



Adsorptive Removal of Methylene Blue From Aqueous Using Moringa Oleifera Extract Loaded Activated Carbon

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Abstract

This study investigates the removal of methylene blue (MB) dye from aqueous solutions using activated carbon (AC) and Moringa oleifera leaf extracts loaded onto AC using hexane (MS-AC) and water (MW-AC). Key operational parameters such as dye concentration, contact time, pH, and adsorbent dosage were optimized. The MS-AC sample achieved 100% MB removal, showing superior performance compared to MW-AC and unmodified AC with a lower dose (0.05 g/L), indicating a much more efficient adsorbent. Adsorption equilibrium followed the Langmuir isotherm model, indicating monolayer adsorption, with high correlation coefficients ($R^2 = 0.9931$ for MS-AC). Freundlich and Dubinin–Radushkevich (D–R) models supported additional surface heterogeneity and confirmed that the process is endothermic and physical in nature. These findings highlight the potential of MS-AC as a low-cost, effective adsorbent for wastewater treatment applications.

Keywords: Adsorption; wastewater treatment; low-cost materials; solid waste management; kinetics and isotherms

1. Introduction

The increasing rate of pollution leads to depletion of Freshwater resource. Water pollution and poor water resource management have become a serious global concern. Colored wastewater is one of the main issues with water contamination, especially in developing nations [1]. These comprise the waste streams from sectors like the food, pharmaceutical, plastic, rubber, textile, leather, paint & pigment, and cosmetics industries.[1, 2]. The rise in dye production in bulk worldwide led to an increase in dye pollution. Meanwhile due to their carcinogenicity, toxicity, mutagenicity, and disruption of aquatic plants' photosynthesis, dye pollution can have a variety of negative consequences on human health as well as ecological degradation[3, 4]. The most common dyes in the textile industry are azo dyes, however because of their toxicity and poor biodegradability, they are categorized as environmentally harmful compounds [4, 5]. Thus, the elimination of azo dyes from the aquatic environment is important for protecting both the environment and public health. Numerous techniques were projected to eliminate dyes from wastewater including chemical precipitation, coagulation, ion exchange, membrane filtration, flocculation, solvent extraction, anaerobic/aerobic biological degradation, precipitation, and advanced oxidation processes. However, it was discovered that these techniques were ineffectual since they resulted in secondary waste[1, 2, 4, 6]. Adsorption is a preferable option because it has demonstrated great potential for dye recovery and removal from wastewater. It is controlled by straightforward operations and adsorbent regeneration. Adsorption also has a positive reputation as a water treatment technique because of its many advantages, including low cost, wide-scale use, ease of use, insensitivity to harmful substances, adaptability, accessibility of different adsorbents, and high efficacy as a practical and economical dye removal method [1, 2, 5, 6]. However, the adsorption process depends on the selection of an economical and effective adsorbent. Numerous inexpensive materials, such as natural materials [7, 8], humic acids [9, 10], algae [11, 12], clay minerals [13-16], magnetic particles, viscose fiber, and wool fiber waste [17-21], were employed as adsorbents. Also activated carbon which prepared from Moringa oleifera leaf or Bark was applied for removing methylene blue (MB) or *Escherichia coli* and *Pseudomonas aeruginosa* from aqueous solution [22, 23]. Due to their ease of production, cost-effectiveness, large surface area, and non-toxicity, activated carbons are becoming increasingly popular as adsorbents in dye removal processes. Their high adsorption capacity and effectiveness in eliminating contaminants from aqueous solutions have made them a preferred choice in water treatment applications. Recently, adsorption techniques utilizing activated carbon have achieved substantial attention in years for their efficiency in purifying wastewater and industrial effluents [24-26]. Despite its high cost, adsorption on activated carbon (AC) is now thought to be the most popular and successful physical technique for removing pesticides and dyes from industrial wastewater [27]. The objective of the current work is to remove MB from aqueous solutions under various conditions using activated carbon (AC), hexane, and aqueous moringa extract loaded on AC (MS-AC&MW-AC) made from Moringa oleifera leaf as renewable, and inexpensive adsorption method.

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Several methods were used to describe the generated samples, and their effectiveness in eliminating methylene blue (MB) from an aqueous solution was thoroughly investigated. The isothermal models were employed to assess the adsorption data.

2. Materials and Methods

2.1. Adsorbate

Methylthioninium chloride (Methylene blue, $C_{16}H_{18}N_3SCl \cdot 3H_2O$) was supplied by Merck and was employed with no additional refinement. To prepare the MB-solution, the needed quantity of MB-dye was dissolved in distilled water.

2.2. Adsorbent

Moringa oleifera leaf was obtained from a local plant in Saudi Arabia, washed multiple times with distilled water to eliminate adherent contaminants from its surface, and then refluxed in water or hexane for four hours before cooling to room temperature. As a result, the extract was refluxed with 10 gm of commercial activated carbon for 2 hours. The samples produced were designated as AC, MS-AC, and MW-AC based on the solvent employed for extraction.

