Eco-Friendly Adsorptive Treatment of Methylene Blue in Aqueous Media with Agricultural Waste

Soukaina Nehhal[1], *Majda* Ben Ali[1], *Younes* Abrouki[1], *Khalid* Ofqir[1], *Souad* El Hajjaji[1]

[1]Laboratory of Spectroscopy, Molecular Modeling, Materials, Nanomaterials, Water and Environment (LS3MN2E), Department of Chemistry, Faculty of Sciences, Mohammed V University in Rabat, Morocco

Abstract. This research focuses on assessing the potential of raw artichoke waste powder as an economical and sustainable biosorbent for the removal of methylene blue (MB) from aqueous solutions. The prepared artichoke waste powder was characterized using FT-IR and XRD, confirming the presence of functional groups (hydroxyl, carboxyl, carbonyl) and a largely amorphous structure favorable for adsorption. Batch experiments were conducted to evaluate the effects of solution pH, adsorbent dosage, initial dye concentration, and contact time on MB removal. Optimal adsorption was observed at alkaline pH (10–12) and an adsorbent dosage of 0.6–1 g/L, achieving nearly complete dye removal. Adsorption isotherms were fitted using Langmuir, Freundlich, and Sips models, with the Sips model showing the lowest sum of squared errors (SSE = 156.9), indicating predominantly monolayer chemisorption with slight surface heterogeneity. Kinetic studies showed that the pseudo-second-order model (SSE = 31.41) accurately described the adsorption process, highlighting the importance of chemical interactions between MB molecules and the functional groups on the adsorbent surface. These findings demonstrate that raw artichoke waste powder is an effective, eco-friendly, and economical adsorbent for cationic dye removal from water.

Keywords: Methylene blue; Adsorption; artichoke watse powder; Langmuir isotherm; Pseudo-second-order kinetics; SSE; Chemisorption; Waste valorization

1. Introduction

Agricultural waste management is a growing environmental concern due to the large volumes of residues generated by intensive farming [1, 2, 3, 4]. These residues are often left to decompose in fields or burned openly [5, 6], contributing to air pollution, soil degradation, and groundwater contamination [7]. Among these wastes, lignocellulosic residues such as artichoke waste are abundant, renewable, and rich in cellulose, hemicellulose, lignin, and bioactive compounds [8, 9]. Recent research has highlighted the potential of untreated agricultural residues as low-cost and sustainable adsorbents for the removal of pollutants from water [10, 11]. The natural structure of these residues, including their fibrous network and inherent functional groups, allows for interactions with various organic compounds through mechanisms such as hydrogen bonding, Van der Waals forces, and electrostatic interactions [12, 13, 14]. Synthetic dyes, including methylene blue (MB), are common industrial pollutants that are chemically stable, highly soluble, and resistant to conventional wastewater treatment methods [15, 16, 17] . MB can cause significant environmental and health problems, such as toxicity to aquatic organisms and potential effects on the respiratory, digestive, and nervous systems in humans [18, 19]. The use of raw agricultural residues as adsorbents offers a cost-effective and environmentally friendly solution to remove such dyes from aqueous media [20, 21]. Artichoke waste, as a raw lignocellulosic biomass, presents a promising alternative for water purification due to its porous structure and surface functional groups capable of interacting with dye molecules [22]. This approach not only valorizes agricultural waste but also provides a sustainable method for reducing water pollution without the need for chemical or thermal modification [23, 24]. The present study focuses on evaluating the adsorption capacity of raw artichoke waste for methylene blue removal. This work contributes to sustainable water treatment strategies by demonstrating that untreated agricultural residues can serve as effective, low-cost adsorbents for dye removal, offering both environmental and economic benefits.

* Corresponding authors: s.elhajjaji@um5r.ac.ma

2. Materiels and methods

2.1 Preparation of Raw artichoke waste for Adsorption Experiments

In this study, artichoke waste was collected from Marrakech, Morocco. The raw biomass was first carefully washed with distilled water to remove soil particles, dust, and other visible contaminants. To further eliminate organic residues and potential surface impurities, the samples were subsequently rinsed with ethanol. After washing, the artichoke waste was allowed to dry in an oven at 60°C for 24 hours to remove moisture while preserving its structural integrity. Once completely dried, the material was stored in a clean, airtight container until it was used for the adsorption experiments. This thorough preparation ensured the removal of impurities and provided a consistent, reproducible raw adsorbent for subsequent studies (Figure 1).

