sodium, possibly from atmospheric exposure, contaminants, or from additives used during activation. The nitrogen content is notably higher compared to the untreated sample, which might indicate that some nitrogenous compounds were introduced during the physical activation process. Chemically Activated Sample Figure (C) Carbon Content (61.3%)The carbon content in the chemically activated sample is somewhat higher than in the physically activated one but lower than the untreated sample, indicating that the chemical activation process altered the carbon structure. Chemical activation often involves the use of reagents (such as acids, bases, or salts) that can introduce new surface chemistry or modify the material Oxygen Content (34.4%) The oxygen content is lower than in the physically activated sample, which suggests that the chemical activation process did not oxidize the material as much as physical activation did. Chemical activation typically involves reactions with chemicals that can introduce specific functional groups without overly oxidizing the structure Sodium (3.4%) The high sodium content in the chemically activated sample could be due to the use of sodium-based reagents or salts during activation, which are commonly used in chemical activation processes. This may indicate that sodium compounds are incorporated into the structure of the material, affecting its chemical reactivity and properties Nitrogen (0.9%) The nitrogen content is slightly higher than in the untreated sample, suggesting that some nitrogenous species were introduced during chemical activation, possibly through the use of nitrogen-containing reagents [48-51].

Table 1,
Elemental Composition of Samples

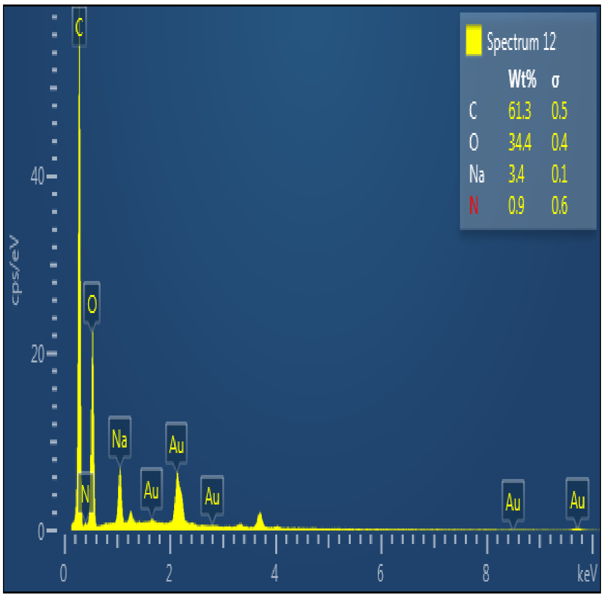
Element	Raw Sample (%)	Physically Activated Sample (%)	Chemically Activated Sample (%)
Carbon	67.7	49.5	61.3
Oxygen	32.1	48.0	34.4
Nitrogen	0.2	1.4	0.9
Sodium	0.1	1.1	3.4



A: EDX analysis of AL Haji powder



B: EDX analysis of physical activated carbon



R: EDX analysis of AL Haji chemical activated carbon

Fig. 10. EDX(A) AL Haji powder (B) AL Haji physical activated carbon (R) AL Haji chemical activated carbon

4. Adsorption Isotherms

The isotherms adsorption method is employed to evaluate experimental data to characterize the equilibrium correlation between the dose of the adsorbent and the adsorption of the adsorbate over a period of time. As shown in Figures (11) (12) the experimental equilibrium data of (MB) adsorption on AC were analysed using two linearized isotherm models, namely Langmuir and Freundlich. The Langmuir isotherm and Freundlich isotherm calculations for (MB) onto AC were conducted based on Eqs.3 and 4 respectively.

The Langmuir model, based on adsorption equilibrium assumptions, is given by Equation (3).

$$q_e = \frac{\text{qmKaCe}}{1 + \text{KaCe}} \quad \text{... (3)}$$

While The Langmuir model's linearized form could be expressed using Eq. (4):

$$\frac{1}{qe} = \frac{1}{qm} + \frac{1}{\text{qmKa}}\,\frac{1}{ce} \quad \text{... (4)}$$

Where:

q_e: is the amount of sorbate adsorbed per unit mass at equilibrium(mg/g).

q_m: is the maximum adsorption capacity(mg/g).

