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EXPLORING THE POTENTIAL OF AGRICULTURAL WASTE AS NATURAL RESOURCE-BASED ADSORBENTS FOR METHYLENE BLUE REMOVAL

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Abstract

Excessive agricultural waste in the agricultural industry leads to various forms of pollution, including water pollution. To address this issue, there's a growing interest in finding alternative methods. One approach is to utilize agricultural waste as natural resource-based adsorbents to eliminate contaminants, such as the case of methylene blue (MB) in this study. The study specifically focuses on using rice husk (RH) from a local rice mill in Perlis, Malaysia, to absorb methylene blue. The structure of rice husk, characterized by scanning electron microscopy (SEM), reveals a coarser and more compact outer area, contributing to its absorption capacity for methylene blue. The study on rice husk involves three main aspects: contact time, adsorbent dosage, and dye concentration. The removal percentage of MB increased as the three studied adsorption parameters increased. The adsorption data were analyzed using Langmuir and Freundlich adsorption isotherms, with the the Freundlich Isotherms were found to be more suitable based on higher coefficient of correlation (R^2) values compared to Langmuir. The pseudo-second-order kinetics model demonstrated a higher R^2 value (1.00) compared to the pseudo-first-order model (0.747). The results indicate promising potential for addressing pollution through sustainable means and provide insights into the adsorption process under varying conditions.

***Keywords**: Adsorption; Dye; Methylene blue; Rice husk; Isotherms; Kinetics*

Introduction

In the present era, prioritizing environmental protection against the adverse impacts of modern technology and industrial pollution has emerged as a paramount objective due to potential hazards to public health and overall well-being. The presence of color in water serves as an unmistakable indicator of contamination, evoking substantial concerns regarding environmental soundness. The discharge of colored wastewater from a wide spectrum of industries engaged in dye and pigment utilization across sectors such as textiles, paper, cosmetics, agriculture, plastics and leather, poses profound environmental complexities. The harmful attributes of specific dye substances not only jeopardize the natural scenery but also disrupt the ecological equilibrium

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among aquatic life forms, vegetation and, ultimately, human welfare within the intricate web of the food chain [1, 2].

Methylene blue (MB) plays diverse roles in fields like microbiology, surgery, diagnostics, and acts as a sensitizer in the photo-oxidation of organic pollutants [3, 4]. Extensive research has been conducted on MB, particularly in representing dye compounds within aqueous solutions. Its versatility is showcased through applications like paper coloring and fabric dyeing. However, MB's presence as an environmental contaminant in surface water can lead to detrimental effects on both human health and ecosystems [5, 6].

Addressing the removal of color from effluents before their discharge into the environment becomes a critical consideration. Conventional chemical wastewater treatment methods, such as biological oxidation and chemical precipitation, target this challenge, primarily proving cost-effective for higher dye concentrations. Consequently, the exploration of alternative techniques, particularly adsorption, has gained significant traction due to its capacity for sludge-free operations and the efficient elimination of dyes from diluted solutions [7].

Adsorption has proven to be an effective approach for eliminating colors from wastewater. Various adsorbents, including activated carbon, date fiber, sawdust, oat bran and chitosan, have been investigated for their adsorption capabilities [8]. Although commercially efficient activated carbon is highly effective, its costliness prompts environmental engineers to explore cost-effective absorbents derived from agricultural waste sources [9, 10]. Agricultural waste offers a practical and bio-friendly option for these absorbents. The benefits include affordability, high efficacy, minimal waste generation and the potential for recycling [9].

Recent attention has focused on rice husk (RH) as a promising adsorbent for removing acid dyes, such as acid yellow [7] and methylene blue [5]. These studies demonstrate the inherent dye adsorption potential of raw RH. Additionally, enhancing the performance of RH through pyrolysis and carbonization to produce rice husk ash (RHA) has yielded improved dye removal results [11, 12]. However, these well-controlled manufacturing processes tend to require significant energy and time investment.

Dibuk Rice Factory Sdn. Bhd., operating within the Group Consortium International Rice Engineering (Thailand) Bernas Factory Buddies, functions as a commercial rice mill situated in Perlis, Malaysia. It specializes in the industrial processing of paddy rice and serves as a significant supplier of rice to major wholesalers across the country. On a daily basis, residual rice husk is generated as a byproduct of the rice milling process at this facility.