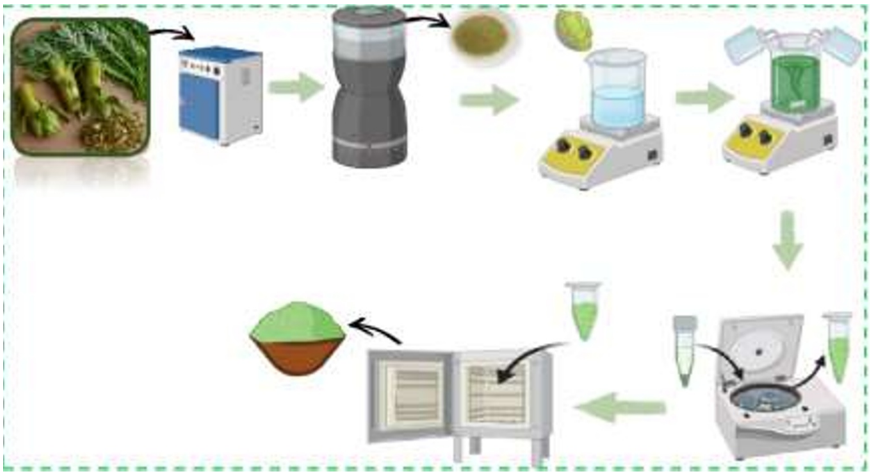

Fig. 1. Preparation process of raw artichoke waste biomass.

2.2 Adsorbate (MB) preparation

Methylene blue (MB), a cationic dye with the chemical formula $C_{16}H_{18}N_3SCl{\cdot}3H_2O$ (structure shown in Figure 2), was selected as the adsorbate for this study. A stock solution was prepared by dissolving 1 g of MB in 1 L of distilled water. This stock solution was then diluted to obtain working concentrations ranging from 20 to 60 mg L^{-1} for use in the adsorption experiments.

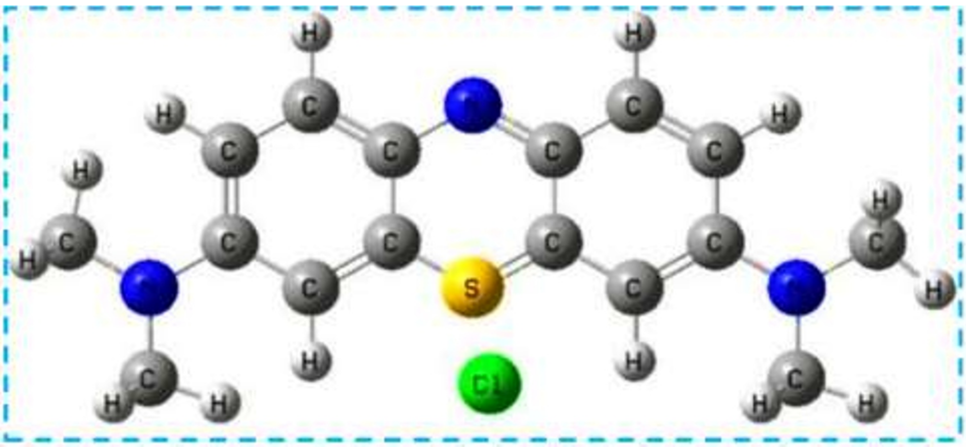

Fig. 2. Molecular structure of methylene blue used in adsorption experiments.

2.3 Characterizations of artichoke

The FT-IR spectrum of the prepared artichoke waste was measured in the range of 400–4000 cm^{-1} using a Bruker Tensor 27 spectrometer in reflection mode with a KBr disc. The crystalline structure and phase composition of the material were determined by X-ray diffraction (XRD).

2.3.1 Adsorption Experiments

In the adsorption tests, 0.6 g of the synthesized artichoke-derived material was added to 50 mL of methylene blue solution (60 mg/L) at room temperature and pH 8.5. The suspensions were stirred for 3 h to ensure equilibrium, after which they were filtered using a membrane with pores of 16–40 µm. Residual MB concentrations were determined at 664 nm by UV–Vis spectrophotometry, and the adsorption capacity was calculated according to Equation (1) [25].

$$q = \frac{(C_0 - C_e) \times V}{m} \tag{1}$$

In this expression, C_0 is the initial concentration of methylene blue ($mg \cdot L^{-1}$), C_e is its equilibrium concentration ($mg \cdot L^{-1}$), V indicates the volume of the solution (L), while m refers to the amount of artichoke used as adsorbent (g).