C_e: is the equilibrium adsorbate concentration (mg/l).

K_a: is the equilibrium constant of adsorption (l/mg).

The Langmuir constants, qm, and Ka, are calculated from the slope and intercept of the 1/qe vs. $1/C_e$ plot.

The separation factor, denoted as RL, is a dimensionless constant that represents the fundamental properties of the Langmuir isotherm. It may be calculated using equation (5) as described by [52].

$$RL = \frac{1}{1 + \text{KaC}a} \quad \text{... (5)}$$

Applying the Freundlich model by the equation (6).

$$qe = K_F C_e\, 1/n \quad \text{... (6)}$$

A linearized version of the above equation is given in Eq (7):

$$\ln q_e = \ln K_F + (1/n)\ln C_e \quad \text{...(7)}$$

Where:

K_f: Freundlich adsorption constants showing adsorption capacity (mg/g)

N: Constants of Freundlich adsorption(l/mg)

C_e: is the equilibrium concentration(mg/l)

q_e: is the amount of sorbent adsorbed per unit mass at equilibrium(mg/g).

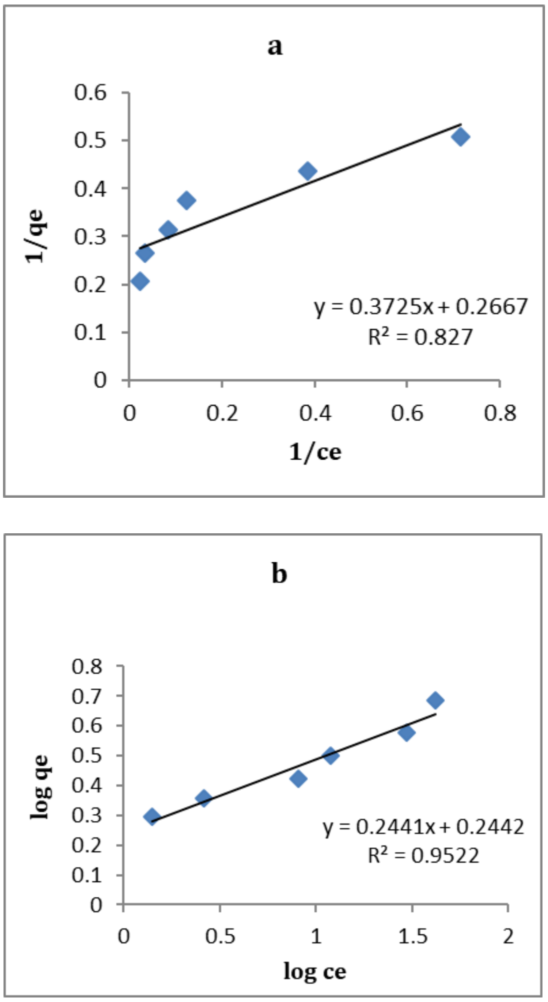


Fig. 11. Linear form of the isotherm models for sorption of (MB) onto AC (a) Langmuir, (b) Freundlich for cane physical AC.