In light of this context, the present research aims to underscore the potential utility of rice husk (RH) extracted from Dibuk Rice Factory Sdn. Bhd., in its original form as an adsorbent for MB dye removal. The study seeks to demonstrate the applicability of RH without requiring further modifications. Key aspects such as adsorbent dosage, retention time, dye concentration, adsorption kinetics and isotherm characteristics will be comprehensively investigated as part of this research endeavor.

Experimental part

Adsorbent

In this research, rice husk (RH), a residual product from the local rice milling industry, was employed as an adsorbent. The RH was initially subjected to a series of preparatory steps. Initially, any foreign particles were manually removed, followed by thorough rinsing with tap water. Subsequently, the RH underwent multiple rinses with distilled water until the elimination of the yellow color caused by lignin was achieved. The dried RH was then placed in an oven at 60°C for 6 hours for further drying. Subsequently, the dried RH was processed into a powdered form using a grinder machine. To ensure uniformity, the RH powder was generated by grinding and sieving, aiming for a consistent particle size of 300μm [13].

Adsorbate

To prepare the dye stock solution (1000mg/L) for the adsorbate, 1.0g of methylene blue (MB) was dissolved in 1 liter of distilled water. All reagents used in this study were of analytical grade. To obtain various solution concentrations, required dilutions were achieved using distilled water, starting from the stock solution. The dye stock solutions employed in the experiments were set at 20mg/L. For quantifying the adsorbate concentrations, absorbance measurements were taken at 664nm using a UV–Vis spectrophotometer both before and after each experiment.

Chemical composition and morphology test

Scanning Electron Microscopy (SEM) with Energy Dispersive X-Ray Analysis (EDX) was utilized to analyze the texture, elemental composition and chemical properties of rice husk (RH). Prior to analysis, the RH underwent sieving with a 75µm sieve size (No. 200 sieve) to eliminate larger particles that might interfere with the analysis due to their significant size variation. The SEM machine was utilized to meticulously scan the surface of the FA sample using an electron beam. The test incorporated secondary and backscattered electron detectors, which enabled the interaction signals to generate a visual representation of the morphology and chemichal properties of the RH sample [14].

Batch adsorption experiment

In this study, 50mL of dye solutions containing 20mg/L of dye were transferred to a conical flask and mixed with 1.0g of the adsorbent until equilibrium was attained. Subsequently, a volume of approximately 2 milliliters cube of the solution was directly transferred to a cuvette, and the absorbance of the dye was measured using a UV-vis spectrophotometer at different time intervals. All experiments were conducted at room temperature and were repeated three times to ensure the precision of the collected data. The efficiency of methylene blue dye removal (expressed as a percentage) adsorbed at various intervals (q_t, mg/g) and at equilibrium (q_e, mg/g) was determined using Equations 1, 2 and 3.

$$Percentage\ Removal\ (\%\ R) = \frac{C_o - C_e}{C_o} \times 100 \tag{1}$$

$$q_t = \frac{(C_o - C_t)v}{w} \tag{2}$$

$$q_e = \frac{(C_o - C_e)v}{w} \tag{3}$$

where: C_o - initial concentrations of MB (mg/L), C_t - time concentrations of MB (mg/L), C_e - equilibrium concentrations of MB (mg/L), W - the weight of adsorbent (g), V - the solution volume (L).

Effect of contact time

The experimentation aimed to determine the optimal adsorption equilibrium time. The assessment involved a series of trials with varying mixing durations: 5, 10, 15, 30, 45 and 60 minutes, respectively. The dose of adsorbent used was 1.0g with 20mg/L concentration adsorbent and 50mL of the adsorbate. Then, the samples were shaken using orbital shaker at the constant rate (160rpm) at room temperature.

Effect of adsorbent dosage

A range of adsorbent dosages was employed in this experiment. The adsorbent dose was subjected to incremental variations, including amounts of 0.1, 0.2, 0.3, 0.5, 0.9 and 1.0g. These quantities were mixed with 50mL of the adsorbate containing a concentration of 20mg/L, with a fixed contact time of 2 hours. The samples were shaken using an orbital shaker at room temperature (29°C) and a consistent rotation speed of 160 revolutions per minute (rpm).

Effect of dye concentration

This experiment aimed to investigate the influence of dye concentration and contact time. The study involved preparing a series of stock solutions with different concentrations, specifically 40, 60, 80, 100 and 120mg/L. Each concentration was mixed with 50mL of adsorbate.

