American Journal of Applied Chemistry
2024, Vol. 12, No. 6, pp. 149-157
https://doi.org/10.11648/j.ajac.20241206.13

Research Article

Simple Use of Low-cost and Available Adsorbent for Cationic Dye Adsorption from Aqueous Solution

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Abstract

The aim of this work was to study the adsorption of methylene blue (MB) on local agricultural waste in the batch process. The adsorbent, washed rice husk (WRH) was subjected to some physical and chemical analyses such as EDX/SEM, FT-IR, XRD and XRF. The adsorbent showed an amorphous structure and dominated by silica in its composition. The influence of adsorption parameters such as contact time, adsorbent dose and initial concentration was studied. The mass of washed rice husk (WRH) varied at 1 to 6 g, the initial concentrations MB (50-250 mg/L) and the contact time (10-90 min). The MB removal percentage reached 86% with a WRH amount of 4.5 g and an optimal time of 30 min. Langmuir and Freundlich isotherm models were used to process the adsorption data. Q_{max}, the maximum adsorption capacity of MB from Langmuir' model was 13.23 $mg.g^{-1}$. The values of the R_L constant varying between 0-1 for the initial concentrations studied proved that the adsorption is favorable. The value of the parameter n being less than 1obeys the condition of heterogeneity. Pseudo-first order and pseudo-second order models were used to study the kinetic adsorption process. The kinetic parameters calculated from each model showed that the adsorption of MB on the WRH could be describe by the pseudo-second order.

Keywords

Adsorption, Rice Husk, Methylene Blue, Agricultural Waste

1. Introduction

Industrial dyes have very complex aromatic molecular structures. Therefore, Polluted water from the textile and dyeing industries is very difficult to eliminate because the complexity of the structures of the molecules makes them difficult to biodegrade and digest aerobically [1, 2]. The dyes used in dyeing have adverse effects on human health and the environment and even at low concentrations, pose a serious threat to the aquatic environment [3]. Studies have reported in the literature that some azo dyes have carcinogenic properties, particularly carcinogenic aromatic amines [4].

Thus, different methods for dye elimination were developed to limit their impact in the environment, including ozonation [5], coagulation-flocculation [6], microbial decomposition [7], photocatalytic degradation [8],

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Received: 17 November 2024; **Accepted:** 29 November 2024; **Published:** 16 December 2024

sono-chemical [9], filtration and membrane separation [10], electrochemical methods [11] and adsorption [12]. The adsorption process is one of the effective methods used for the removal of ionic dyes in aqueous media [13]. Activated carbon is cited as a good adsorbent for good removal of a wide range of dyes but its disadvantage is its cost [14]. In order to reduce the cost of treating wastewater from textile and dyeing units, attempts have been made to find inexpensive adsorbents. A lot of researches were used the activated carbon from biomass or agricultural wastes such as sisal fiber [15], surgarcane bagasse [16], rice husk [17] for the assessment of dyes in wastewaters. The limit of the use of activated carbon is the cost of preparation, the availability. Among agricultural by-products, rice husk is included in the large group because rice is cultivated as a food and cash crop. Our previous studies reported that rice husk contains carbon-rich compounds such as cellulose, hemicellulose, and lignin [18].

Therefore, the aim of our work is to study the adsorption efficiency of local waste (rice husks) to remove MB in an aqueous solution. Batch adsorption study was conducted, and adsorption isotherm, kinetics, and thermodynamics were evaluated. A physicochemical characterization of washed rice husk was carried out by the following methods: XRD, SEM, FT-IR and XRF.

2. Material and Methods

2.1. Preparation and Characterization of Adsorbent

The raw rice husk was obtained from farmers in a locality south of the capital of Burkina Faso. Once in the laboratory, the rice husk was washed several times with distilled water, to clean and remove any unwanted particles such as sand, pieces of wood, etc. It was then dried at 105 ℃ for 24 h and stored for later use. The washed rice husk (WRH) was crushed and then sieved to obtain particles between 0.500 and 0.800 mm. This bioadsorbent of the desired size was washed again, dried and then stored for later use.

Washed rice husk was characterized by FT-IR, XRD, XRF and SEM. Fourier transform infrared spectroscopic analysis (FT-IR) has been used to study the surface chemistry of WRH. The surface morphology of the washed rice husk was examined using an EDX-coupled SEM instrument. XRD and XRF analyses were performed to determine the mineralogical composition of the rice husk.