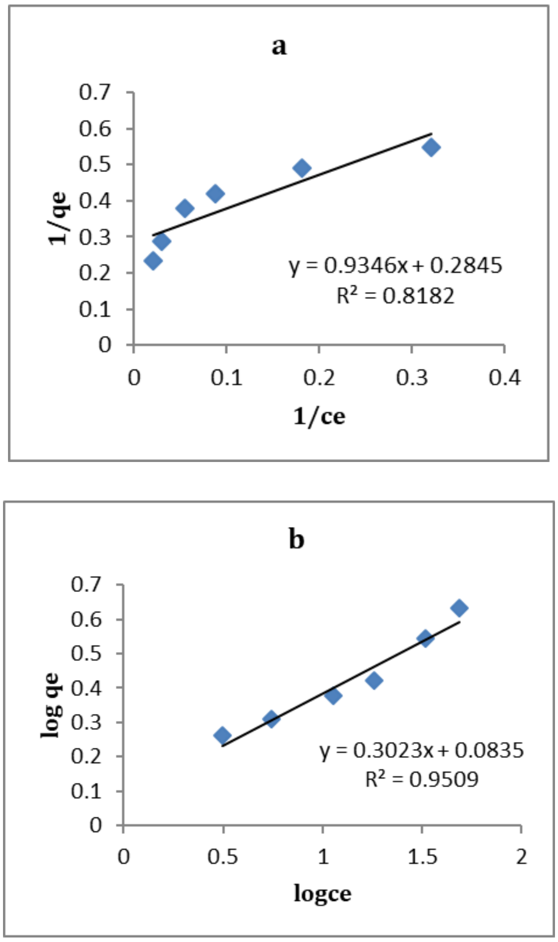


Fig. 12. Linear form of the isotherm models for sorption of (MB) onto AC (a) Langmuir, (b) Freundlich for cane chemical AC.

Table 2,
Parameters of Langmuir and Freundlich equations for sorption of (MB) on AC

Model	Parameters	AL Haji physical activated carbon	AL Haji chemical activated carbon
Langmuir	qmax (mg/g)	5.238345	5.47046
	Ka (L/mg)	0.256759	0.096791
	R^2	0.777	0.9686
Freundlich	Kf (mg/g)	1.646645	1.291814
	N	0.3384	3.1210
	R^2	0.7271	0.9341

The analysis of the data presented in Table 2 reveals that the R^2 values linked to the Langmuir model were superior than those of the Freundlich model. These findings indicate that the adsorption of (MB) on activated carbon (AC) can be better described by the Langmuir model rather than the Freundlich model for AAC. The present work provides evidence of a monolayer adsorption phenomena occurring within a system that is compatible and uniform, thereby correlating with the results previously documented by[53].

5. Sorption Kinetic Models

The adsorption kinetics onto the AL Haji were determined using pseudo-first-order and pseudo-second-order models, as shown in Figures 13,14 and Table 3. Indeed, both models effectively explain the correlations between the empirical and theoretical findings The first-order kinetic model is often used to describe adsorption processes where the rate of adsorption is proportional to the concentration of adsorbate (molecule or ion) remaining in the solution. This model assumes that adsorption occurs on a surface with a finite number of identical sites, and the adsorbate is assumed to form a monolayer on the surface without interaction between adsorbate molecule while The pseudo-second-order kinetic model is widely used to describe the adsorption of solutes (adsorbents) onto solid surfaces (adsorbents). This model is often more suitable for describing the adsorption process in many real-life applications compared to the first-order model, especially when the adsorption is not simply proportional to the concentration of the adsorbate [54, 55] Equivalent equations (8) and (9) denote the non-linear manifestations of pseudo-first-order and pseudo second-order kinetics, correspondingly [56].

$$\frac{dqt}{dt} = k_1\,(qe - qt) \qquad \text{... (8)}$$

$$\frac{dqt}{dt}\,dt = k_2\,(qe - qt)^2 \qquad \text{... (9)}$$

The values of qt and qe (mg/g) indicate the quantities of AC that were adsorbed onto the MB at any given time (t) and at equilibrium, respectively. The rate constants of the pseudo-first-order and pseudo second-order kinetics are denoted by the values k_1(min^{--1}) and k_2 (min^{--1}) correspondingly. Equations (10) and (11) provide the linearized definitions of the pseudo-first-order and pseudo-second-order, respectively[26, 57].