2.3. Experimental

KBr plates were employed to measure of the IR spectra using a Jasco FT/IR-6100A infrared spectrophotometer instrument-Japan, across a spectrum ranging from 400 to 4000 cm^{-1} . The analyses of pore volume and surface area were achieved utilizing nitrogen adsorption at 77 K by a BEL sorp max (BEL Japan Inc). These analyses data were computed utilizing the Brunauer–Emmett–Teller method. **Adsorption studies.**

The methylene blue (Figure 1) was employed as an adsorbent in present investigation. 1000 mg/L stock solution of methylene blue was prepared by dissolving 1.00 gm methylene blue in deionized water. On diluting the stock solution, various working solutions were generated, and their absorbance was determined at wavelength 665 nm by UV–visible spectrophotometer (Shimadzu UV- 2600). The absorbance measurements were plotted versus concentration to produce a standard curve with a correlation coefficient (R^2) of 0.99 (Figures. 2-3).

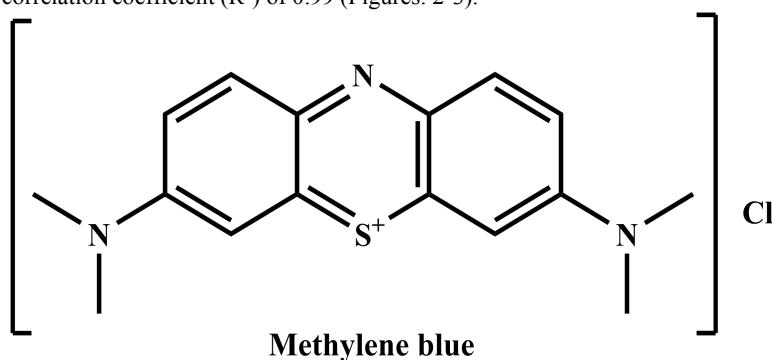


Figure 1: Methylene blue dye structure worked as an adsorbent

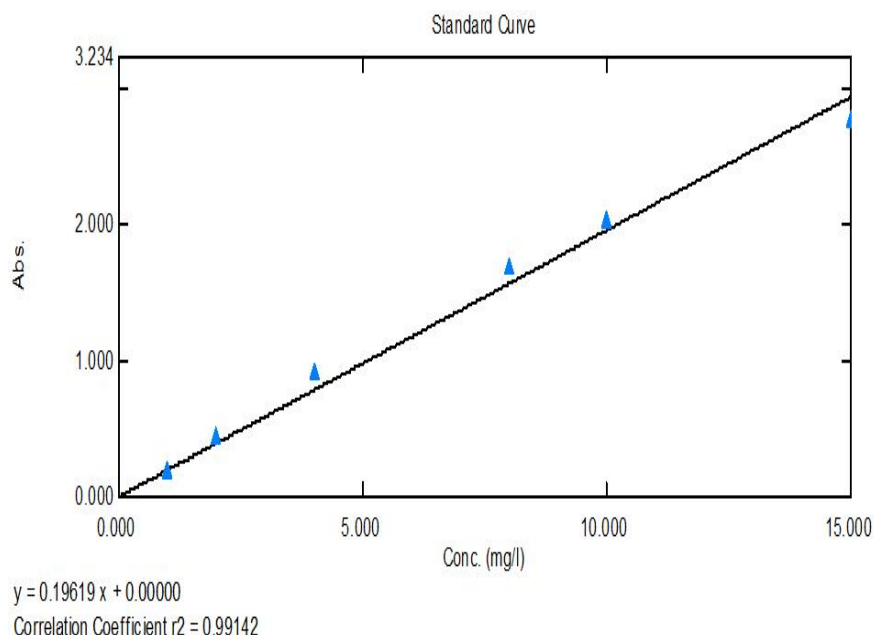


Figure 2 standard curve for MB dye

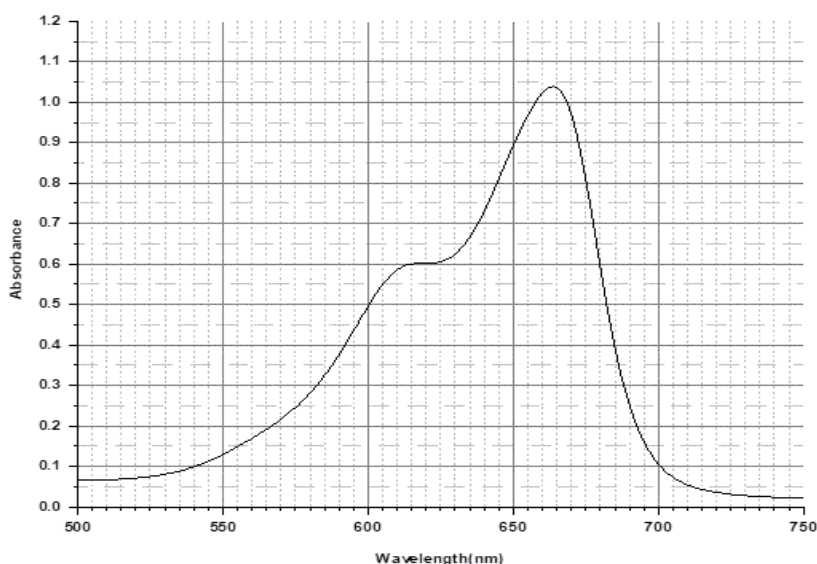


Figure 3: UV-Visible spectrum of MB dye.