2.3.2 Adsorption isotherms

The Langmuir and Freundlich models are critical for elucidating the adsorption mechanisms on surfaces [26]. The Langmuir model, with its equation [27]:

$$\frac{1}{Q_e} = \frac{1}{Q_m K_L} \times \frac{1}{C_e} + \frac{1}{Q_m} \tag{2}$$

elates to monolayer adsorption, where Q_m and K_L are the maximum adsorption capacity and adsorption constant, respectively. The Freundlich model, expressed as [28] :

$$lnQ_e = \ln K_f + \frac{1}{n} \ln C_e \tag{3}$$

describes multilayer adsorption on heterogeneous surfaces, with K_f and 1/n1 indicating adsorption capacity and intensity.

2.3.3 Adsorption Kinetics

Kinetic analyses using the pseudo-first-order and pseudo-second-order models help understand the time-dependent adsorption process [29]. The pseudo-first-order model is depicted as [30] :

$$\mathrm{Ln}(Q_e\text{-}Q_t) = \mathrm{ln}Q_t\text{-}K_1 \times t \tag{4}$$

focusing on the adsorption rate K_1. The pseudo-second-order model [31]:

$$\frac{t}{Q_t} = \frac{1}{k_2 Q_e^2} + \frac{1}{Q_e} t \tag{5}$$

better accounts for the entire adsorption mechanism, with k2 representing the rate constant for the second-order reaction.

3. Results and discussion

3.1 Point of Zero Charge (pHpzc) Measurement

Figure 3 illustrates the experimental determination of the point of zero charge (pH_{pzc}) for the different adsorbent materials, including the raw agricultural waste used as the precursor. The pH_{pzc} values were obtained by adjusting the initial pH of the solutions over a range from 2 to 12. The raw precursor exhibited a pH_{pzc} of 7.79, indicating that its surface is neutral at this pH and becomes positively charged below it and negatively charged above it.

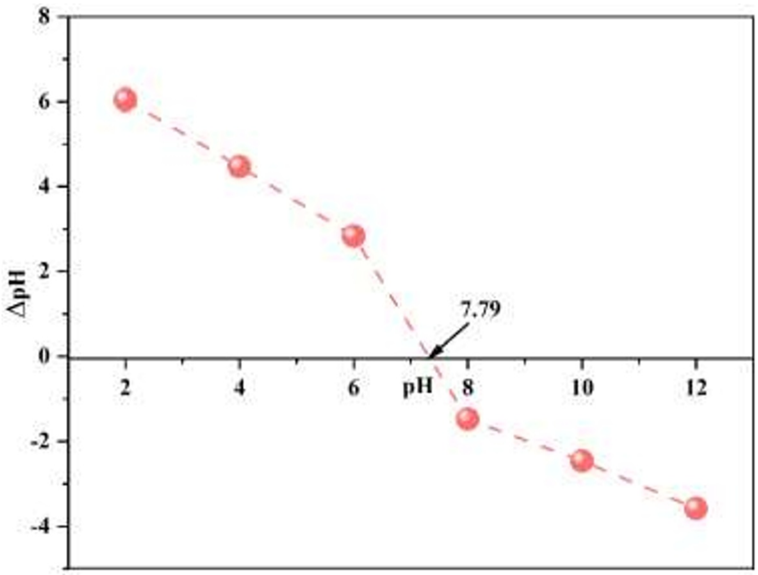

Figure 3. Point of zero charge (pH_{pzc}) of raw artichoke waste.

3.2 XRD measurements

The X-ray diffractogram (Fig. 4) of the plant-based raw material shows a predominantly amorphous structure, as indicated by the broad, diffuse peak centered around 2θ ≈ 22.5°. This characteristic reflection is commonly associated with cellulose crystallites, particularly corresponding to the (002) lattice plane of native cellulose. The broad nature of this peak suggests a low degree of crystallinity, which is typical for lignocellulosic biomass due to the presence of amorphous components such as hemicellulose and lignin [32].