Subsequently, 1.0g of the adsorbent was added to each concentration mixture. The samples were then shaken using an orbital shaker at a constant rotation speed of 160rpm. Sampling was carried out at specific time intervals: 30, 60, 90, 115 and 120 minutes, respectively. This experiment aimed to assess how the dye concentration and contact time influenced the adsorption process.

Adsorbent isotherms and kinetic models

The equilibrium experimental data were analyzed using Langmuir and Freundlich adsorption isotherm models. The linear expression for the Langmuir isotherm is depicted by Equation 4:

$$\frac{C_e}{q_e} = \frac{1}{K_L Q_a^o} + \frac{C_e}{Q_a^o} \quad (4)$$

The expression for the Freundlich isotherm model is depicted by Equation 5:

$$log\ q_e\ = log\ K_F\ + \frac{1}{n} log\ log\ C_e \quad (5)$$

where: $Q^0{}_a$ (mg/g) and K_L (L/mg) are Langmuir constants related to capacity and rate adsorption, subsequently. $Q^0{}_a$ and K_L values can be figured from the linear plot C_e/q_e versus C_e. K_f is the constant represent for the adsorption capacity linked to bond of strength. While 1/n is constant attributing intensity of adsorption. K_F and n were persuaded based on the linear plot of log q_e versus log C_e.

Batch kinetic data collected for MB adsorption onto RH were interpreted utilizing pseudo-first order and pseudo-second-order methods. Equation 6 expresses the pseudo-first-order kinetic equation:

$$log\ (q_e - q_t) = \ log\ q_e\ + \frac{k_1}{2.303}\ t \quad (6)$$

where: q_e and q_t (mg/g) are the dye entrapped on RH at equilibration and time t (min), respectively k_1 (min) denotes the adsorption rate constant.

For the pseudo-second-order model, the equation is expressed as Equation 7:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e}\ t \quad (7)$$

where: k_2 is the rate constant for pseudo-second-order adsorption model.

Results and discussion

Material Characterization

The analysis of raw rice husk was carried out using energy-dispersive X-ray analysis (EDX) and scanning electron microscopy (SEM). These observations were conducted to characterize the material, with the aim of identifying its structural attributes as well as the composition of the elements present within rice husk.

Chemical Composition

The outcomes have been visually presented in figure 1. Analysis of the graph reveals that the sample primarily consists of carbon (C), oxygen (O), silica (S), and potassium (K). Additionally, trace amounts of other elements, such as phosphorus (P) and calcium (Ca), were detected. While the sample also contains minor quantities of elements like magnesium (Mg) and aluminum (Al), their percentages are relatively low. Further analysis of the graph shows that carbon (C) constitutes the most substantial portion within raw rice husk, accounting for 48.59% of its mass, followed by oxygen (O) at 38.72%, silica (S) at 7.92%, potassium (K) at 2.71%, phosphorus (P) at 0.99%, and calcium (Ca) at 0.35%.

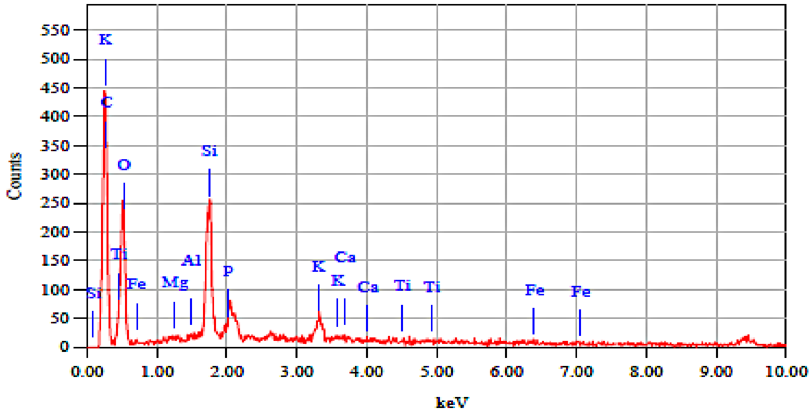

Fig. 1. Chemical composition of raw rice husk

Morphological of rice husk

SEM analysis was conducted to characterize and examine the structural texture of raw rice husk. Figure 2 illustrates the SEM findings, providing magnifications at a) 500x, b) 1000x and c) 2000x. The results reveal that the sample exhibits a rough surface with small particles. Notably, various particles exhibit distinct shapes, including block-like structures reminiscent of mossy lumps and other irregular formations. The modification of the morphological structure was confirmed by the observed increase in specific surface area. These findings align with the research conducted by Khalilollah [15], which similarly indicated that raw rice husk possesses a rough surface.