The adsorption experiments batch were implemented in 250 mL stopper conical flasks at room temperature (25°C). A certain adsorbent weight was dipped in 100 mL of methylene blue solution and shaken at 120 rpm with mechanical shaker (SK-L180 pro, China). The effect of adsorbent dosage, contact time, dye concentration and pH were investigated to achieve the optimal conditions. Samples were withdrawal at specific time intervals, filtered off, and the leftover concentration of methylene blue in the filtrate was determined as described above.

All experiments were repeated three times, and the average values of the results were computed. The percentage relative standard deviations were calculated, and any number more than 5% was disregarded.

2.3.1. Effect of adsorbent dosage

At the equilibration time, each adsorbent was given a dosage ranging from 0.1 to 1 g/L in a volume of 100 mL of 50 mg/L dye solution.

2.3.2. Effect of pH

To investigate the effect of acidic and alkaline media on the process of adsorption, various initial pH (2, 5, 7, 9, and 11) were generated and used at optimal time and dosage of MB adsorption on the prepared samples.

2.4. Adsorption isotherms

Adsorption isotherm experiments were performed by shaking dye solutions of different initial concentrations (25–125 mg/L) using the optimum dosage of each adsorbent at the optimum time and pH. Freundlich, Langmuir and Dubinin–Radushkevich (D–R) isotherm are the three different adsorption isotherms that were used to describe the data of MB adsorption onto the activated carbon (AC) as well as the hexane and aqueous moringa extract loaded on AC (MS-AC&MW-AC) [28].

2.4.1. Langmuir and Freundlich isotherms

The equations of Langmuir and Freundlich are the most commonly applied models for describing the relationship between equilibrium dye uptake (q_e) and final concentrations (C_e) at equilibrium [29]. Langmuir adsorption isotherm proposes a monolayer adsorption on the surface of adsorbent with linear form as follows:

$$C_e/C_{ads} = (1/Q_b) + (C_e/Q) \quad (1)$$

where C_{ads} is the amount of dye adsorbed per unit mass of the adsorbent and C_e (mg/L) is the concentration of dye in solution at equilibrium. Langmuir constants b and Q are linked to the adsorption energy and the monolayer adsorption capacity, correspondingly.

2.4.2. Freundlich Isotherm

Freundlich isotherm is more common than Langmuir isotherm, suggesting surface heterogeneity and forming of multiple layers on the adsorbent surface.

The Freundlich equation logarithmic form is as follows:

$$\text{Log } q_e = \text{log } K_f + (1/n) \text{log } C_e \quad (2)$$

where K_f and n are the Freundlich constants, which are linked to the adsorption intensity or heterogeneity and the adsorption capacity of the adsorbent.

2.4.3. Dubinin-Radushkevich (D–R) isotherm

The D–R isotherm is more common than Langmuir isotherm since it does not presuppose a homogeneous surface or constant potential of adsorption [30]. The nature of adsorption process either chemical or physical can be predicted from the D–R model. The linear form of the D–R isotherm equation can be expressed as follows:

$$\ln q_e = \ln X_m - \beta \epsilon^2 \quad (3)$$

where X_m is the maximum adsorption capacity; q_e is the amount of dye adsorbed per unit mass of adsorbent (mol/g); β is the activity coefficient related to the mean adsorption energy; and ϵ is the Polanyi potential, which is given by:

$$\epsilon = RT \times \ln(1 + (1/C_e)) \quad (4)$$

where R is the general gas constant (J/mol K), and T is the absolute temperature (K). The sorption energy can also be calculated using the following equation:

$$E = \frac{1}{\sqrt{-2\beta}} \quad (5)$$

3. Results and Discussion

3.1. Prepared samples characterization

3.1.1. FT-IR spectra

FTIR-analysis was utilized to identify some distinctive functional groups of activated carbon and moringa extract loaded on AC before and after the methylene blue adsorption. The FT-IR of AC sample displayed a broad absorption peak in the range 3050-3500 cm^{-1} which assigned to the stretching vibration of O-H of alcoholic, phenolic and carboxylic moieties, thus viewing the occurrence of "free" hydroxylic groups on the surface of adsorbent. The other peaks in the 2600-3000, 1550-1750, 1550-1480, and 1150 cm^{-1} may be allocated to the stretching vibration of -CH, C=O, C=C, C-O correspondingly [31]. There is no discernible difference in the FTIR before and after the adsorption process, implying that the adsorption may occur physically on the surface of the AC sample in the present investigation (Figure 4). But in case of moringa extract loaded on AC (MS-AC & MW-AC) there is a decreasing in the intensity of the bands point to the adsorption may occur chemically on the surface of moringa extract loaded on AC (MS-AC & MW-AC) samples (Fiures 5-6)[32].