2.2. Adsorption Experiment

For the adsorption experiment, the dye used is methylene blue (MB), it is a cationic compound. The WRH was immersed in 50 ml of aqueous solution of methylene blue in the concentration range of 50 –250 mg/L for different contacts times. This parameter was studied by varying from 10 min to 90 min and the amount of WRH from 1 to 6 g. The system (WRH + MB solution) was kept under constant agitation at 150 rpm at room temperature. The residual MB concentration was determined by the UV-visible spectrophotometry method at the wavelength λmax = 665 nm. The adsorption percentage R (%) of MB and the amount of MB adsorbed q (mg/g), were calculated using the following equations.

$$R(\%) = \left(\frac{C_0 - C_e}{C_0}\right) x\ 100 \quad (1)$$

$$q(mg/g) = \frac{(C_0 - C_e)\ x\ V}{m} \quad (2)$$

where Co (mg/L) is the initial concentration of MB, Ce (mg/L) is the equilibrium concentration of MB, V (L) is the volume of the solution, and m (g) is the mass of WRH.

2.3. Adsorption Isotherms

Adsorption isotherms are necessary for the analysis of equilibrium data through mathematical equations to design and optimize a large-scale operational procedure. In this study, equilibrium data on methylene blue adsorption were analyzed using two models namely Langmuir and Freundlich.

$$Qe = \frac{Q_m * K_L * Ce}{1 + K_L * Ce} \quad (3)$$

$$Qe = K_f Ce^{1/n} \quad (4)$$

From Langmuir equation (3): Qe, Q_m and K_L are the amount of adsorbed MB at equilibrium, the amount, the maximum adsorption capacity (mg/g) and the adsorption equilibrium constant (L/mg) respectively.

From Freundlich equation (4): k_f (mg/g) and 1/n are Freundlich isotherm constant and Freundlich adsorption intensity parameter respectively.

2.4. Kinetic Adsorption

The data provided by kinetic studies provide fundamental information on the dynamics and mechanism of the adsorption process. Indeed, in this study, two of the most widely used kinetic models were used on the adsorption of MB by WRH: the pseudo-first-order equation and the pseudo-second-order equation. Each model defines the adsorption theory between the adsorbate and the adsorbent differently. The pseudo-first-order equation (5) considers one adsorbate molecule for one active site while the pseudo-second-order equation (6) considers one adsorbate molecule to be adsorbed on two active sites on the adsorbent surface [19].

$$ln(Qe - Qt) = lnQe - k_1 t \quad (5)$$

where K_1 (min^{-1}) is the rate constant of adsorption, Qt (mg/g) and Qe (mg/g) are respectively the amount of MB adsorbed at a given time and the equilibrium.

$$\frac{1}{Qt} = \frac{1}{K_2 Qe^2} + \frac{t}{Qe} \quad (6)$$

where K_2 (g/mg min) is the rate constant of pseudo-second-order biosorption.

3. Results and Discussion

3.1. Characterization of Washed Rice Husk

Figure 1 showed a scanning electron micrograph of washed rice husk. Through this micrograph, it can be observed that the WRH has an amorphous structure and presents an irregular and porous surface. We can note an irregular distribution of pores which gives a heterogeneous surface of WRH.