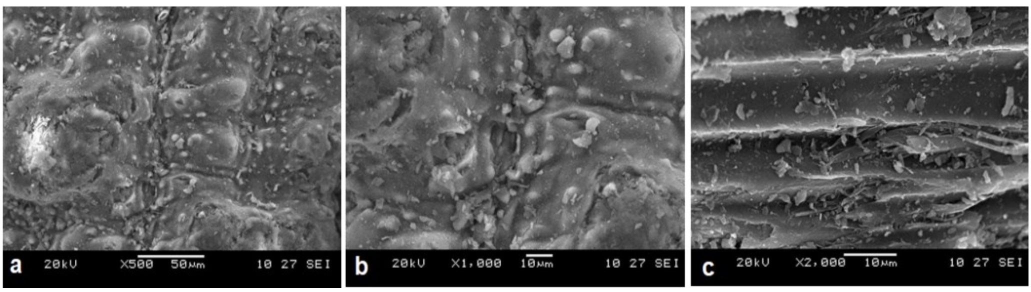

Fig. 2. SEM image of raw rice husk with magnification a) 500, b) 1000 and c) 2000

Effect of contact time

Investigations into the impact of contact time on the adsorption of methylene blue onto rice husk have been conducted. These studies involved the utilization of 1 g of absorbent and a 20 mg/L concentration of dye stock solution. The absorbate employed for this testing comprised raw rice husk with a particle size of 300 µm. Each sample was subjected to varying contact times, specifically 5, 10, 15, 30, 45 and 60 minutes.

As depicted in figure 3, the adsorption of methylene blue exhibited a swift progression in the initial phase, particularly at the 5 minute mark. Subsequently, the adsorption capacity of methylene blue experienced a decline as the contact time was extended beyond 10 minutes. However, the adsorption rate stabilized after the 10 minute mark and remained relatively constant until the conclusion of the 60 minute testing period. This behavior can be attributed to the attainment of adsorption equilibrium, signifying that the adsorbent had reached its saturation point.

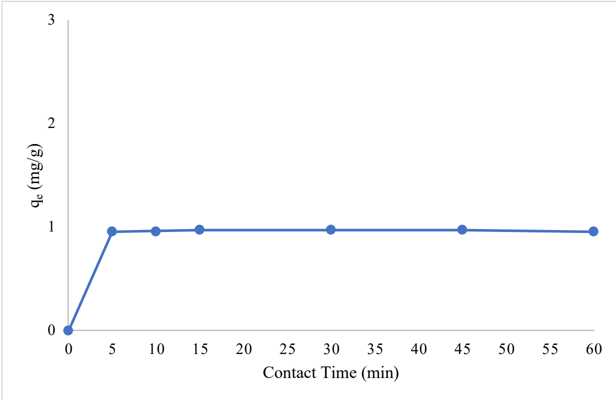

Fig. 3. Effect of contact time for adsorption of methylene blue onto rice husk

Based on the data presented in figure 4, it is evident that the percentage removal of methylene blue by raw rice husk was examined. In this specific experiment, a consistent amount of 1.0g of adsorbent was used for each sample tested. During the initial 5 minutes, a rapid increase in the percentage removal was observed, reaching 95.94%. Notably, at the 10 minute mark, distinct adsorption activities became apparent, resulting in a percentage removal of 96.03%. As the contact time extended to 15 minutes, the percentage removal further increased to 96.34%. Subsequently, over a duration of 30 minutes, the percentage removal demonstrated a gradual rise from 97.27% to 97.40%, and this level of removal remained constant until the conclusion of the hour-long testing period.