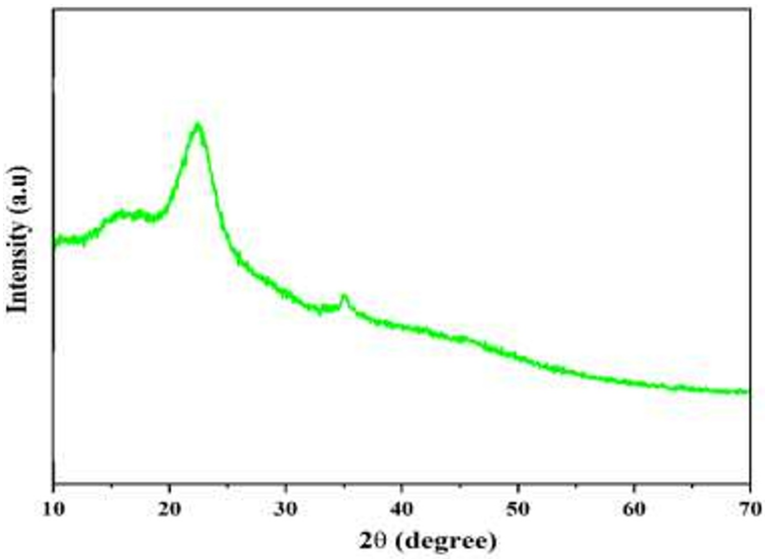

Fig. 4. XRD Diffractograms of artichoke waste.

3.3 Infrared spectrum (FT-IR)

The FT-IR spectrum of the studied adsorbent (Figure 5) displays multiple absorption bands, reflecting its structural complexity and rich organic content. A broad band observed at 3338 cm⁻¹ is attributed to O–H stretching vibrations, typically associated with hydroxyl groups in natural polymers such as lignin, cellulose, and hemicellulose. Peaks at 2926 cm⁻¹ and 2856 cm⁻¹ correspond to asymmetric and symmetric stretching vibrations of $–CH_3$ and $–CH_2–$ groups, indicating the presence of alkyl chains within lignin structures. The band around 1725 cm^{-1} is linked to C=O stretching from carbonyl groups, commonly found in carboxylic acids or esters, while the absorption near 1600 cm^{-1} is assigned to ketonic carbonyl functionalities. The signal at 1430 cm^{-1} represents aliphatic –CH deformations, and the band at 1359 cm^{-1} corresponds to CH_2 bending vibrations in cellulose and hemicellulose frameworks [33]. Moreover, the peak at 1200 cm^{-1} is attributed to C–O stretching, likely from xyloglucan structures. The most intense band in this region, at approximately 1030 cm^{-1}, is associated with C–O–C stretching of primary alcohols in cellulose and hemicellulose. Finally, the absorption observed at 454 cm^{-1} is assigned to Si–O bending vibrations, indicating the presence of siliceous compounds in the material [34].

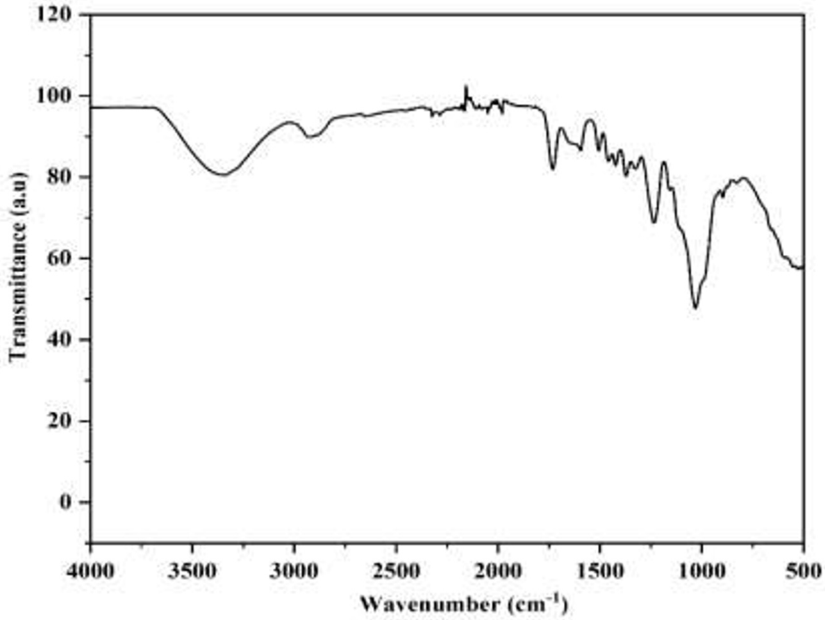

Fig. 5. Infrared Spectra for artichoke waste.

3.4 Effects of Adsorption Parameters

The present work investigated the effect of different operating parameters, considering factors such as the initial dye concentration, the amount of adsorbent, the solution pH, and the contact time on the adsorption process. performance of Artichoke toward methylene blue.

3.4.1 Influence of artichoke *on the adsorption efficiency of MB*

The effect investigating how varying the adsorbent dosage influences the removal performance of MB using artichoke powder was systematically investigated (Figure 6).