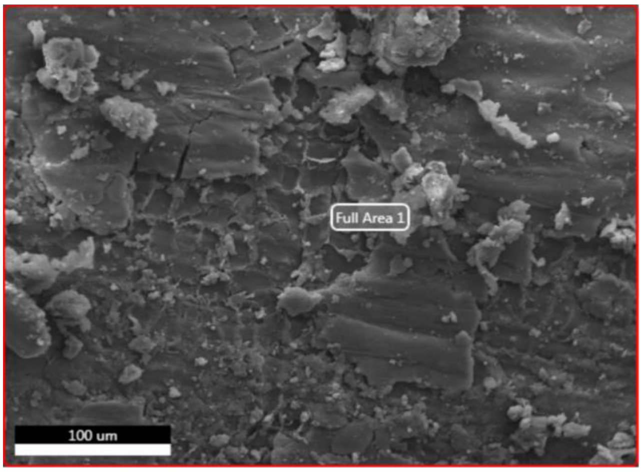

Figure 1. *Scanning electron micrograph of WRH.*

The chemical composition and percentage of elements of washed rice husk are recorded in Table 1. Trivially, five main elements stand out in their content from the values observed in the table, which are silicon (Si), phosphorus (P), potassium (K), calcium (Ca) and magnesium (Mg). Our findings support those of Adamu et *al*. who found these major elements, with the exception of phosphorus [20]. The chemical composition of washed rice husk consists of thirteen elements in the form of oxides, the major oxides of which are silicon dioxide 67.48% and phosphorus 16.8%. Comparable results are reported in the literature such as 61.4% [21]. The elemental composition of washed rice husk is related to their content in their oxide forms. These results correlate with those of the elemental analysis, in which silica represents 31.5% and phosphorus 7.34%. The total rate of the element was 49.28% and the remaining percentage would represent the carbon content of the washed rice husk; 50.72%.

Table 1. *Chemical composition of rice husk.*

Component	Weight (%)	Element	Concentration (%)
SiO_2	67.48	Si***	31.5
P_2O_5	16.8	P**	7.34
K_2O	4.67	K*	3.88
MgO	3.45	Mg*	2.08
SO_3	2.3	S	0.921
CaO	1.92	Ca*	1.37
Al_2O_3	1.7	Al	0.898
Fe_2O_3	0.834	Fe	0.583
MnO	0.365	Mn	0.282
Cl	0.27	Cl	0.271
TiO_2	0.12	Ti	0.0693
ZnO	0.0603	Zn	0.0485
Rb_2O	0.0379	Rb	0.0387
Total wt	100.00		49.282

The X-ray diffraction of washed rice husk shown in Figure 2 shows a mixture of amorphous and crystalline silica phases. The broad peak with maximum intensity at 2θ = 22° is observed and according the previous studies, this peak confirms the amorphous nature of silica [22]. The peak corresponding to 2θ = 22° is identified as cristobalite [23].

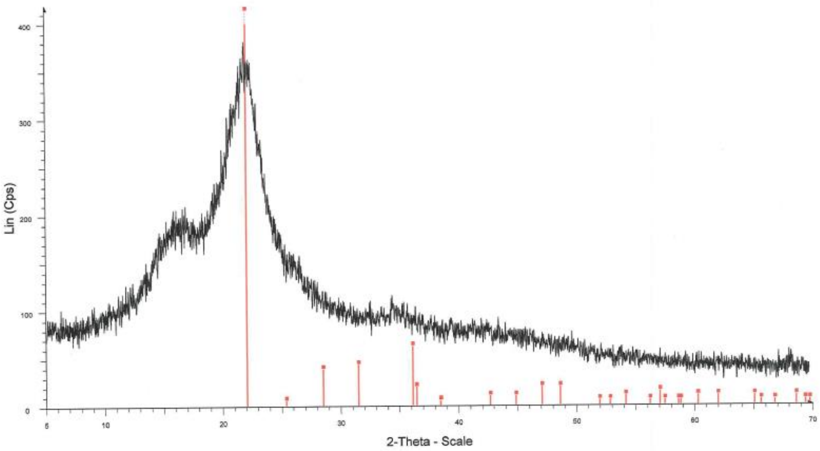

Figure 2. *XRD diffractogram of WRH.*

The figure 3 shows the infrared spectrum of washed rice husk recorded between 400 and 4000 cm^{-1}. Characteristic bands are observable through remarkable peaks and are summarized in the table 2. A broad bound at 3427 cm^{-1} was attributed to the silanol group (Si-OH) characteristic of rice husk, a biomass rich in silica. Previous study indicated that this peak was the one responsible of the adsorption property of rice husk [24]. Others bounds were observable at 2927 and 2866 cm^{-1}, according to the previous studies, were C-H in alkane and aldehyde stretching [24]. Double bounds C=O and C=C at 1743, 1655 cm^{-1} attributed to aromatic groups indicated the lignin in rice husk.