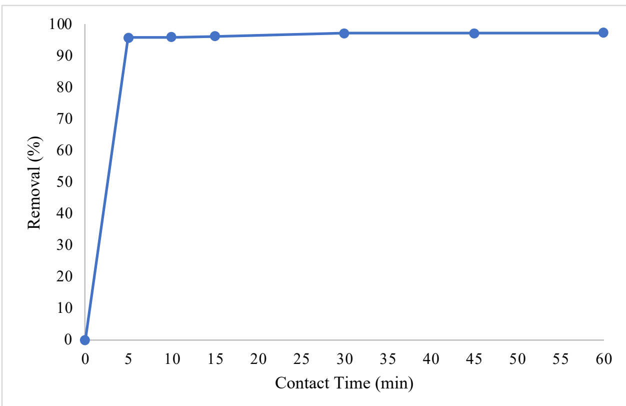

Fig. 4. Percentage of removal for effect of contact time for methylene blue adsorption onto rice husk

This study shares similarities with the research conducted by Ahmad Nasir *et al.* [13], where they observed the effect of contact time on adsorption. Notably, they found that the greatest impact was achieved when the initial dye concentration was 50mg/L, and the adsorbent dose was 0.05g. The rate of adsorption displayed a notable increase in the initial stages, yet the amount of methylene blue (MB) adsorbed decreased as contact time was extended. This downward trend persisted until saturation was achieved. After a cumulative period, no further change in the MB concentration was evident, indicating that equilibrium was attained. This phenomenon arises from the saturation of active sites, preventing further adsorption. Similar findings were also reported by Hameed *et al.* [16].

Effect of adsorbent dosage

Figure 5 illustrates the graphical representation of methylene blue adsorption on rice husk, revealing a distinct trend in the adsorption pattern. Notably, smaller dosages of the adsorbent exhibited the ability to adsorb methylene blue with efficiency at low concentrations, whereas the adsorption process was relatively delayed for higher concentrations and larger dosages. The recorded adsorption values for various dosages are as follows: 0.1g resulted in 10.07mg/g, 0.2g yielded 5.03mg/g, 0.3g showed 3.34mg/g, 0.5g displayed 2.00mg/g, 0.9g exhibited 1.10mg/g, and 1.0g demonstrated 0.96mg/g. As the adsorbent dosage increased, the uptake capacity of rice husk declined.

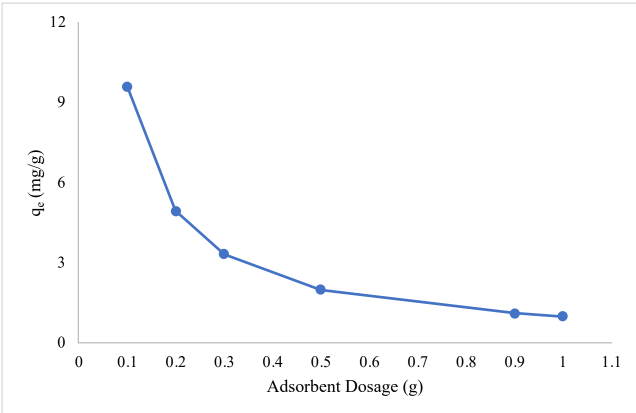

Fig. 5. Effect of adsorbent dosage for methylene blue adsorption onto rice husk

Similar to the findings of Ahmad Nasir *et al.* [13], who investigated the impact of adsorbent dosage, this study also revealed a comparable trend. Specifically, it was observed that the equilibrium dye absorption quantity (q_e) exhibited a decrease as the adsorbent dosage was increased, while maintaining an initial dye concentration of 50mg/L. This observed phenomenon can be attributed to the augmented adsorbent dosage, which consequently led to an increase in available surface area and a greater abundance of adsorption sites. Notably, similar trends have been documented in other studies, such as those conducted by Malik *et al.* [17] thereby validating the consistency of this observation across different contexts.

The outcomes depicted in figure 6 portray the percentage removal of methylene blue onto rice husk, elucidating the effect of various dosages.

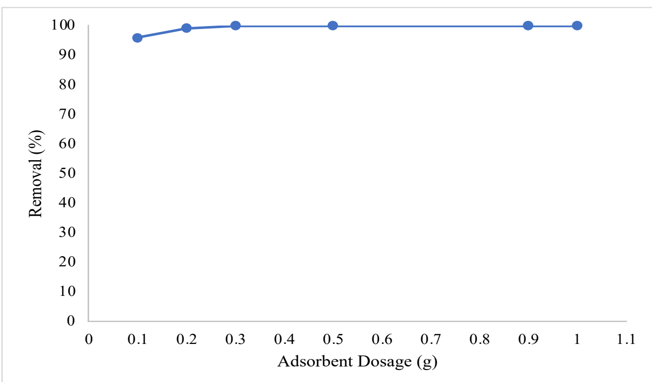

Fig. 6. Percentage removal of adsorbent dosage effect for methylene blue adsorption onto rice husk