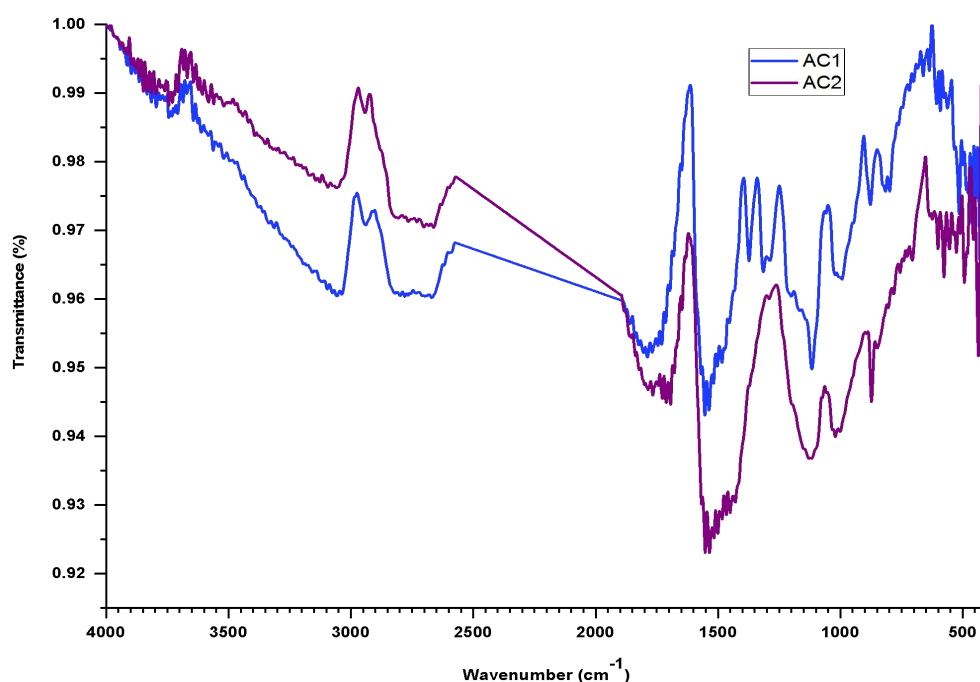


Figure 4: FTIR analysis of the activated carbon before (AC1) and after (AC2) the adsorption of methylene blue

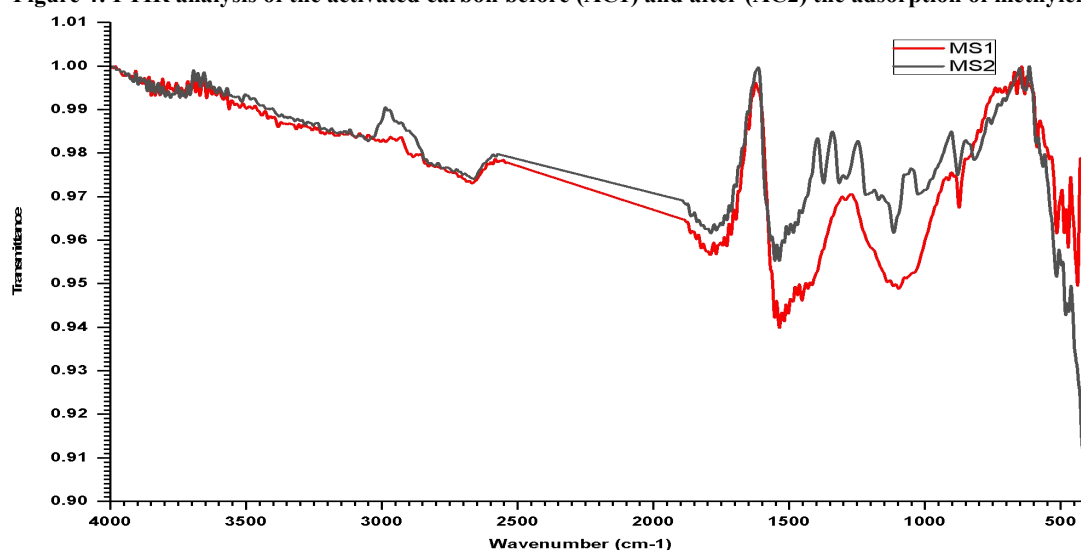


Figure 5: FTIR analysis of the hexane extracted of moringa before (MS1) and after (MS2) the adsorption of methylene blue.