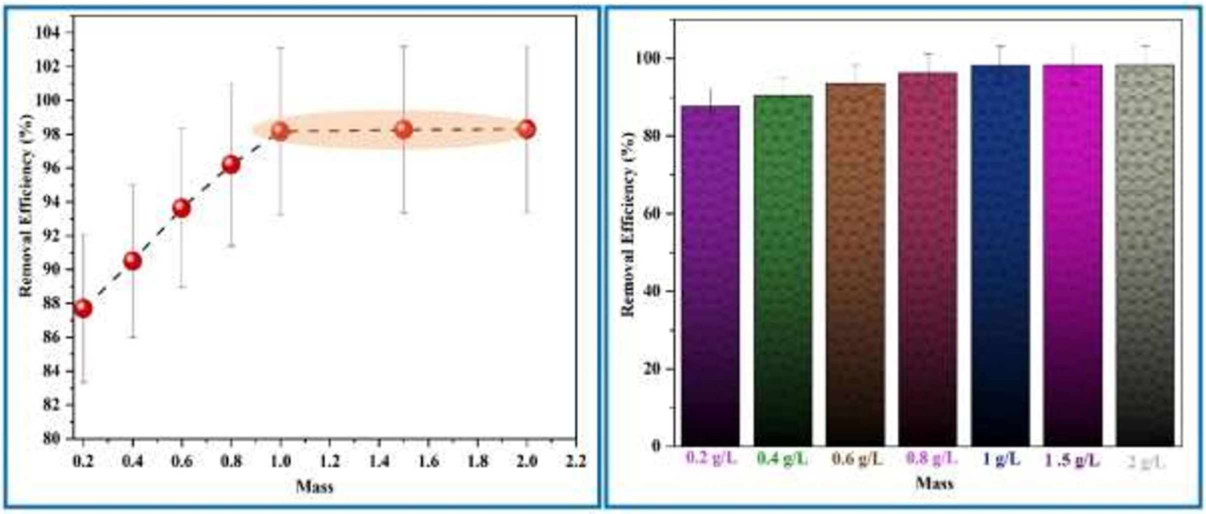

Fig. 6. Variation of MB removal efficiency with increasing artichoke powder dosage (pH of the solution; initial MB concentration = 60 ppm).

Figure 5 shows that increasing the adsorbent dosage leads to a marked improvement in the performance of the adsorption process in removing the dye of methylene blue (MB) by artichoke powder. At a low amount of 0.2 g/L, the capacity to eliminate the dye from solution already reaches 87.7%, highlighting the strong affinity of this biosorbent for dye molecules even at small amounts. As the dosage increases, the efficiency rises significantly, reaching 90.5% at 0.4 g/L, 93.6% at 0.6 g/L, and 96.2% at 0.8 g/L, which is associated with the greater accessibility of active adsorption sites capable of capturing a larger fraction of MB from the solution. Beyond 1 g/L, the removal efficiency stabilizes at around 98.3%, indicating that most MB molecules have already been adsorbed and that further increases in adsorbent mass do not provide substantial improvement. Therefore, an optimal dosage can be considered between 0.6 and 1 g/L, where nearly complete dye removal is achieved while avoiding unnecessary excess of adsorbent material.

3.4.2 Impact of pH on the Adsorption of Methylene Blue from Aqueous Solutions

Figure 7 illustrates the Role of solution pH in controlling the removal efficiency of methylene blue (MB) using artichoke powder as an adsorbent.