Table 2. *Main peaks of FTIR analyses of the WRH.*

Wavenumber (cm^{-1})	Functional group	references
3427	-OH, Si-OH	[24]
2927	C-H (alkanes)	
2866	Aldehyde C-H stretching	[24]
1743	C=O (aromatic groups)	[25]
1655	C=C (alkanes and aromatic group)	[26]
1514	C-C=C asymmetric stretching	[27]
1426; 1463	CH_2 and CH_3	[25]
1377	Aromatic C-H and carboxyl-carbonate	[28]
1079	Si-O-Si	[29]
793	Silane S-H	[25]

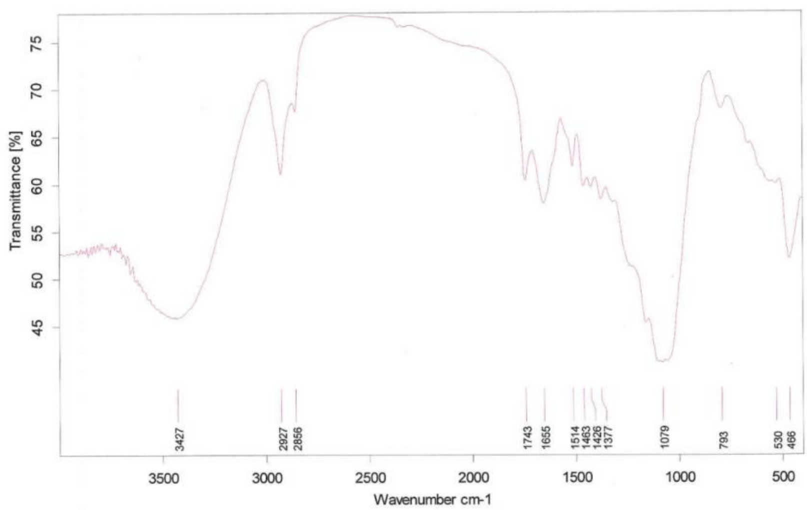

Figure 3. *FT-IR spectrum of WRH.*

3.2. Studies of the Adsorption

3.2.1. Effect of Adsorbent Dose

The effect of adsorbent dose on the cationic dye removal rate studied under laboratory conditions is shown in Figure 4. It can be observed that the percentage of the adsorbed dye evolves very slowly before increasing rapidly from a certain dose, reaching saturation. This performance may be due to the increase in active sites available for adsorption due to the increase of adsorbent dose. An adsorbent dose greater than or equal to 4.5 g has no remarkable effect on the adsorption of methylene blue. Clearly and as already reported by many authors [30], we find that the adsorption percentages increase with the reef ball used, reaching here more than 86% reduction of methylene blue. The maximum percentage varied from 85 to 86% between the dose of 4 and 5 g of adsorbents. Previous studies have reported that at high adsorbent doses, the number of MB dye molecules becomes insufficient compared to the active sites available on the adsorbent, leading to a steady state and a decrease in adsorption capacity (mg/g) [31]. According to Rida et *al*., the rate of adsorption reached the maximum after the critical concentration of the adsorbent has been used [32].

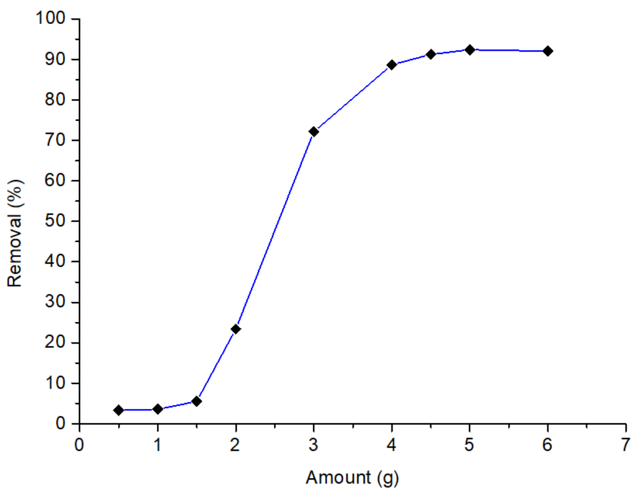

Figure 4*. Effect of the adsorbent dose on MB adsorption.*

3.2.2. Effect of Dye Concentration and Contact Time

In water and wastewater treatment, the contact time required for equilibrium and the maximum concentration of pollutants required by the adsorption experimental setup are important according to Ghanizadeh et *al*. [33]. Indeed, the graph in the figure 5 shows the evolution of the adsorption of methylene blue as a function of the contact time and at different concentrations varying at 50 to 250 mg/L and at room temperature. In a very short time, the amount of adsorbed BM increased and reached equilibrium after 30 min, reflecting the adsorption rate of BM on rice husk. In general, the adsorption phenomenon evolved rapidly in the first phase and remained constant during the second phase. This phenomenon can justify the high value of the slope of the adsorption line in the early stages of the reaction [33]. The rapid saturation may be due to the fact that the rice husk has not undergone chemical or physical activation that could increase the number of active sites.