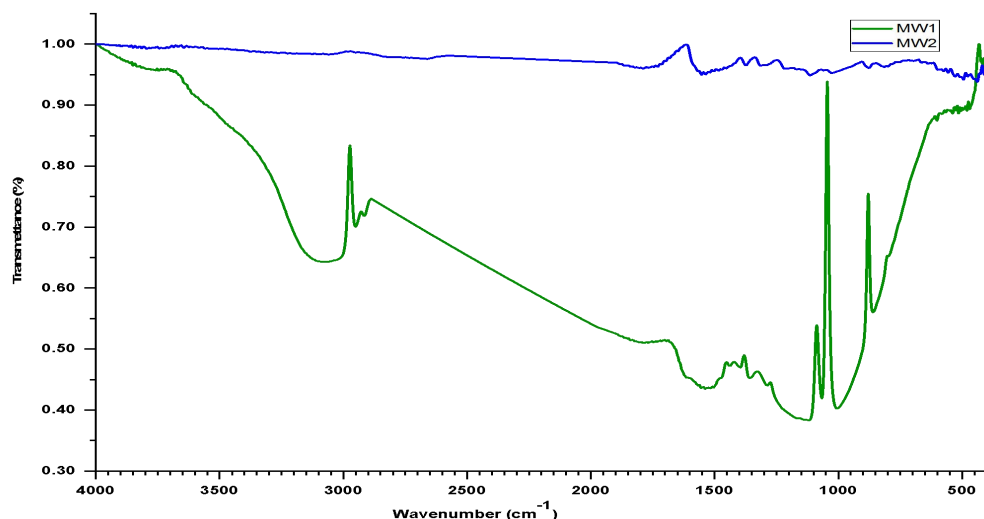


Figure 6: FTIR analysis of the aqueous extract of moringa before (MW1) and after (MW2) the adsorption of methylene blue.

3.1.2. BET specific surface area.

The nitrogen adsorption desorption data are presented in table 1. Their values indicate that the BET specific surface area was 761.2 and 722.6 m²/g for the MS-AC and MW-AC, respectively. It is noted that the BET surface area of moringa extract by water loaded on AC is less than that extract by hexane indicated the solvent effect on the BET surface area. The values of pore volume and pore size do not differ significantly between the two samples.

Table 1. Some characteristics of the prepared moringa extract loaded on AC.

Sample	Pore volume (cm ³ /g)	Pore diameter (nm)	S _{BET} (m ² /g)
MS-AC	0.3978	2.09	761.2
MW-AC	0.4042	2.24	722.7

3.2 Adsorption Studies

3.2.1 Effect of contact time

The contact time effect on the elimination of MB using the activated carbon and the moringa extract loaded on AC are displayed in Figure 7. The adsorption process consists of two phases. The first phase is quick and occurs within the first five minutes. Then the rate of adsorption gradually slows down in the second stage until the equilibrium is reached. The optimal contact time for three samples (MW-AC, MS-AC, AC) were found to be equal 5 min, 15 and 60 min respectively, therefore, these optimal contact time will be applied for the remainder of experiments. Figure 7 shows that MS-AC eliminated nearly 100% of the MB after 40 minutes of contact time, whereas other samples removed 95.56% and 58.4% after 60 minutes and 5 minutes, respectively.

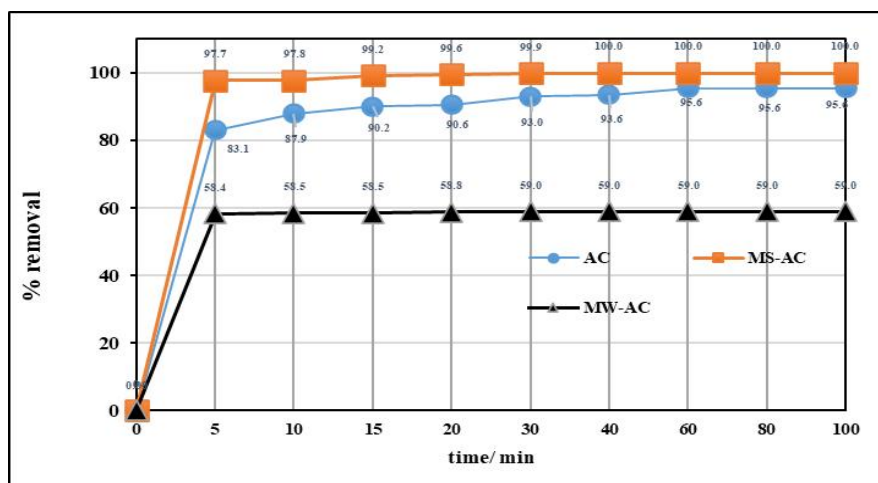


Figure 7: Effect of contact time for the removal of MB by the Activated carbon and the moringa extract loaded on AC (MB conc. 50 mg/L, dosage 0.05 g/L pH 7)

3.2.2 Effect of dose

Figure 8 showed the adsorbent dosage effect on the elimination of MB utilizing the Activated carbon and the moringa extract loaded on AC. The results discovered that, the MB removal percentage was raised with raising the three adsorbents dosage used due to the increase of the surface area and availability of more binding sites until a certain optimal dose where the adsorption reached equilibrium and levelled off due to the formation of aggregates at higher dosages [10]. MS-AC is the most effective adsorbents as noted in Figure 8. It attained 100% removal of the MB at dosage 0.05 g/L. However, at dosages of 0.08 and 0.1 g/L, the AC and MW-AC removed the dye by 95.6% and 57.9% respectively. MS-AC demonstrated the greatest removal percentage of MB because of the extraction with hexane may be led to increase the extraction of non-polar components from Moringa leaf, which in turn are linked to AC. This indicates to the adsorption MB by these non-polar groups. This character increases the chemical adsorption.

