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Sustainable Use of Silk Vine Roots(S-VPR) as Low Cost Adsorbent for the Removal of Methylene Blue Dye from Its Aqueous Solution

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A B S T R A C T

This study examines the adsorption of methylene blue dye from an aqueous solution using silk vine roots derived from dairy powder. The adsorbent material was utilized in its natural state, without any chemical or physical treatment. The adsorption process was conducted under different conditions, including pH, adsorbent weight, initial dye concentration, and contact time at room temperature. The adsorption capacity exhibited an increase (49.9 mg/g), whereas the adsorbent weight and initial concentration increased until equilibrium was reached within 20 minutes. The adsorption process followed second-order kinetics and displayed compatibility with the Freundlich isotherm coefficient. It was determined to be a multilayer, heterogeneous adsorption process with a maximum adsorption capacity value (qe_{max} 49.9 mg/g). Based on the results, it can be confirmed that wild dairy (silk vine) is capable of effectively adsorbing pollutants, such as the blue methylene dye.

1. Introduction

Water plays a vital role in the material composition of the Earth's surface. Physical and chemical properties of water are essential for ensuring water safety and effective treatment methods. As urban areas expand and various activities introduce pollutants into different water systems, the significance of water purity has increased. This situation necessitates that water treatment professionals manage a diverse array of water properties and employ a broader range of treatment techniques.

Dyes are substances containing chromophores, which are responsible for color, allowing them to impart color to materials. They are widely used in textiles, cosmetics, plastics, and other applications (Papagiannaki et al., 2022).

Pollution resulting from sewage discharged by various industries, including leather production, printing presses, and textiles, presents a significant environmental challenge (Adachi et al., 2023). Pigments are classified based on their composition or chemical structure. Annually, approximately 700,000 tons of tailings are generated, along with over ten thousand different dyes and synthetic dyes worldwide. Each year, 200,000 tons of pigments utilized in the textile industry are released as liquid waste. These dyes enter natural water sources and persist in various environments, leading to numerous harmful effects. The colored complexes of these dyes contribute to their reduced biodegradability (Chequer et al., 2013).

In recent years, the development of economical and environmentally friendly adsorbents for wastewater purification has garnered significant interest. Conventional methods for removing metals from industrial effluents include chemical precipitation, coagulation, solvent extraction, electrolysis, membrane separation, ion exchange, and adsorption. The cost of water purification using these technologies, excluding adsorption, ranges from 10 to 450$ per cubic meter of treated water. In contrast, the cost of water treatment utilizing activated carbon adsorption ranges from 5 to 200$ per cubic meter of water (Ali, 2010). Various

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Inexpensive agricultural waste materials are currently used to remove dyes from waste water. Sorbents made from agricultural peels have demonstrated their environmental friendliness, cost-effectiveness, sustainability, availability, and efficiency, making them suitable for developing sorbents to combat water pollution (Amalina et al., 2020). This research aims to investigate the use of wild plants, specifically silk vine roots, as a cost-effective and readily available material for the removal of blue methylene dye from aqueous solution.

2. MATERIAL AND METHODS

2.1. Material

Silk-Vine Roots Plant (*SVR*) was collected from Hrawa village, located near Sirte in northern Libya, to be used as an adsorbent after. The silk vine roots plant were first collected, then washed with distilled water, and finally dried in ambient air. After that, they were further dried in a drying oven at a temperature of 110°C. The dried plant was subsequently ground into a fine powder using a mill and then sieved to achieve a particle size of 500 µm. . Methylene Blue dye was obtained from Riedel-De Haen AG, while sodium hydroxide (NaOH) was purchased from T-Baker Lab Chemicals, India. Hydrochloric acid (HCl) was obtained from ChiMiE-Plus Laboratories, France.

2.2. Instrument.

Laboratory equipment used included a Memmert/W. Germany D06062 MODEL L600 oven, an electronic balance (AAA 250 L) from ADAM, a genuine Electric Grinder Moulinex from France, an ASTM (prutsied/Siev a.500 mm) from Retsch, Germany, a pH meter (Thermo-pH meter, Orion 2 Star) from the USA, and a Thermo-UV/Vis Spectrophotometer (Nicolet eval.).