$$\frac{dqt}{dt}\,dt = k2\,(qe - qt)^2 \qquad \text{... (10)}$$

$$\frac{t}{qt} = \frac{1}{k2qt} + \frac{t}{qt} \qquad \text{... (11)}$$

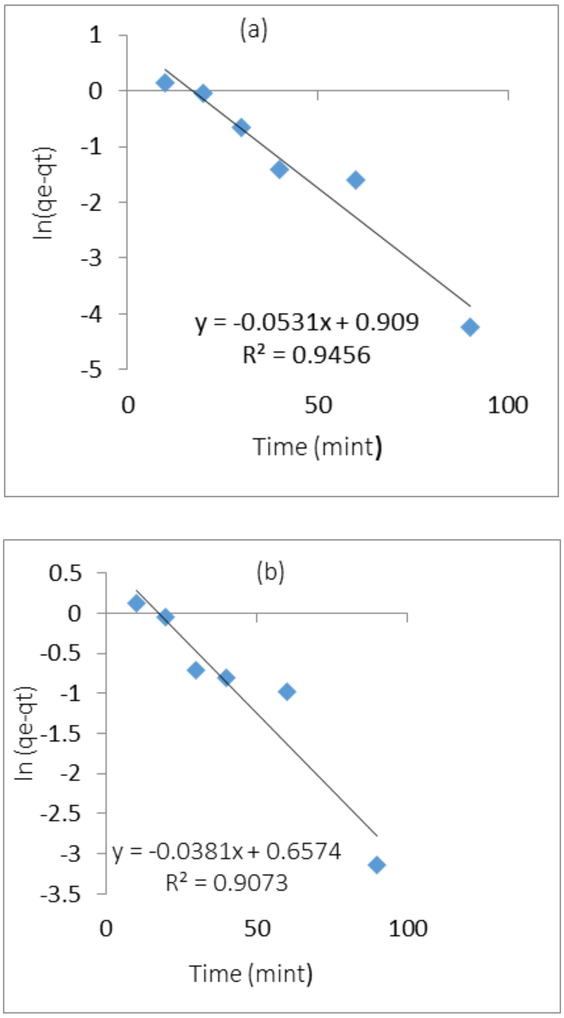
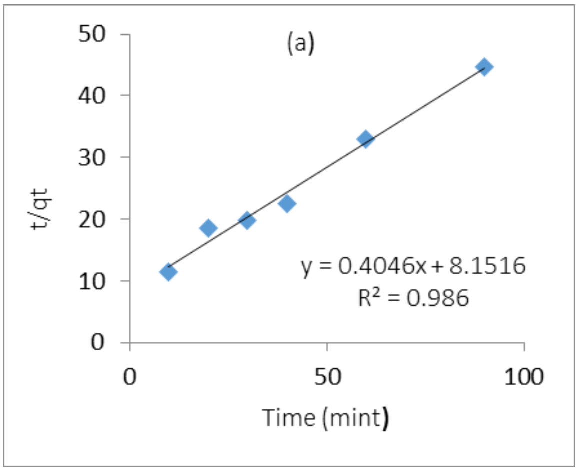


Fig. 13. Kinetic model for sorption of MB on AL Haji (a)pseudo- First order kinetic for physical activated (b) pseudo- First order kinetic for chemical activated



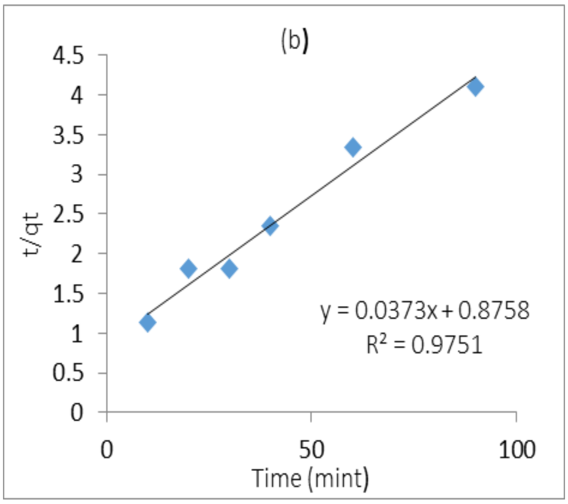


Fig. 14. Kinetic model for sorption of MB on AL Haji(a)pseudo- Second order kinetic for physical activated (b) pseudo- Second order kinetic for chemical activated

Table3,
Adsorption kinetics parameters of pseudo-first-order rate and pseudo-second-order rate equations for (AL Haji plant)