The study encompassed dosages of 0.1, 0.2, 0.3, 0.5, 0.9 and 1.0g of the adsorbent, the particle size was 300µm, and the experimental conditions encompassed a concentration of 20mg/L and a contact time of 2 hours. The results reveal that for the dosages of 0.1 and 0.2g, the corresponding percentage removals were 95.94% and 99.05%, respectively. In contrast, the other adsorbent dosages, namely 0.3, 0.5, 0.9 and 1.0g, exhibited a 100% percentage removal of methylene blue. The attainment of 100% removal for these dosages is attributed to the complete adsorption of methylene blue by the rice husk. However, for the lower dosages of 0.1 and 0.2g, although the adsorption was also complete, the extended stirring duration led to a gradual fading of methylene blue from the rice husk.

The testing outcomes ultimately suggest that even small dosages of rice husk can effectively adsorb methylene blue across varying concentrations, particularly for low concentrations. Nonetheless, for higher concentrations, a 2-hour contact time may prove insufficient, as the inherent adsorption capacity of raw rice husk becomes limited.

Effect of dye concentration and contact time

The investigation into the effect of dye concentration and contact time was conducted using 1.0g of the adsorbent, encompassing various concentrations (40, 60, 80, 100, 120 mg/L) and contact durations (30, 60, 90, 105 120 minutes). This study aimed to elucidate the potential impact of contact time on distinct dye concentrations.

As depicted in figure 7, the adsorption process exhibited a consistent upward trend for both different contact times and varying concentrations. The observed pattern showcased a continuous increase in adsorption efficiency from the initial 30 minutes of testing up to the 120-minute mark across the range of concentrations (40, 60, 80, 100 120mg/L). This progressive rise in adsorption can be attributed to the ongoing adsorption activity, which persists until the conclusion of the test duration. Notably, this trend is more pronounced for higher dye concentrations, wherein the extended contact time is needed for raw rice husk to effectively adsorb methylene blue due to the heightened concentration of the dye.

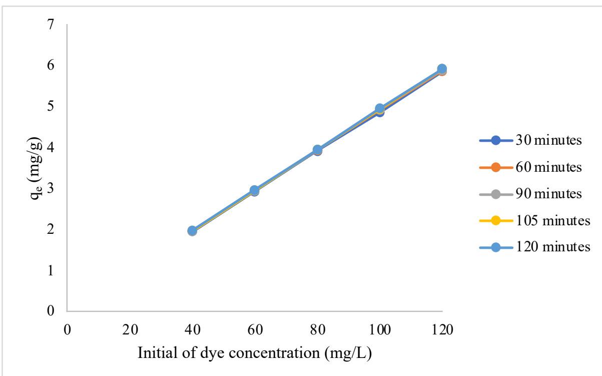

Fig. 7. Effect of dye concentration and contact time for adsorption of methylene blue onto rice husk

The findings from figure 8 reveal certain inconsistencies in the percentage removal of methylene blue for samples tested at 30 and 120 minutes of contact time. Ideally, the results should exhibit a consistent increase as the contact time lengthens. However, these irregularities can be attributed to potential contamination or alteration of the samples during storage or retrieval. It is possible that the samples were exposed to plain water or other external factors after being stored in the refrigerator, leading to variations in the observed percentage removal.

On the other hand, for the samples tested at 60, 90 and 105 minutes of contact time, a clear increasing trend in the percentage removal can be observed for concentrations of 40, 60 and 80mg/L. However, for concentrations of 100 and 120mg/L, the adsorption percentage decreases.

This decline is likely due to the high concentration of the adsorbate relative to the limited capacity of the small dosage of raw rice husk to adsorb.