2.3. Standard solution of the methylene dye.

A standard dye solution (1000 ppm) was prepared by dissolving 1 g of dye in deionized water in a volumetric flask (1 liter). Necessary solutions (1, 3, 5, 7, and 10 ppm) were prepared through appropriate dilution. The absorbance of these diluted solutions was measured using a UV-Vis measurements were carried out at wavelengths ranging from 615–662 nm to create a calibration curve for the dye, which would be used to determine the remaining concentrations in adsorption tests of dye on Silk-Vine Plant Roots (S-VPR) (Al-Azza and Shehadeh, 1980).

2.4. Characterization of natural of silk vine roots plant (S-VRP)

2.4.1. Zero equivalent point (PHz).

A 0.1 N solution of sodium chloride (NaCl) was prepared. Next, 50 mL of the solution was divided into five separate flasks. To each flask was added 0.5 g of the plant powder. The pH value of each flask was adjusted at 3, 5, 7, 10, and 12. The flasks were then stirred at a speed of 150 rpm and left at room temperature for 48 hours. Afterward, the contents of each flask were filtered using filter paper. The change in pH (pHf) for each flask was measured, and the relationship between pHz and pHi was determined by subtracting pH_f from pHi according to equation (1) below:

$$pHz = pH_i - pH_f \quad \ldots\ldots (1)$$

Where PHz= pH at zero, pHi =pH initial, pHf = pH final

2.4.2. Moisture Content

The moisture content was measured according to ASTM 2867-99. A 2.0 g samples of Silk-Vine Plant Roots (SVRP) were placed in a weighed ceramic crucible and dried in a furnace until a constant weight was achieved. After cooling, the samples were weighed again. The moisture content was calculated using Equation (2) as follows:

$$\text{Moisture } \% = \frac{W_{m3} - W_{m2}}{W_{m1}} \times 100 \quad \ldots\ldots (2)$$

Where: W_{m3} is the weight of crucible containing 2.0 g of adsorbent plant. W_{m2} is the weight of the crucible containing dried adsorbent plant and W_{m1} is the weight of the original adsorbent plant used.

The pH of natural S-VPR solutions was determined using a modified method based on ASTM D3838-80 (Ekpete et al., 2011). A 1.0 g sample of natural S-VPR was added to 100 ml of deionized water and stirred for 1 hour. The pH of the filtered solution was subsequently measured using an electronic pH/conductivity meter.

2.4.3. Ash content

Ash content was determined following the ASTM D2866-94 method (Ekpete et al., 2011). A 1.0 g sample of the dried S-VPR was placed into a porcelain crucible and transferred to a muffle furnace at 1000 ℃ for 1 hour. The ash content was then calculated using the following equation (3):

$$\text{Ash } \% = \frac{W_{\text{ash}}}{W_{\circ}} \times 100 \quad \ldots\ldots (3)$$

Where: $W_{\circ}$ is the weight of the dried sample before burned and W_{ash} is the lost weight.

2.4.4. Solubility in aqueous solution

The solubility of natural S-VPR samples in aqueous solutions was determined using a modified version of ASTM E1148-02(2008) (Hashem & Elhmmali, 2006). A total of 0.50 g of S-VPR was placed into an Erlenmeyer flask with a ground plastic stopper, containing 100 ml of distilled water. The mixture was shaken for 1 hour at 60°C. After shaking, the mixture was filtered, and 5 ml of the filtrate was placed onto a glass plate of known weight and dried at 105°C in an oven until a constant weight was achieved. The following equation (Equation 5) was used:

$$\text{Solubility } \% = \frac{\text{Weight difference}}{0.5 \times 5} \times 100 \quad (4)$$

2.4.5. Cellulose content

The cellulose content was determined using the ASTM-D 1105-96. A specific weight of the sample (W1) was dissolved in 2N HCl and boiled for 2 hours. The resulting mixture was filtered, and the residual sample was washed several times with distilled water, dried, and weighed (w2) (Hill, 1979).