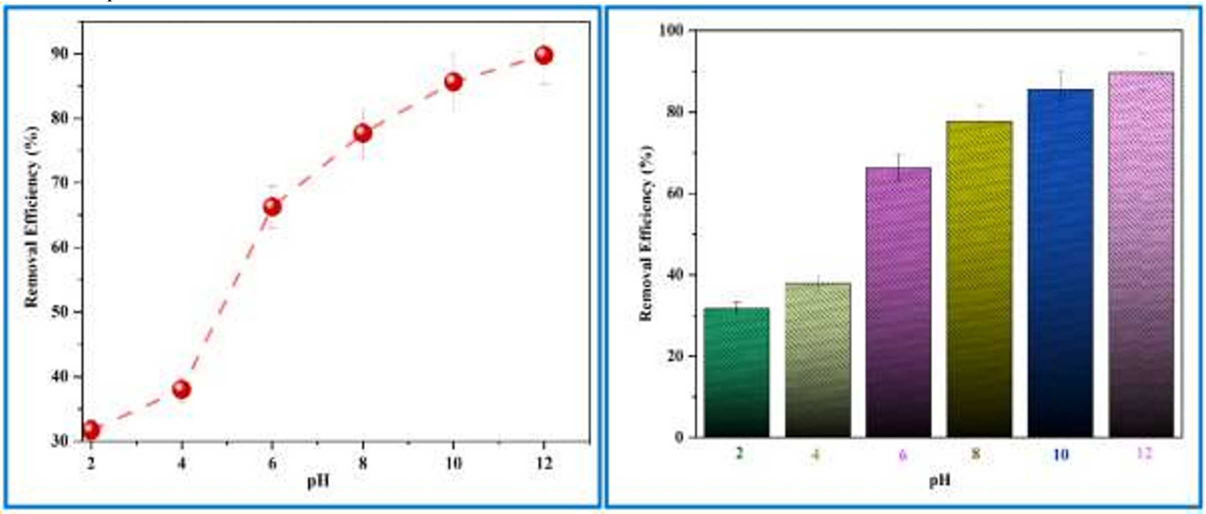

Fig. 7. Effect of pH on MB adsorption by artichoke powder (adsorbent mass = 0.6 g; initial MB concentration = 60 ppm).

At strongly acidic conditions (pH 2), the removal efficiency is relatively low, approximately 31.7%. This can be attributed to the high concentration of H^+ ions in the solution, which compete with the cationic MB molecules for the limited adsorption sites on the surface of the artichoke powder, leading to reduced dye uptake. As the pH increases to 4, the removal efficiency slightly improves to 38.0%, indicating that the electrostatic repulsion between the positively charged adsorbent surface and MB molecules begins to decrease. A more pronounced increase is observed at pH 6, where the removal efficiency reaches 66.3%. This suggests that a greater number of adsorption sites become accessible as the surface functional groups start to deprotonate, reducing competition with H^+ ions and enhancing the adsorption capacity. Under alkaline conditions, the removal efficiency rises sharply, reaching 77.7% at pH 8 and 88.6% at pH 10, before stabilizing near 89.8% at pH 12. This behavior can be explained by the increased deprotonation of functional groups (such as hydroxyl and carboxyl groups) on the artichoke powder surface at higher pH, which generates more negatively charged sites. These negatively charged sites strongly attract the cationic MB molecules through electrostatic interactions, thereby maximizing dye removal.

Overall, the observed trend indicates that alkaline conditions, particularly in the pH range of 10–12, are optimal for MB removal by artichoke powder, highlighting the significant role of solution pH in controlling adsorption efficiency through modulation of surface charge and electrostatic interactions [35].

3.4.3 Isotherm data analysis

Figure 8 presents the adsorption isotherms of methylene blue (MB) on artichoke powder, fitted using the Langmuir, Freundlich, and Sips models, with the quality of each fit evaluated through the sum of squared errors (SSE).

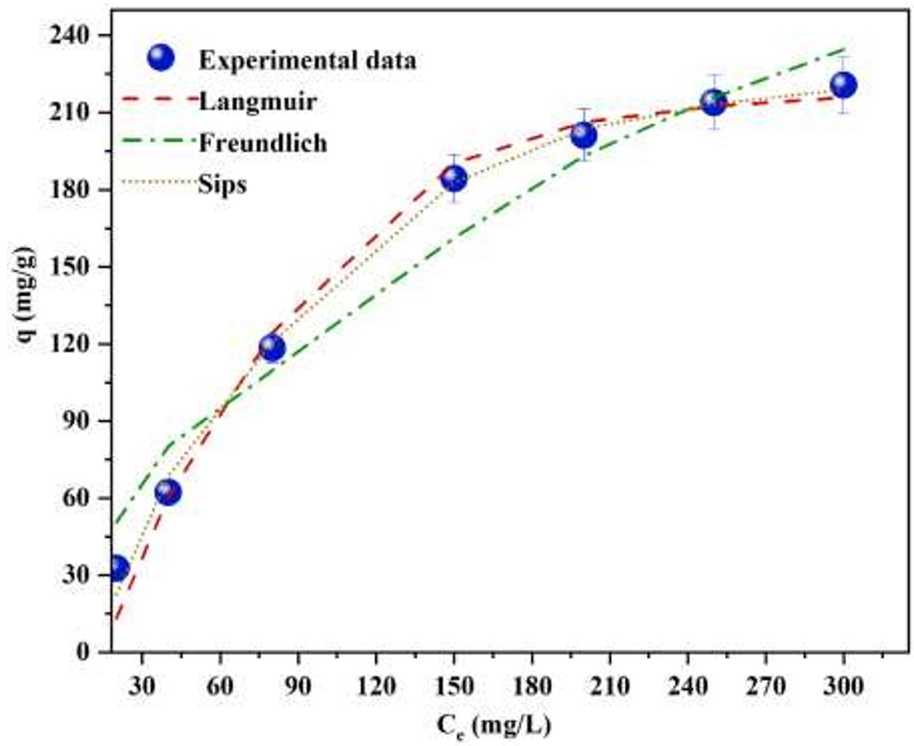

Fig. 8. The equilibrium adsorption data were interpreted using Langmuir, Freundlich, and Sips models for methylene blue adsorption on artichoke powder (adsorbent mass = 0.6 g; pH of the solution= 10).