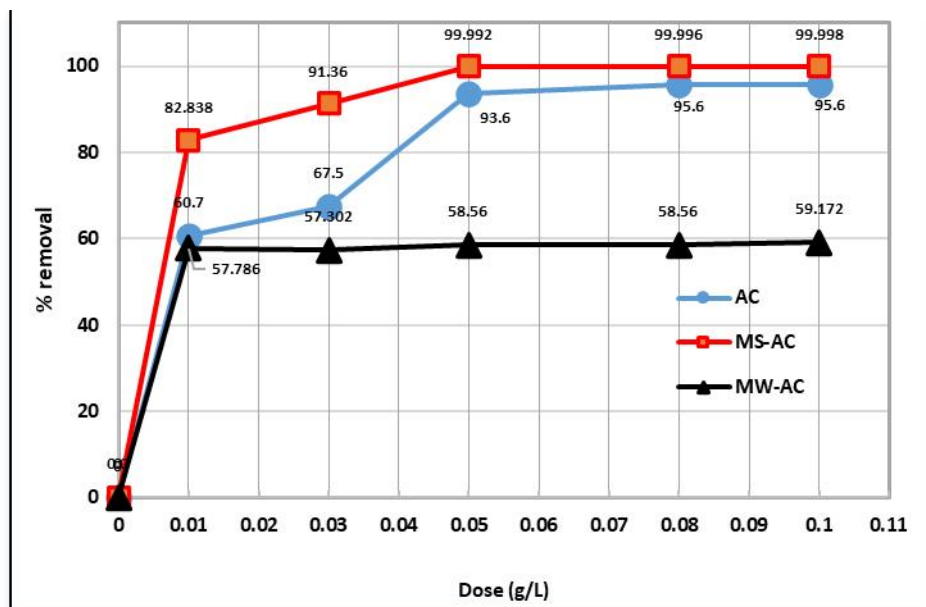


Figure 8: Dosage effect on the elimination of MB by the Activated carbon and the moringa extract loaded on AC (MB conc. 50 mg/L, contact time; 5, 40 and 60 min for MW-AC, MS-AC and AC, pH 7)

3.2.3 Effect of pH

The pH has an essential role in the adsorption process due to the difference of the charges existed on the surface of adsorbent in different pH medium which may increase or decrease the efficiency of the adsorption process. The effect of pH variation on the adsorption of MB by the Activated carbon and the moringa extract loaded on AC samples is presented in Figure 9. It was found that there are two trends can be seen, the 1st trend demonstrated by sample of MW-AC which displayed no effect of pH change on the adsorption process. The 2nd trend exhibited by the AC and MS-AC samples in which, there is a great adsorption of MB at strong acid medium (pH =2) consequently decreases at pH 5 then steady at pH =7, because of the strong attraction of MB functional groups and the surface of activated carbon.

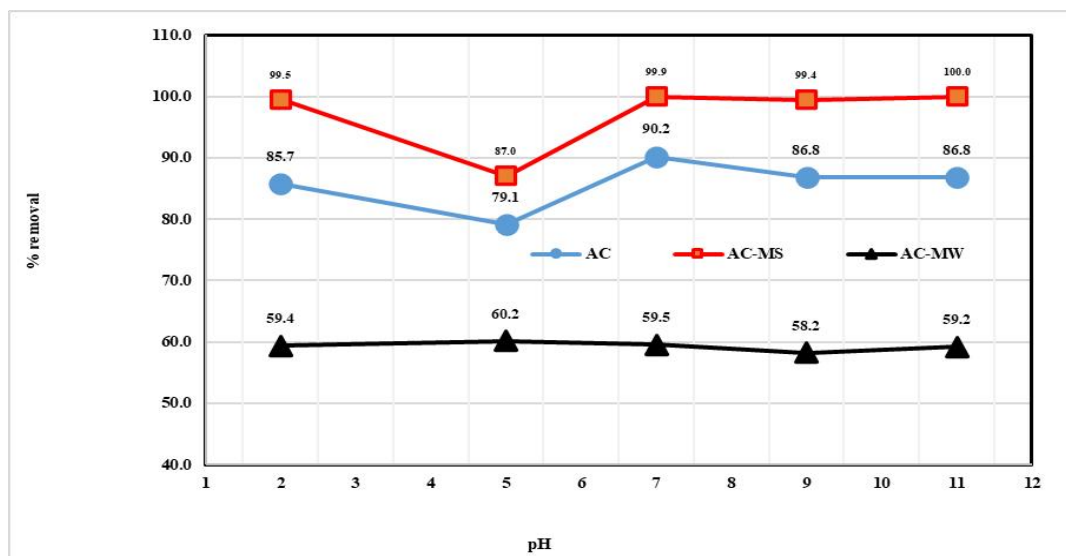


Figure 9: pH effect on the MB adsorption by the Activated carbon and the moringa extract loaded on AC (MB conc. 50 mg/L, contact time; 5, 40 and 60 min for MW-AC, MS-AC and AC)

3.3 Isotherm models

Langmuir isotherm

According to the Langmuir isotherm model, adsorption occurs uniformly on the active sites of the adsorbent, and once the adsorbate has occupied a particular site, no more adsorption can occur there [29]. The affinity between the adsorbent and adsorbate can be determined using the Langmuir isotherm's parameters. The adsorption of MB onto the Activated carbon and the moringa extract loaded on Activated carbon (AC, MS-AC, and MW-AC) was best fitted to Langmuir isotherm with correlation coefficient values (R^2) = 0.9542, 0.9931, for AC and MS-AC, respectively (Table 2, Figure 10).