$$Cellulose\ \% = \frac{W_1 - W_2}{W_1} \times 100 \quad \dots \quad (5)$$

2.5. Adsorption process:

The Silk Vine Roots Plant (SVRP) was used to remove methylene blue dye from an aqueous solution as a sustainable, low-cost option. To determine the effects of various factors on the adsorption process, such as dye weight, pH, initial dye concentration, and contact time, each experiment was conducted under uniform chemical conditions. 0.1 g of Silk Vine Plant Roots (S-VR) were added to 50 ml of the dye solution, which had a known initial concentration (C_0) and initial pH at room temperature (25°C). The mixture was agitated at a constant speed of (150 rpm) for a 120 min to reach equilibrium. After equilibrium was achieved, the samples were separated by fast filtration. Absorption was measured using an Ultraviolet-Visible (UV-Visible) Spectrophotometer at a wavelength of 665 nm to determine the remaining concentration (Ct). The adsorption capacity and percentage of residual concentration to be eliminated were calculated using the following relationships:

$$percentage\ removal = \frac{(C_0 - C\ t\ )}{C_0} \times 100.. \ (6)$$

Where: C_0 = Initial dye concentration (mg/l), C_t = dye concentration (mg/l) at time (min).

$$\text{Adsorption capacity } q_e = \frac{(C_0 - C_e)}{M} \times V \quad . (7)$$

Where: V = Solution volume, M = adsorbent weight (g), C_0 = Initial dye concentration (mg/l)

3. RESULTS AND DISCUSSIONS

3.1. Determination of the Standard Curve for Blue Methylene Dye

Figure 1 presents the standard curve for methylene blue dye, demonstrating the relationship between absorbance and concentration as described by Lambert's law. According to this law, an increase in absorbance corresponds to an increase in concentration. By using the equation derived from this relationship, the concentration can be calculated at various time intervals.

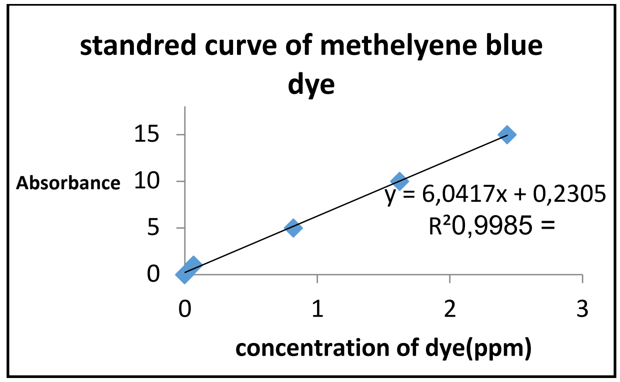


Figure 3. 1: standard curve of methylene blue dye.

3.2. Physical properties of the adsorbent (S-VRP).

The following table (Table 1) indicates that the pH of the silk vine roots plant (S-VR) was found to be acidic (4.01). The solubility of S-VR was 5.12, and the particle size was sieved to range from 0.63 to 0.125 mm. The cellulose content was measured at 5.3%, the ash content was 0.979%, and the moisture content was 7.015. These values are considered suitable for the adsorption process of methylene blue dye, as supported by previous studies (Erhayem et al., 2020). The cellulose content was determined to be appropriate and highly significant for the chemical composition of plant fibers, contributing to their strength and stability. This suggests that the physical properties of the silk vine roots plant (S-VRP) are favorable, as noted by Faraja et al., (2019). Additionally, the physical properties of S-VRP were found to be satisfactory, as reported in the literature. The high moisture content observed can be attributed to the plant's persistence and poor mechanical properties (Hameed et al., 2007; Zhang et al., 2011). The low ash content indicates a lack of metallic contaminants. The particle size ranged from 0.125 to 0.063 mm; smaller sizes provide a larger surface area, enhancing the adsorption process.

Table1: physical properties of the adsorbent material (SVRP).

Solubility (g/L)	Particles size (mm)	Cellulose content (%)	Ash content (%)	pH	Moisture
5.12	0.63-0.125	5.3	0.979	4.01	7.015

The results presented in Table 1 indicate that the pH of the *silk vine roots plant (S-VRP)* as raw was found to be acidic (4.01), the Solubility of *(S-VRP)* was 5.12, and Particles size was by sieved 0.63-0.125, Cellulose content 5.3%, Ash content 0.979, Moisture 7.015. These values are considered suitable for the adsorption process of methylene blue dye, as supported by previous studies (Erhayem et al., 2020).The cellulose content was determined to be appropriate and highly significant for the chemical composition of plant fibers, as it contributes to their strength and stability. This suggests that the physical properties of the *silk vine roots* (SVR) are favorable (Faraja, et al., 2019), and satisfactory to, the physical properties can be attributed to the plant's persistence and poor mechanical properties (Hameed et al., 2007), (Zhang et al., 2011). The low ash content value indicates a lack of metallic contaminants. The particles size ranged from 0.125 to 0.063mm. This small size leads to a larger surface area, which enhances the adsorption process.