The Freundlich model exhibits the highest SSE (1504.4), indicating a poor agreement with experimental data and suggesting that multilayer adsorption on heterogeneous sites is not the predominant mechanism in this system. The Langmuir model (Table 1), with SSE = 507.7, better captures the adsorption trend, supporting the assumption of monolayer adsorption on a largely homogeneous surface. However, the Sips model shows the lowest SSE (156.9), indicating the closest match to the experimental data by combining the advantages of both Langmuir and Freundlich models. This low SSE implies that while MB adsorption is predominantly monolayer, slight surface heterogeneity exists, allowing some adsorption sites to exhibit stronger or weaker affinities for MB molecules. Mechanistically, this adsorption is dominated by chemisorption, primarily through electrostatic interactions between the negatively charged functional groups of artichoke powder (hydroxyl, carboxyl, and carbonyl groups) and the cationic MB molecules. Additional contributions from hydrogen bonding and π–π interactions between the aromatic structures of MB and the lignocellulosic components of the adsorbent may also play a role. The comparison of SSE values therefore highlights that the Sips model provides the most accurate description of the adsorption behavior, confirming that MB uptake is efficient, largely monolayer, and influenced by minor variations in site energy across the adsorbent surface [36].

Table 2 shows that the artichoke waste investigated in this study exhibits the highest adsorption capacity for methylene blue (MB), reaching 140.47 mg/g. This value exceeds those reported for other adsorbents, including KOH-activated urban sludge (81.04 mg/g), KOH-activated biomass waste (136.5 mg/g), $ZnCl_2$-activated rice husk (9.73 mg/g), and H_3PO_4-activated Spathodea campanulata (86.21 mg/g). Under comparable conditions, particularly pH around 8 and low adsorbent dosages, these findings indicate that artichoke waste is an especially effective and promising low-cost material for dye removal applications.

Table 1. Isotherm parameters for MB adsorption onto artichoke waste

Models	Parameters	BK
Langmuir	q_m (mg/g)	225.12
	K_L(L/mg)	0.14
	SSE	507.72
Freundlich	n	3.86
	K_F (mg/g) $(mg/L)^{-1/n}$	62.19
	SSE	1504.42
Sips	n_S	0.56
	q_m (mg/g)	244.33
	K_S (L/mg)	0.11
	SEE	156.94

Table 2. Literature Survey of MB Adsorption Capacities Across Various Adsorbents

Adsorbents	pH	Mass (g/L)	Q_{max} (mg/g)	Ref
Activated carbon by KOH from urban sludge	8	0.06	81.04	[15]
KOH activated biomass waste	8	0.8	136.5	[37]
Activated carbon by $ZnCl_2$ from rice husk	8	12	9.73	[38]
Activated carbon of spathodea Campanulate byH_3PO_4	10	2	86.207	[39]
Artichoke waste	8	0.03	244.33	**In this Study**

3.4.4 Kinetic Analysis of Methylene Blue Adsorption on Artichoke Powder

The experimental data reveal a marked difference in the performance of the kinetic models (Figure 9), which becomes particularly evident when considering the sum of squared errors (SSE).