Model	**Parameters**	**AL Haji AAC**	
		physical AC	chemical AC
Pseudo-first order	K_1 (min^{-1})	-0.00059	-0.00042
	qe (mg/g)	-18.8324	-26.2467
	R ^{2}	0.9456	0.9073
Pseudo-second order	K_2 (mg/g min)	0.020082	0.001589
	qe (mg/g)	2.471577	26.80965
	R ^{2}	0.986	0.9751

Based on the previous analysis, the pseudo-second-order model demonstrates a higher capability to describe the adsorption process in the studied system. This is not only due to the high $(R)^2$ values but also because of the positive and logical values of the adsorption capacity (qe) and the rate constant (K). These results reflect the multifaceted nature of the adsorption mechanism and highlight the importance of considering multiple factors when selecting the appropriate model.

6. Thermodynamic Parameters

Three thermodynamic parameters, namely enthalpy change ΔH^o, Gibbs free energy change ΔG^o, and entropy change ΔS^o, were examined to analyse the thermodynamic behaviour of the relationship among the thermodynamic parameters enthalpy change ΔH^o, entropy change ΔS^o, and Gibbs free energy change ΔG^o is defined by the equation(12) [58, 59] .

$$\Delta G^o = RTl(K_C) \qquad \ldots (12)$$

where R is the universal gas constant (R =8.314 $kJ/kmol.\ K$), T is the absolute temperature of the solution (K), and K_C is the distribution coefficient that can be calculated by Eq. (13).

$$K_C = \frac{qe}{Ce} \qquad \ldots (13)$$

Where q_e represents the quantity of adsorbed Methylene blue per unit weight of activated carbon at equilibrium concentration (mg/g), and C_e denotes the equilibrium concentration of Methylene blue (mg/l). The relationship between the thermodynamic parameters, enthalpy change (ΔH°) and entropy change (ΔS°), determines the spontaneity of the adsorption process. Consequently, the alteration in Gibbs free energy, ΔG^o, is delineated by the equation (14)

$$\Delta G^o = \Delta H^o - T\Delta S^o \qquad \ldots (14)$$

Substitution Eq. (10) in Eq. (8) gives

$$\ln Kc = -\frac{\Delta So}{R}\frac{\Delta Ho}{RT} \qquad \ldots (15)$$

The values of $\ln(K_C)$ were calculated by Eq. (9) at different temperatures. Plotting $\ln(K_C)$ vs. 1\t gives a linear relation from which the values of ΔH^o and ΔS^o are calculable. Fig 15 illustrates that the value of Kc escalates with an increase in temperature parameters ΔG° , ΔH° , and ΔS° at various Temperatures for the adsorption of Methylene blue onto AAC.