3.3 Zero equivalent point (PHz) of Silk Vine Roots.

According to Figure 2, the zero point shipment of silk-vine roots (PHz) is utilized to elucidate the adsorption mechanism as well as the characteristics of active and inactive surface sites. In this context, it is observed that the initial pH (PHi) is equivalent to PHz, encompassing both negative and positive surface concentrations. When PHi is less than PHz, the surface loading is positive; conversely, when PHi exceeds PHz, the surface loading becomes negative.

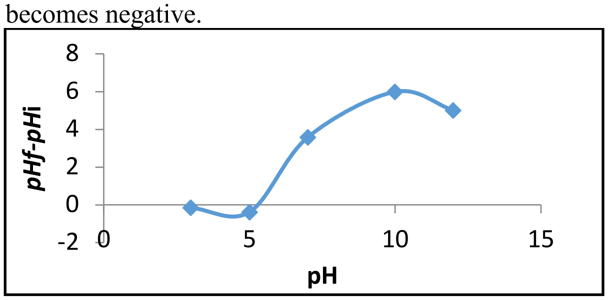


Figure 3.2: The zero point shipment of the adsorbent (SVRP).

3.4 The effect of pH on adsorption removal Methylene Blue Dye.

Figure 3 illustrates the importance of pH in controlling adsorption efficiency, as it affects the degree of ionization of molecules and their physical properties (Baek et al., 2010; Sharma et al., 2009). The adjusted pH values ranged from 3 to 12 at room temperature. As shown in the figure, the adsorption capacity and percent removal were lower at a pH of 3 due to competition with H^+ ions. Conversely, at higher pH levels, the adsorption capacity of MB and MG dyes increased because the negative surface charge intensified and H^+ decreased. Consequently, which increase the electrostatic attractions between the cationic dye molecules and the surface increased (Chieng et al., 2014; Selen et al., 2016).

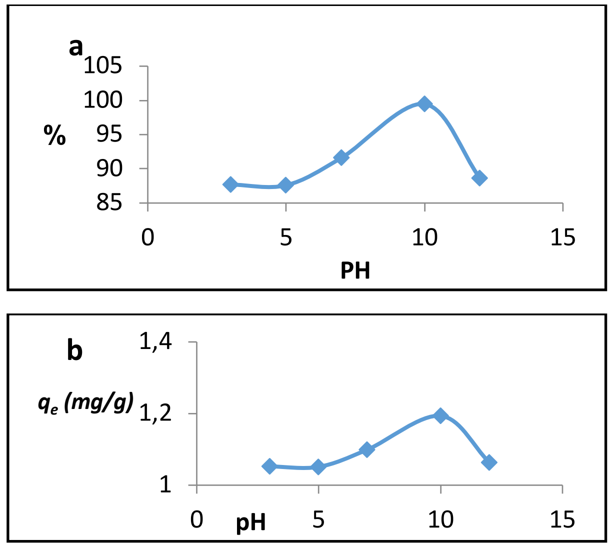
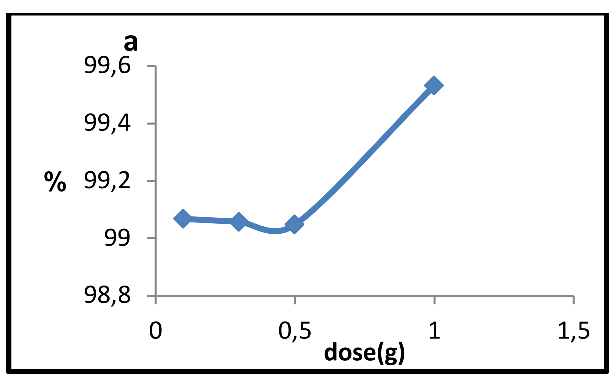


Figure 3: (a) The effect of pH on percent removal and (b) the adsorption capacity of blue methylene dye by the roots of silk vine.