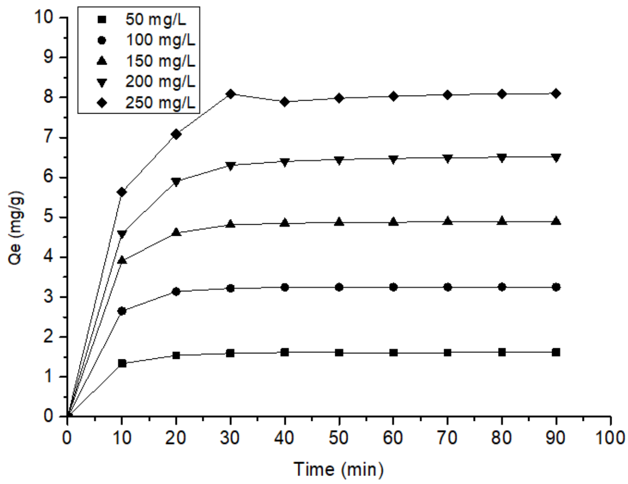

Figure 5*. Effect of contact time on MB adsorption.*

3.2.3. Kinetic Parameters of Adsorption

In the present study, pseudo-first-order and pseudo-second-order models are used to study the adsorption of methylene blue in water by rice husks. From the different initial concentrations of methylene blue (25–200 mg/L), pseudo first-order linear curves are shown in Figure 6(A). The main parameters such as the values of the first-order rate constant (k_1), the adsorption capacity (Qe) and the regression coefficient (R^2) are reported in the table 3. Generally, the values of R^2 obtained with the pseudo-first order model are very low according to Table 3 and the calculated Qe values are different from the experimental values. Previous studies have already shown the inadequacies of the pseudo-first-order model through studies on the adsorption of organic molecules by different adsorbents [19, 34].

Figure 6(B) corresponds to the graph of the pseudo-second-order linear model of the kinetic study representing t/Qt versus time. From the figure, it is observable that the graphs are closely linear and the parameters are recorded in Table 3. The calculated Qe value of first-order and pseudo-second-order kinetics is very close to the experimental Qe value. However, the correlation coefficient obtained for all the initial concentrations investigated was closed at 0.999 tending toward 1, showing the kinetic study obeys the pseudo-second-order model.

These findings suggest that the adsorption system under consideration follows a pseudo-second-order kinetic model. Previous studies have concluded that the pseudo-second-order kinetic model refers to chemisorption because it controls the adsorption rate [35, 36].

Table 3. Pseudo-First-Order model and Pseudo-Second-Order model form removal of MB by WRH.

C_0 (mg/L)	Q_e, exp (mg/g)	Pseudo-First Order Model			Pseudo-Second Order Model			
		k_1 (min^{-1})	Q_e, (mg/g)	R^2	k_2(g/mg.min)	h (mg/g.min	Q_e, (mg/g)	R^2
50	1.611	0,031	0.369	0.638	0.370	0.998	1.643	0.999
100	3.254	0.095	0.986	0.889	0.091	1.001	3.318	0.999
150	4.89	0.076	1.205	0.978	0.0397	0.998	5.016	0.999
200	6.506	0.076	2.023	0.954	0.022	1.014	6.790	0.999
250	8.093	0.058	1.402	0.581	0.014	1.004	8.469	0.998