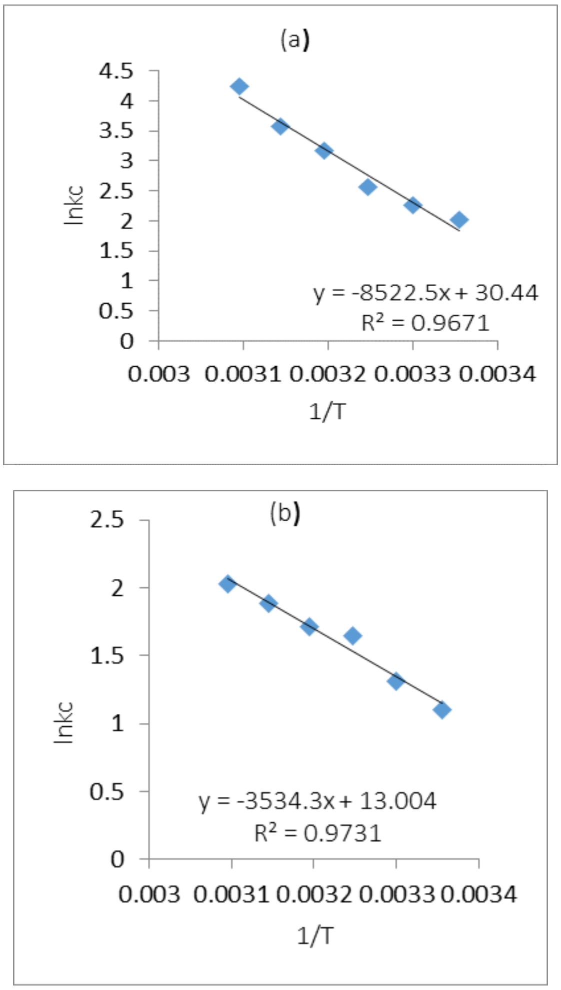


Fig. 15. Thermodynamic model for sorption of MB on AL Haji (a)physical activated carbon (b) chemical activated carbon

Table 4,
Thermodynamic parameters for the adsorption of MB on AC (AL Haji)

T(C)	physical activated carbon			chemical activated carbon		
	ΔG° (kJ /mol)	ΔS° (J.mol^{-1} .k^{-1})	ΔH° (kJ /mol)	ΔG° (kJ /mol)	ΔS° (J.mol^{-1} .k^{-1})	ΔH° (kJ /mol)
298	-5.02223	253.07816	70.85607	-2.73391	108.1153	29.384
303	-5.69883			-3.30176		
308	-6.58155			-4.20247		
313	-8.24698			-4.46239		

The adsorption process for both physical and chemical activation is endothermic, as indicated by the positive ΔH values meaning it requires heat energy for the adsorption to occur. This explains why adsorption increases with higher temperatures, as heat energy supports the process. The negative values of ΔG indicate that the adsorption process is spontaneous at the studied temperatures. As the ΔG values become more negative with increasing temperature, this suggests that the process becomes more favourable and spontaneous at higher temperatures. The positive values of ΔS mean an increase in randomness or entropy when reacting. In the context of adsorption, this means that the particles of the adsorbent (solid surface) and the adsorbed (methylene blue) move to a more random state after absorption, such as the random distribution of particles on the surface [60, 61].

7. Conclusions

In this study, the AL Haji plant was successfully used for the removal of methylene blue dye from aqueous solutions. The results indicate that the plant's physical and chemical properties played a significant role in the adsorption process. The plant demonstrated good potential as an eco-friendly and cost-effective adsorbent material for the removal of methylene blue, achieving notable decolourization and reduction of dye concentration. Physicochemical Characteristics the adsorption process was influenced by various factors such as the plant's surface area, functional groups, and particle size. The plant's cellulose and lignin content, along with its natural surface charge, may have contributed to the dye adsorption efficiency. This achieved a 98.552% removal of methylene blue with a mixing duration of 90 minutes, a pH of 4, a stirring rate of 400 rpm, a dosage of 3 g/100 ml of activated carbon, and an initial methylene blue concentration of 25 ppm. The results suggest that the adsorption process likely involves a combination of physical and chemical interactions between the dye molecules and the surface of the adsorbent. The study suggests that both Van der Waals forces and electrostatic interactions might be involved in the removal process. Comparative effectiveness when compared to other traditional adsorbents, the AL Haji plant appears to be a competitive alternative, offering a more sustainable approach to treating wastewater contaminated with dyes like methylene blue.

8. Limitations and Challenges

1. Necessity for Additional Inquiry into Adsorption Mechanism: Although the study yielded encouraging outcomes, further comprehensive investigations are required to thoroughly clarify the adsorption mechanism. Comprehending the specific nature of the interactions (e.g., ion-exchange, hydrogen bonding, or surface complexation) is essential for enhancing the material's efficacy.
2. While a single variable change approach was used to study individual effects, the interactions between variables were not examined in this research. It is suggested that a more comprehensive experimental design (such as full factorial design) be used in the future to study the possible overlaps between different factors.
3. Lack of Comprehensive Statistical Analysis: The study did not include a thorough statistical analysis of the data collected during the experiments. Statistical analysis is essential for determining the reliability of the results and assessing variability in performance across different samples. For instance, analysis of variance (ANOVA) or t-tests could be used to test for significant differences between means and to ensure that the results are not due to chance. Additionally, statistical analysis could help identify the most influential factors on the adsorption process and contribute to optimizing the experimental design.
4. The study did not include a control experiment using the raw, untreated material. This was a deliberate decision, as the primary focus was on evaluating the enhancements achieved through chemical and physical activation processes. However, including a control experiment could provide additional insights and may be considered in future research.