3.5 The effect of the adsorbent dose on the adsorption process of MB Dye.

Figure 4 illustrates the effect of varying doses of silk vine root plant (SVR) on percentage removal and adsorption capability, with doses changing from 0.1 to 1 g while keeping other factors constant. The adsorption capacity decreases as the weight of the adsorbent increases. This reduction in the amount of adsorbed dye (qe) is likely due to the overlapping agglomeration of particles, which results in a decreased surface area. However, the percent removal increases (Da Silva et al., 2011).



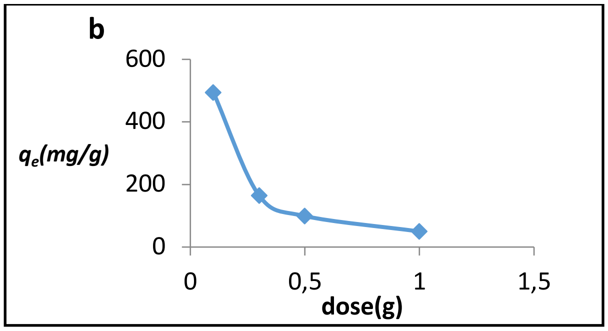


Figure 4: The effect of the adsorbent dose. (a) Removal percentage (%) and (b) adsorption capacity (q_e).

3.6 Effect of initial concentration on the adsorption process of MB Dye.

The effect of the initial concentration of the dye on the removal percentage and adsorption capacity of the adsorbent was studied. Dye concentrations ranging from 30 to 300 mg/L at pH 10 were used. Figure 6 illustrates the inverse correlation between the removal rate and dye concentration. Conversely, increasing the primary dye concentration also enhanced the capacity of the adsorbent (Figure 8). This may be attributed to the strength required to resist the movement of the dye molecules between the adsorbent and the dye solution (Bukallah et al., 2007; Han et al., 2007).

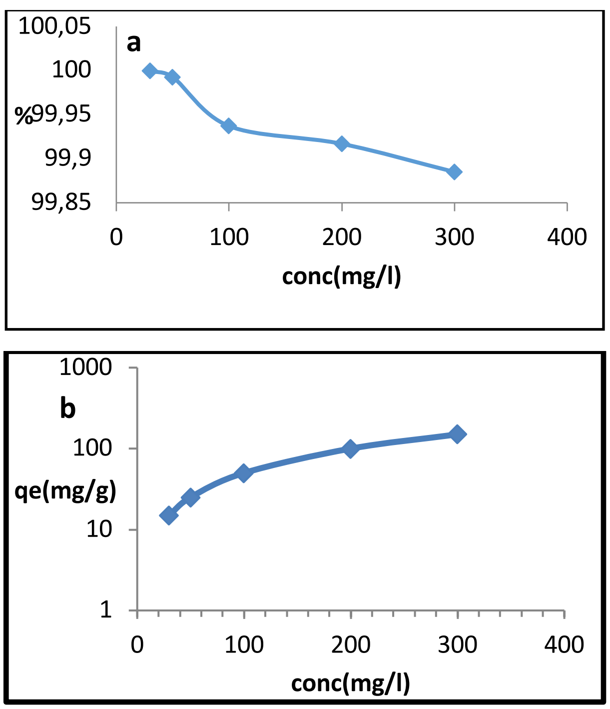


Figure 5: The effect of the initial concentration of adsorbent material (SVRP). (a) present (%) and (b) adsorption capacity (q_e).

3.7. Effect of contact time on the adsorption process of MB Dye.

Figure 6 presents the effect of contact time on the adsorption of MB using the S-VR adsorbent. It also shows the percentage removal and adsorption capacity for methyl blue dye, which varies with contact time from 0 to 120 minutes. Equilibrium was achieved at 20 minutes, as this is when the surface of the adsorbent material becomes gradually saturated with dye molecules (Foo & Hameed, 2010; Verma et al., 2012).