Table 2: The summary of isotherm model parameters for Moringa Olifera extract loaded activated carbons adsorbents

Sample	Freundlich model			Langmuir model		
	K_f	$1/n$	R^2	$Q_{max}(mg/g)$	$1/b$	R^2
AC	0.0237	2.6715	0.6932	70.92	38.22	0.9542
MS-AC	86,5	0.246	0.9843	169.49	0.389	0.9931
MW-AC	2.96	7.041	0.9522	9.901	24.883	0.8864

This reveals that the surface of Activated carbon and the moringa extract loaded on Activated carbon (AC, MS-AC,) was covered by a monolayer of the MB. The monolayer adsorption capacity Q_{max} in table 2 indicated the following sequence for adsorption of MB: MS-AC>AC>MW-AC. The results agree with the data obtained in Figs. 7, 8 and 9.

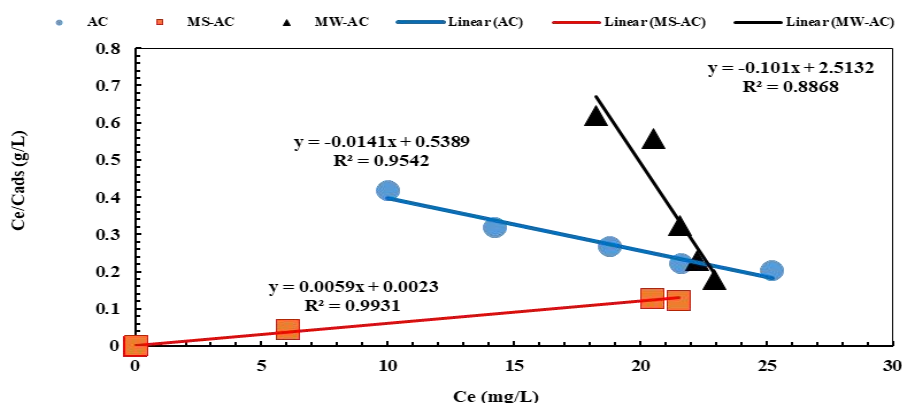


Figure 10: Langmuir isotherm for MB adsorption by the Activated carbon and the moringa extract loaded on AC (MW-AC, MS-AC)

Freundlich isotherm.

Since the Freundlich isotherm does not imply monolayer adsorption and deals with surface heterogeneity, it is more general than the Langmuir isotherm. [33]. The Freundlich parameters K_f and $1/n$ elucidate the affinity between the adsorbent and adsorbate. A high K_f value indicates that the adsorbent has a high absorption capacity, while a low value of $1/n$ indicates a high affinity between the adsorbent and adsorbate. The R^2 in Table 2 and Figure 11 for the samples (AC, MS-AC, and MW-AC) were 0.6932, 0.9843 and 0.9522 correspondingly. These results show that the Freundlich isotherm model can only explain the MB adsorption on the surface of MS-AC and MW-AC. The high affinity of the MS-AC sample for the adsorption of MB from aqueous solutions is indicated by its high K_f value (86.49). This behavior of MS-AC suggests that both the Freundlich and Langmuir models may explain the adsorption of MB onto MS-AC. The Langmuir adsorption capacity is in agreement with this conclusion.

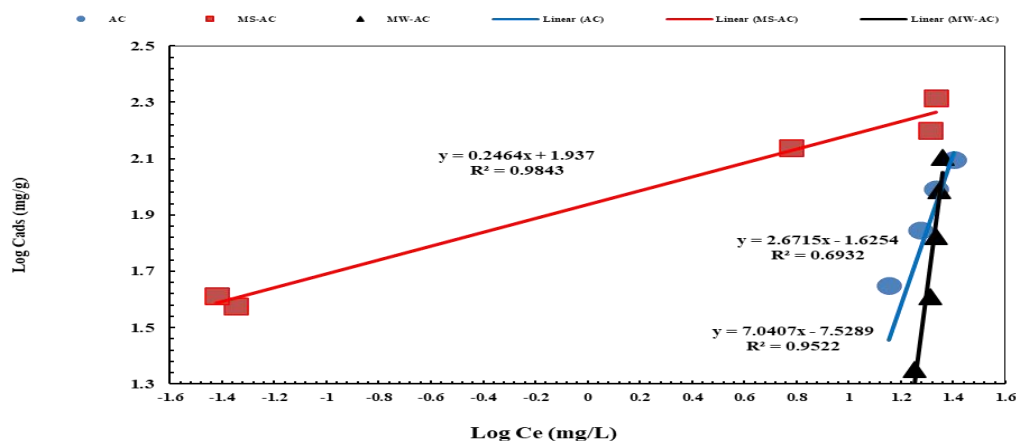


Figure 11. Freundlich isotherm for MB adsorption by the Activated carbon and the moringa extract loaded on AC (MW-AC, MS-AC)

D-R isotherms.