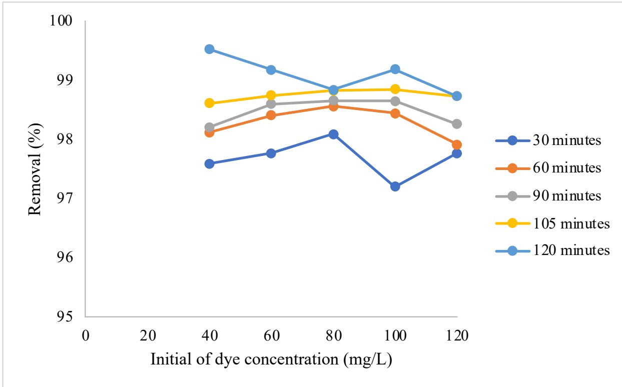

Fig. 8. Percentage removal of effect of dye concentration and contact time for adsorption of methylene blue

In conclusion, for higher concentrations such as 120mg/L and a dosage of 1.0g of adsorbent, a longer contact time is required to achieve effective adsorption of methylene blue. The percentage removal increases progressively with extended contact times: 97.77% at 30 minutes, 97.91% at 60 minutes, 98.26% at 90 minutes, 98.73% at 105 minutes and 98.73% at 120 minutes.

The gradual increase in the percentage of methylene blue removal can be attributed to the inherent structural characteristics of raw rice husk. The composition and texture of raw rice husk create a situation where a longer contact time is necessary for effective adsorption of methylene blue, even with a small dosage of the adsorbent. This behavior can be understood as a result of the complex surface features of raw rice husk, which require more time for the dye molecules to interact and bind with the available adsorption sites. As a consequence, the adsorption process becomes more time-dependent, causing the percentage of removal to increase slowly over extended contact periods.

In line with the research conducted by [13] on the impact of dye concentration and contact time for MB dye, a comparison can be drawn. When employing lower dye concentrations, along with an adsorbent dosage of 0.05g, the study observed nearly complete removal of the dye, reaching almost one hundred percent efficiency. However, a decline in the percentage removal was noted when higher dye concentrations were utilized, resulting in removal rates of 93 percent and 95 percent, respectively. This trend could be attributed to the relationship between the ratio of dye molecules and the availability of adsorption sites. At lower concentrations, the dyes exhibit a higher affinity for the adsorption sites due to the relatively lower competition for these sites [18].

Adsorption isotherms

The adsorption behavior of methylene blue onto raw rice husk was investigated through the application of Langmuir and Freundlich isotherms. The equilibrium isotherm parameters play a crucial role in the design and optimization of aqueous phase adsorption systems [19]. To ascertain the validity of the adsorption experiment, rigorous isotherm models were employed. The correlation coefficients derived from the curve fitting of these adsorption models indicated a closer agreement between the methylene blue adsorption data and the Langmuir and Freundlich isotherm models. The Langmuir isotherm model is employed to characterize monolayer adsorption, providing insights into the adsorption capacity of a material. In contrast, the Freundlich isotherm model is utilized to elucidate biosorption on heterogeneous surfaces and to account for multilayer adsorption phenomena. The coefficients corresponding to both isotherms are depicted in figures9 and 10.

According to figure 10, the adsorption data align more closely with the Freundlich model, as indicated by the higher coefficient of correlation (R^2) value of 0.9305. In contrast, the Langmuir model yields an R^2 value of 0.8505 as seen in figure 9. This suggests that the Freundlich model provides a better fit for the adsorption data. The Freundlich isotherm's parameter (1/n), representing the adsorption intensity on the heterogeneous surface of the bio-sorbent, underscores the ease with which the MB dye can be taken up.