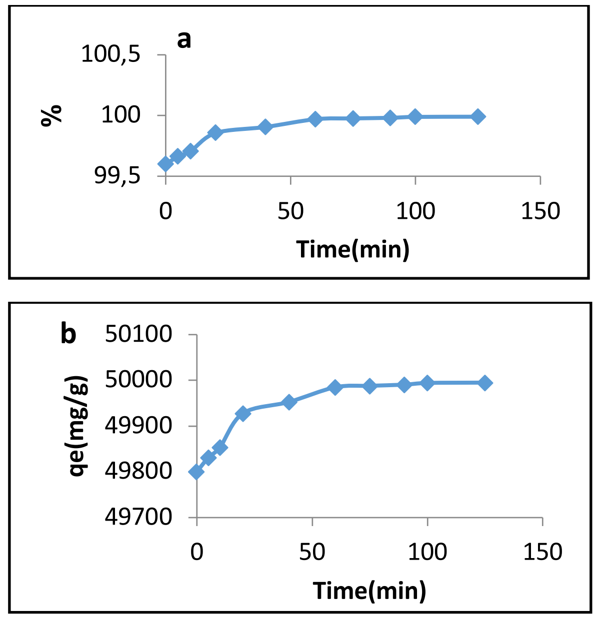


Figure 6: The effect of the contact time on the adsorption. (a) Present (%) and (b) the adsorption capacity (q_e).

3.8. The studied process was kinetic adsorption of MB dye by SVRP.

The study of adsorption kinetics is important as it provides a clear understanding of the absorption rate. It offers valuable information for visualizing the design and modelling of the adsorption process. This study involves first-order and second-order interactions, which are used to evaluate the adsorption mechanism in the plant (Suteu & Malutan, 2012). Table 2 illustrates the kinetic properties of the studied dye material.

Table 2: The reaction kinetic factors of the adsorption process.

R_2	$_1K$	$q_{e\ cal}$	$q_{e\ exp}$	Frist order
0.962	0.036	26.47	49.96	
R^2	K_2	q_{ecal}	$q_{e\ exp}$	Second order
1	0.02	62.33	49.96	

Pseudo first order kinetic model of Lagergren equation was derived by scientist Lagergren for a liquid/steel system. It depends on the adsorption capacity of the solid state:

$$Log(q_e-q_t)=logq_e-(k_1/2.303)t \quad .... (8)$$

In this equation, qe and qt represents the adsorption capacity at equilibrium and at time t, respectively. The tendency and intersection resulting from the plot of log (q_e-q_t) against time t at different concentrations are used to determine k_1 and the calculated qe (Holkar et al., 2016). Figures 7 and 8 illustrate the first- and second-order reactions for the adsorption.

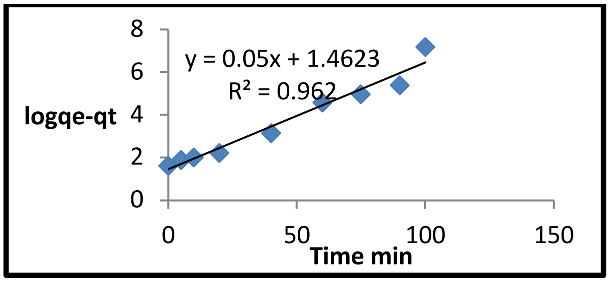


Figure 7: The first-order reaction for the adsorption process.

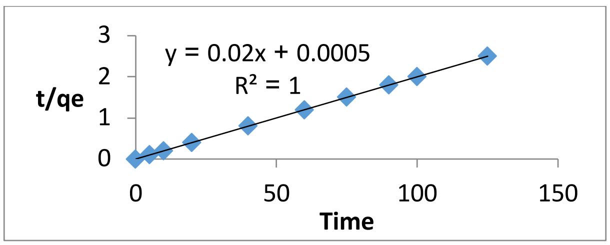


Figure 8: The second-order reaction of the adsorption process.

HO and MCkay are based on solid-state adsorption capacity. The equation is as follows:

t/qt = 1/k2q 2e + 1/qe t ………………. (9)

The linear relationship between t/qt and t is used to determine qe and k2 for the slope and intercept (Hamdaoui, & Chiha, 2007), as indicated by the R_2 values obtained in Table 3. The results showed that the R^2 value for the second degree is greater than R^1 for the first degree. Therefore, pseudo-second-order kinetics is more suitable for the absorption of methylene dye by silk vine roots (Wang et al., 2011).