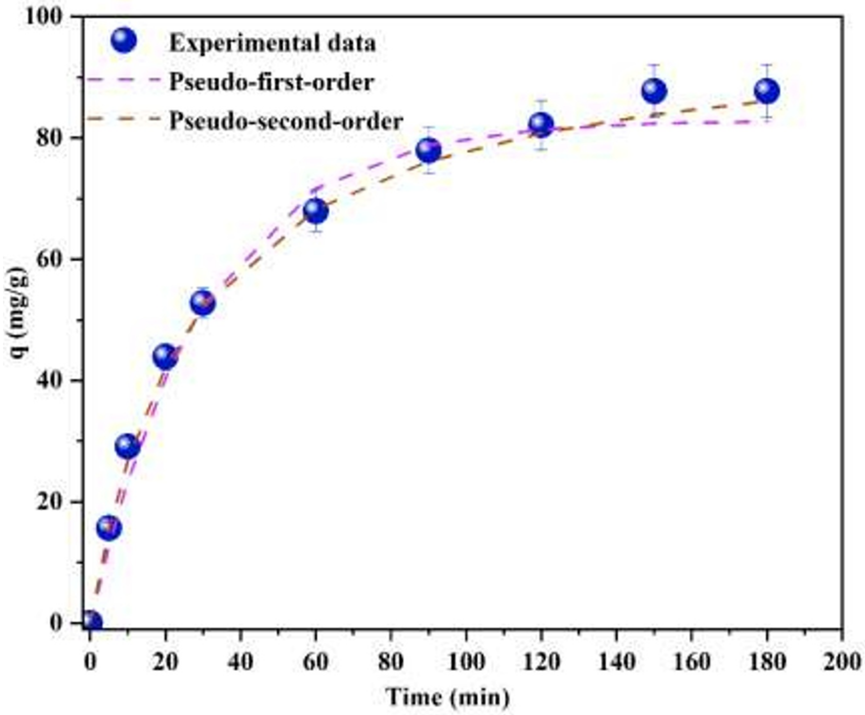

Fig. 9. Experimental data and fitting of methylene blue adsorption on artichoke powder to pseudo-first-order, and pseudo-second-order kinetic models (adsorbent mass = 0.6 g; initial MB concentration = 60 ppm; pH of the solution= 10).

The pseudo-first-order model (Table 3) exhibits a relatively high SSE value of 120.75, indicating a substantial deviation between the experimental adsorption capacities ($q_{e,exp}$ = 82.94 mg/g) and the values predicted by the model ($q_{e,cal}$ = 82.18 mg/g). This large error suggests that the pseudo-first-order model inadequately captures the actual adsorption behavior of methylene blue on artichoke powder and fails to account for the complex interactions between the dye molecules and the functional groups of the adsorbent. In contrast, the pseudo-second-order model displays a much lower SSE of 31.41, reflecting a much closer alignment between the experimental data ($q_{e,exp}$ = 83.56 mg/g) and the model predictions ($q_{e,cal}$ = 98.93 mg/g). The significant reduction in SSE indicates that this model is far superior in describing the kinetics of MB adsorption, accurately capturing both the rapid initial uptake and the gradual approach to equilibrium. The marked difference in SSE values between the two models provides insight into the adsorption mechanism. The superior fit of the pseudo-second-order model suggests that the adsorption process is dominated by chemisorption, involving electron sharing or exchange between the cationic MB molecules and the negatively charged functional groups (such as hydroxyl, carboxyl, and carbonyl groups) on the artichoke powder surface. Physical adsorption alone cannot account for the low SSE observed with the second-order model, implying that chemical interactions at the solid-liquid interface play a major role in controlling the rate and extent of MB removal. Overall, the comparison of SSE values clearly demonstrates that the pseudo-second-order model provides the most accurate representation of the adsorption kinetics, highlighting the importance of chemical interactions and active site availability in achieving efficient dye removal by artichoke powder [40-41].

Table 3. Adsorption kinetic parameters and model fitting for methylene blue on artichoke powder

Models	Parameters	Artichoke
Pseudo-first-order	K_1 (min^{-1})	0.033
	$q_{e,exp}$ (mg/g)	82.938
	$q_{e,th}$ (mg/g)	82.177
	SSE	120.746
Pseudo-second-order	K_2 (min^{-1})	0.00037
	$q_{e,exp}$ (mg/g)	83.561
	$q_{e,th}$ (mg/g)	98.932
	SSE	31.406

4. Conclusion

The study confirms the high potential of raw artichoke waste powder as an efficient biosorbent for methylene blue removal from aqueous solutions. Adsorption was strongly influenced by solution pH and adsorbent dosage, with optimal removal achieved under alkaline conditions (pH 10–12) and dosages between 0.6 and 1 g/L. Isotherm analysis indicated predominantly monolayer adsorption, as the Sips model provided the lowest SSE, while kinetic studies showed that the pseudo-second-order model best describes the adsorption process, pointing to chemisorption as the main mechanism. Functional groups on the artichoke waste powder, including hydroxyl, carboxyl, and carbonyl moieties, facilitate MB binding via electrostatic interactions, hydrogen bonding, and π–π interactions. Overall, raw artichoke waste powder is a sustainable, low-cost adsorbent offering environmental and economic advantages for water purification.

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