The adsorption mechanism was not revealed by the Langmuir and Freundlich isotherms. The D-R isotherm model can be used to forecast whether the adsorption process will be chemical or physical. Figure 12 displays the D-R plots of MB's adsorption onto the various samples. Table 3 also displays the model parameters. The adsorption's average free energy which computed from the D-R plot can imply the type of adsorption chemical or physical. The positive values of the adsorption energy ϵ in Table 3 pointed out that the MB's adsorption on the surface of Activated carbon and the moringa extract loaded on Activated carbon (AC, MS-AC, and MW-AC) samples is endothermic process[34]. The values of adsorption's average energy in Table 3 were 6.436, 16.299 and 2.613 kJ/mol for these samples (AC, MS-AC, and MW-AC) correspondingly. These adsorption free energy values <40 kJ/mol) denoted that, the adsorption process occurs physically [35].

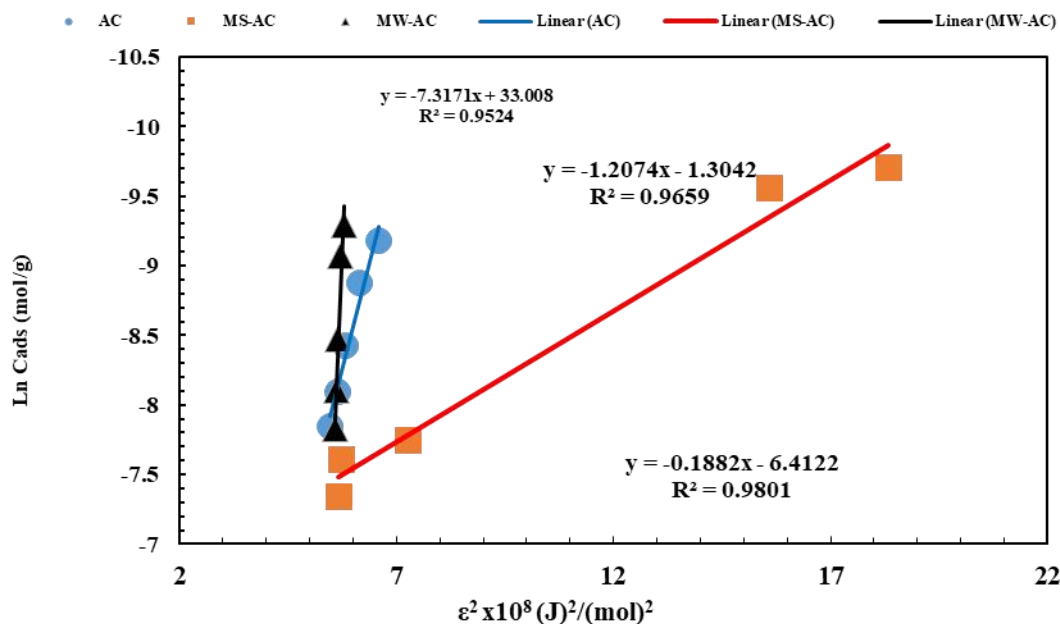


Figure 12. DKR sorption isotherm for MB adsorption by the Activated carbon and the moringa extract loaded on AC, MW-AC and MS-AC

Table 3: The summary of D-R model parameters for removal of MB

sample	Xm (mol/g)	β (mol ² /j ²)	Adsorption Energy (E, KJ/mol)
AC	0.271	-1.2074x 10 ⁻⁸	6.436
MS-AC	1.643x 10 ³	-0.1882x 10 ⁻⁸	16.299
MW-AC	2.16x 10 ¹⁴	-7.317x 10 ⁻⁸	2.613

4. Conclusion

Hexane and aqueous moringa extract loaded on AC (MS-AC&MW-AC) prepared from *Moringa oleifera* leaf has significant probable for the elimination of MB dye from aqueous solution under diverse conditions and over a widespread range of concentrations. The prepared samples' adsorptive was in the following order: AC-MS>AC>AC-MW, with 100% elimination for AC-MS. For the produced samples, the Langmuir isotherm model provided the best description of the adsorption data for AC and MS-AC samples, suggesting monolayer surface adsorption. While the Freundlich adsorption isotherm was found to be best fitted by MS-AC and MW-AC samples suggesting a heterogeneity of adsorption. It was established that the adsorption process is endothermic and occurs physically. MS-AC sample can be used as potential adsorbent for water treatment. On comparing, MS-AC&MW-AC have superior adsorption performance due to the synergistic effect of Moringa extracts with activated carbon, especially the hexane-extracted version (MS-AC) because of Higher efficiency, Faster kinetics, Lower adsorbent dose, Broader pH tolerance, Greater real-world potential [22, 23]. This study highlights the effectiveness of *Moringa oleifera* extract-loaded activated carbon, especially MS-AC, in removing methylene blue from water. Future work should focus on testing the adsorbent with real wastewater, exploring its regeneration and reuse, and expanding its application to other contaminants such as heavy metals or pharmaceuticals. Enhancing the material through modifications (e.g., metal doping or magnetization), scaling up to pilot systems, and evaluating environmental and economic impacts are also recommended. Additionally, alternative green extraction methods may improve preparation efficiency and sustainability.

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Conflicts of interest

The authors declare that they have no conflict of interest

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