3.9 Isotherm for the adsorption process.

The Langmuir isotherm, particularly relevant to the adsorption of gases on solid materials, indicate that a monolayer or a single layer of molecules is formed on a homogenous surface. The energy is uniform, with dissonance between the molecules. There is partial adhesion of the dye at specific sites on the adsorption material, and the adsorption energy remains constant (Abechi et al., 2010). The Langmuir Equation is illustrated as follows**:-**

C_e/q_e=1/q_m.b+C_e/q_m …………………… (10)

As b is constant Langmuir and q_m maximum adsorption capacity and C_e concentration of equilibrium. Observing the isotherm, Langmuir in Table 3 reveals that it is highly compatible with experimental data, indicating homogeneous adsorption. The R_L value we find less than one and greater than zero suggests that adsorption is favorable acceptable and confirms strong uniform adsorption on the surface. Figure 9 illustrates the Langmuir isotherm of the process**.**

Table 3: The isotherm constants for adsorption process.

R^2	R_L	B	q_m	T	Langmuir isotherm
0.859	0.961	0.0004	49.99	25°C	
n	R^2	1/n	Kf	T	Freundlich isotherm
3.26	.93770	0.306	5.087	25 °C	

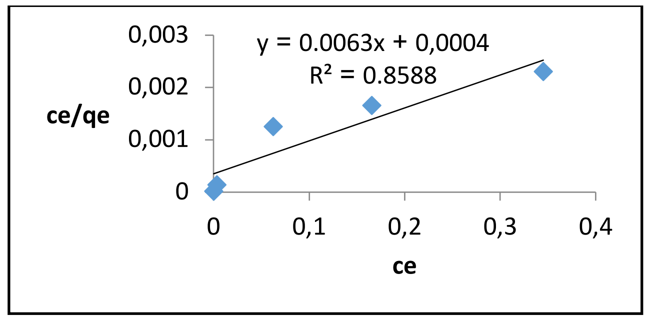


Figure 9: The Langmuir isotherm of methylene dye adsorption on (silk vine roots).

Freundlich isotherm

The Frendlich Isotherm is an experimental model that applies to heterogeneous, layered surfaces with different energetic sites (Raghuvanshi, 2004). The equations bellow demonstrate the Frendlich isotherm:

qe = k_fC1/n………………………… (11)

Log Ce +1/nLog qe = logKf ……….. (12)

The K_f constant equilibrium and the 1/n heterogeneity coefficient indicate a strong adsorption relationship between the surface of the adsorbent material and the dye. The lower value of 1/n (0.306 < 1) suggests a strong adsorption process between the dye and the adsorbent material (Azhar et al., 2005). This is further supported by the value of n (3.26), which is greater than one, indicating that the adsorption conditions are favorable.

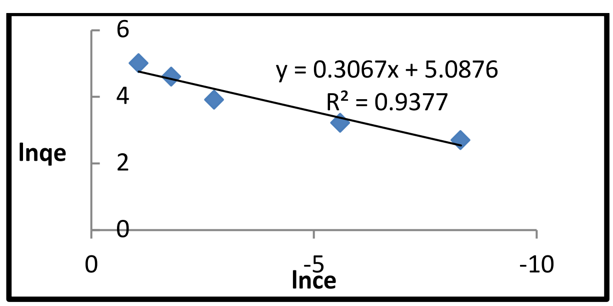


Figure 10: The Frendlich Isotherm of methylene dye adsorption on (silk vine roots).

Conclusion

This study investigated the adsorption of methylene blue dye from an aqueous solution, prepared in the laboratory, using the powder of wild silk vine roots as a low-cost agricultural material without any chemical or physical treatment. The adsorption process was conducted under varying conditions of pH, adsorbent dose, and initial dye concentration over different time periods at room temperature. The removal rate at a pH of 10 was 99.9%, with an adsorption capacity of 49.9 mg/g. The removal efficiency increased with higher doses of the adsorbent and initial dye concentration until equilibrium was reached within 20 minutes.

The physical properties of the adsorbent, such as ash content, moisture content, and neutral pH, were found to be suitable for the adsorption process. The adsorption data fit second-order kinetics and aligned with the Freundlich isotherm, indicating that the process involves multilayer adsorption on a heterogeneous surface. The maximum adsorption capacity (qe_{max}) was determined to be 49.9 mg/g. Based on the results obtained, we can confirm that wild silk vine roots are effective in adsorbing pollutants, such as dyes and heavy metals, from solutions.

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