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إزالة الميثيلين الازرق من مياه الصرف الصحي بواسطة نبات الحاجي كمادة مازة منخفضة التكلفة وصديقة للبيئة

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المستخلص

يبحث هذا العمل في استخدام النفايات الزراعية وتحديدا ساق نبات الحاج، يشير مصطلح "نبات الحاج" غالبًا إلى نباتات من جنس الحاج ، وخاصة Alhagi maurorum أو Alhagi camelorum، وهي نباتات صحراوية تنتمي إلى عائلة البقوليات. تُعرف هذه النباتات بقدرتها على التكيف مع البيئات الجافة وفروعها الشائكة. تُستخدم تقليديًا في الطب الشعبي لخصائصها المضادة للالتهابات والمدرة للبول والمضادة للميكروبات. وفي بعض الثقافات، يرتبط هذا النبات بـ"المنّ"، وهو إفراز سكري يتشكل على أغصانه. الذي يتم إنتاجه عن طريق تحويل مواد النفايات إلى مواد ممتازة بيولوجية فعالة. تعتبر هذه المواد الممتزة موردا مجديا تقنيا واقتصاديا لمعالجة مياه الصرف الصحي. تم وصف عملية تحضير الكربون المنشط (AC) المصنوع من بقايا نبات الحاجي. تم التحقيق في الخصائص الفيزيائية والكيميائية وكفاءة امتصاص الكربون المنشط المنتج. التنشيط باستخدام هيدروكسيد الصوديوم (NAOH) والكربنة عند ٣٠٠ درجة مئوية هما الخطوتان الرئيسيتان في تصنيع التيار المتردد. تم استخدام الكربون المنشط لامتصاص الميثيلين الأزرق (MB). تمت دراسة معلمات مختلفة في درجة حرارة الغرفة لإظهار آثارها على كفاءة امتزاز الميثيلين الأزرق. تم فحص تأثير المعلمات التشغيلية الرئيسية، بما في ذلك الرقم الهيدروجيني والوقت ومعدل التحريك وتركيزات الصبغة الأولية وجرعة الممتزات ودرجة الحرارة على عملية إزالة الصبغة. تم إجراء فحص للممتازات الحيوية باستخدام المجهر الإلكتروني المسح الضوئي (SEM) والأشعة تحت الحمراء المحولة من فورييه (FTIR) ومساحة سطح بروناور وإيميت وتيلر (BET). يظهر تحقيق المجهر الإلكتروني المسح الضوئي (SEM) مساما ملحوظة في عينة نبات الحاج التي يمكن أن تعزز امتصاص صبغة الميثيلين الأزرق (MB). تشير النتائج إلى أن مساحة سطح BET للكربون المنشط المادي هي ٢٥,٥٧٩٦ م^2/جم. تم الإبلاغ عن مساحة سطح الكربون المنشط الكيميائي على أنها ١٦,٢٧٦٧ m^2/g $m2g{-}1$. أظهرت الصبغة حركية امتزاز زائفة من الدرجة الأولى، مع متوسط ثوابت معدل ٠,٠٢٠٠٨٢ و٠,٠٠١٥٨٩ لنهج التنشيط الفيزيائي والكيميائي، على التوالي. أجريت دراسة متساوي الحرة باستخدام متساوي الحرة لامتزاز Langmuir بقيمة R^2 أكبر من ٠,٨٢٧، مما أدى إلى قدرة امتزاز قصوى تبلغ ٥,٢٣٨٣٤٥ ملغ g-1. يوضح التحقيق في الديناميكا الحرارية للامتزاز أن امتزاز MB على AAC ماص للحرارة وعفوي.