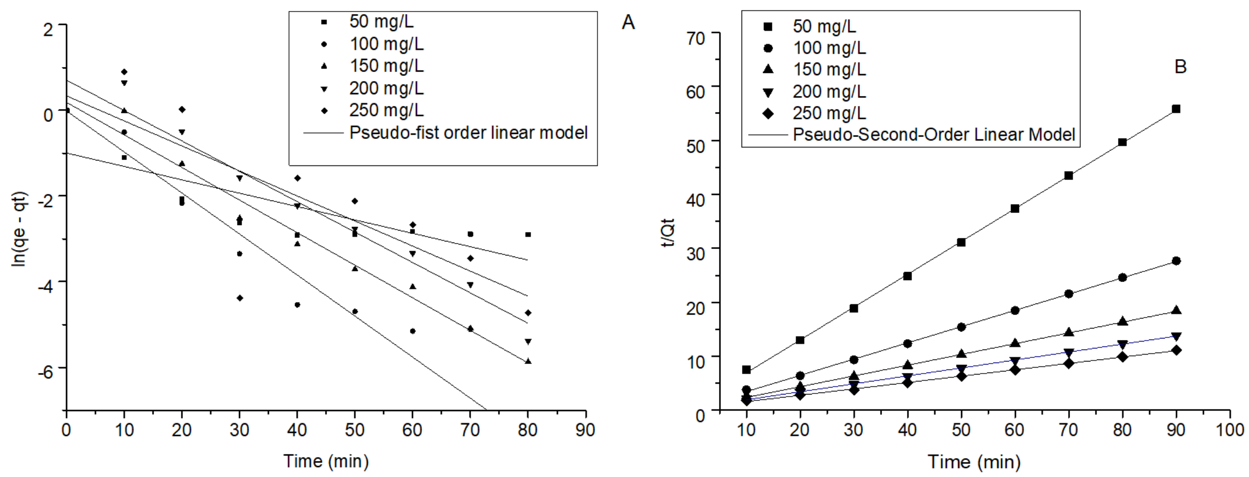

Figure 6. Adsorption kinetic fit for the removal of MB.

3.2.4. Adsorption Characteristics

The non-linear form of these models is presented on figure 7 and the parameters of the adsorption isotherm are summarized in Table 4. From table 4, it is indicated that the best fit of the experimental data is obtained with the Langmuir model, with a correlation coefficient R^2 of 0.98 compared to the Freundlich model (R^2= 0.94). The maximum adsorption capacity Q_m from the Langmuir model was 13.222 mg/g and it was compared with other data from the previous works. There is a good affinity between the dye molecules and the adsorption sites of the adsorbent when the K_L values are high and our result (0.117) is comparable to that of Melo et *al.* (0.139) [37]. It is reported in the literature that adsorption is favorable for n > 1 [38]. In our study, 1/n = 0.598 implies that n =1.67, then confirms that the adsorption of MB on washed rice husk is favorable.

Table 4. Isotherm parameters and correlation coefficients calculated for the adsorption of MB.

Model	Parameters	Values
Langmuir	Q_m (mg/g)	13.227
	K_L (L/mg)	0.117
	R^2	0.979
Freundlich	K_f (mg/g)	1.758
	1/n	0.598
	R^2	0.942

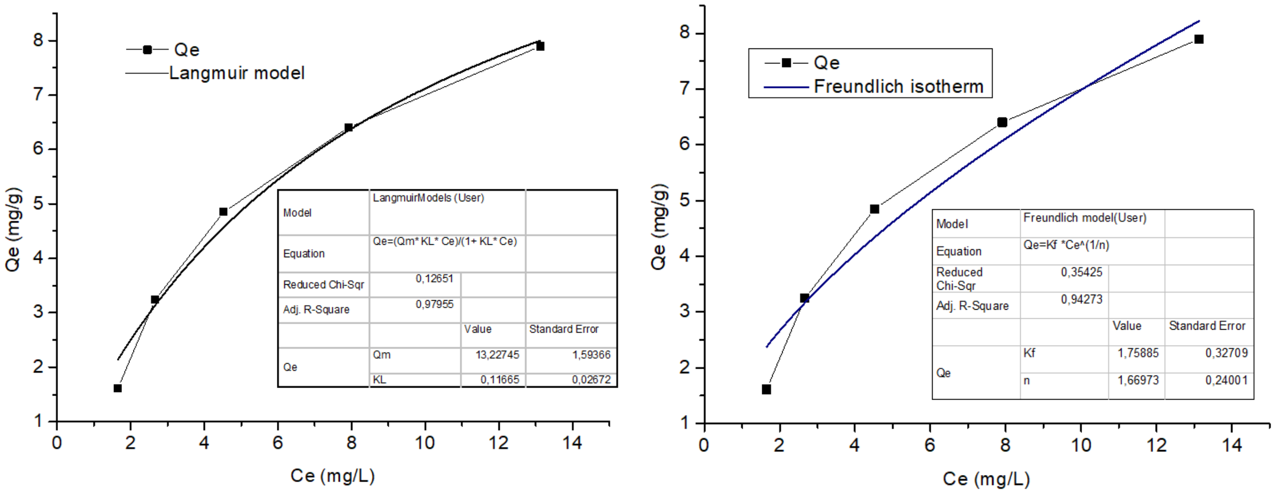

Figure 7. *Experimental points and non-linear fitted isotherm curves: MB onto WRH.*