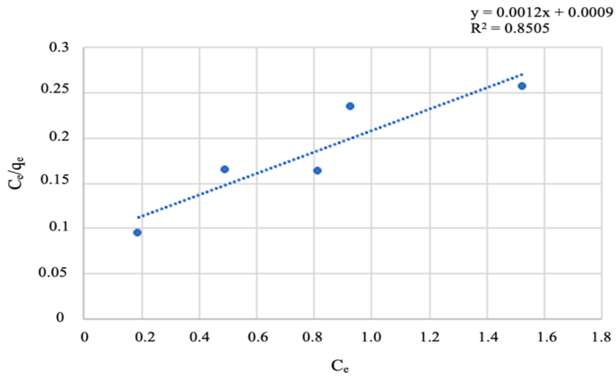

Fig. 9. Adsorption of methylene blue for Langmuir Isotherms

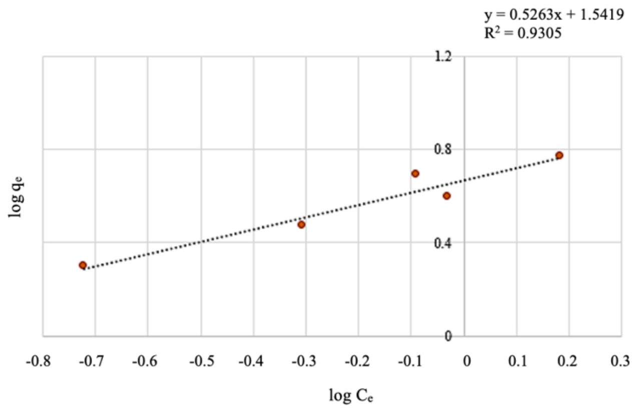

Fig. 10. Adsorption of methylene blue for Freudlich Isotherms

Kinetic models

The adsorption kinetics of methylene blue (MB) onto rice husk (RH) were investigated using the pseudo-first-order and pseudo-second-order kinetic models. The equations representing these models are provided as Equations 6 and 7, respectively. The outcomes of the experimental data were compared of the pseudo-first order and pseudo-second-order models for MB adsorption on RH, as depicted in figures 11 and 12. The analysis of figure 12 reveals a consistent pattern in the R^2 values obtained from the pseudo-second-order kinetic model, with an R^2 value of 1. This indicates that the kinetic adsorption data of methylene blue (MB) is better fitted by the pseudo-second-order model.

In the study conducted by A.N. Laban *et al.* [13], it was observed that the pseudo first-order model did not adequately describe the kinetic adsorption data for methylene blue (MB). Instead, the research revealed that the kinetic adsorption data exhibited a closer fit to the pseudo-second-order model, as evidenced by the obtained R2 values approaching 1.

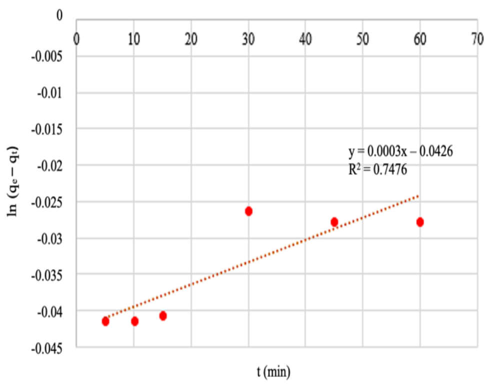

Fig. 11. Pseudo-First-Order of adsorption kinetic model

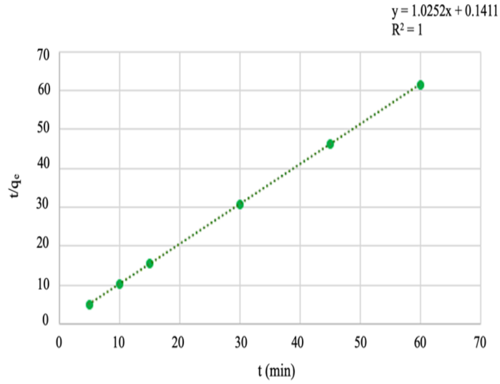

Fig. 12. Pseudo-Second-Order of adsorption kinetic model

Thus, the findings of this study support the conclusion that the pseudo-second-order model provides a more accurate representation of the kinetics of MB adsorption onto rice husk.

Conclusions

In this study, the potential of rice husk obtained from a local rice mill in Perlis was investigated for its ability to adsorb methylene blue from wastewater. The characterization of rice husk through Energy Dispersive X-ray (EDX) and Scanning Electron Microscopy (SEM) analyses revealed that it possesses a rough surface with small particles. The elemental composition analysis indicated the presence of carbon, oxygen, silica, and kalium in rice husk, along with traces of elements such as phosphorus and calcium.

The effective adsorption capacity of rice husk for methylene blue can be attributed to its unique textural structure, characterized by porosity. This porosity provides rice husk with the advantage of adsorbing a significant amount of methylene blue from wastewater. The utilization of rice husk as an adsorbent offers a promising alternative for efficient methylene blue removal, considering its affordability and easy availability, especially given the abundant rice production in Malaysia.

In terms of adsorption kinetics, the results support the effectiveness of the pseudo-second-order model, as indicated by the perfect fit with an R^2 value of 1. As for the adsorption isotherms, the higher correlation coefficient values (R^2) obtained from the Freundlich model suggest that methylene blue adsorption onto rice husk is well-described by this model.

In conclusion, the study underscores the potential of rice husk as an effective adsorbent for methylene blue removal from wastewater. The unique textural properties of rice husk, along with its affordability and easy accessibility, make it a viable and environmentally friendly option for wastewater treatment applications.

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