A dimensionless parameter called the separation factor, R_L is related to the adsorption property and its calculated values at different initial methylene blue concentration are shown in Figure 8. According to the literature, adsorption is favorable for RL values between 0 and 1 [39]. According to the data in Figure 8, the $R_L$values range from 0.02 to 0.15, which then confirms that the adsorption of MB on WRH is favorable. The decrease in R_L with increasing initial MB concentration is in agreement with literature results [12], indicating favorable adsorption at high concentrations.

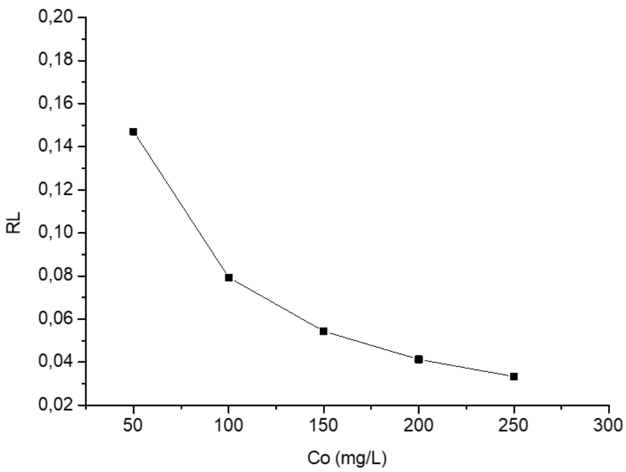

Figure 8. *Calculated R_L values at different initial concentration of MB.*

3.3. Adsorptions Mechanisms

According to the results of physicochemical analysis, washed rice husk has the best assets of a better candidate for MB adsorption. The composition of washed rice husk is rich in silica (O-Si-O and Si-O-H) which is a good adsorbent for organic pollutants by the possible formation of hydrogen bonds with the free electron pairs of oxygen. Previous work has shown that the identified C–OH, C=O, C–O and −OH functional groups were responsible for the adsorption of MB dye [40]. These functional groups are present in the compounds that group the lignin family. Therefore, previous studies have reported that chemisorption can be carried out through polar lignin compounds such as alcohols, ketones, aldehydes, phenolic hydroxyls and ethers as they are chemical bonding intermediates [19]. Previous studies have shown that the adsorption mechanism of MB from aqueous solution onto solid adsorbent would be mainly due to a number of chemical interactions such as electrostatic attraction, hydrogen bonding, π-π bonding, and ion exchange [41]. All these findings also support the hypothesis on the pseudo-second order model according to which the MB adsorption phenomenon is due to chemisorption.

4. Conclusion

In our study, the use of a local adsorbent, available for the study of a dye, was discussed. Physicochemical analyses showed that WRH is a silica-rich carbonaceous material full of chemical functions (-OH, Si-O-Si, C=O). In addition, it has an amorphous structure dominated by cristobalite. The K_L parameter of the Langmuir model showed an affinity of MB with the WRH. The R_L values calculated for all different initial concentrations are between 0 and 1, showing the MB adsorption process is favorable. The kinetic study showed that the adsorption of MB follows the pseudo-second order model attesting to chemical sorption. Washed rice husks can be a low-cost solution to environmental pollution caused by wastewater from industrial dyes used in dyeing textiles and fibers.

Abbreviations

XRD X Ray Diffraction
SEM Scanning Electronic Microscopy
FT IR Formed Transformed Fourier
XRF X Ray Fluorescence

Author Contributions

Nombamba Ouéda: Conceptualization, Data curation, Formal Analysis, Funding acquisition, Investigation, Methodology, Project administration, Resources, Writing – original draft, Writing – review & editing
Samadou Sanni: Software
Moctar Limam Bawa: Validation

Conflicts of Interest

The authors declare no conflicts of interest.

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