Assessing the Viability of Orange Charcoal as an Adsorbent for the Removal of Methyl Orange Dye from Aqueous Solutions

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تقييم جدوى فحم البرتقال كمادة مازة لإزالة صبغة الميثيل البرتقالي من المحاليل المائية

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Abstract

This study aimed to explore the use of orange charcoal, which can be obtained at a low cost from local markets in Libya, as an effective adsorbent for the removal of Methyl Orange (MO) from aqueous solutions. The disposal of dyes into the environment, including MO, has become a global concern due to its negative impact on water resources. The study investigated the effects of various parameters such as concentration, contact time, pH, and adsorbent dosage, on the adsorption process in batch experiment at 25^oC, and evaluate the adsorption efficiency, kinetics, and equilibrium isotherms associated with MO removal using activated carbon derived from orange charcoal as an adsorbent. The results showed that the orange charcoal activated with HCL was an effective adsorbent for the removal of MO, and the adsorption capacity of the adsorbent was determined using the Freundlich isotherm. The study concluded that the pseudo-second-order model was well-fitted to the experimental data. In summary, this study suggests that orange charcoal activated by HCL (OCAC) could be a useful and cost-effective method for the removal of MO from aqueous solutions.

Keywords: *Methyl orange, Orange charcoal, Isotherms, Kinetics, Adsorption, Chemical activation.*

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الملخص

هدفت هذه الدراسة إلى استكشاف استخدام فحم البرتقال، والذي يمكن الحصول عليه بتكلفة منخفضة من الأسواق المحلية في ليبيا، كممتز فعال لإزالة الميثيل البرتقالي من المحاليل المائية. أصبح التخلص من الأصباغ في البيئة، بما في ذلك الميثيل البرتقالي مصدر قلق عالمي بسبب تأثيره السلبي على موارد المياه. بحثت الدراسة في تأثير العوامل المختلفة مثل التركيز ووقت التلامس ودرجة الحموضة وجرعة الممتزات وسرعة التحريض على عملية الامتزاز في التجارب على دفعات عند درجة حرارة 25°م لكل التجارب وتقييم كفاءة الامتزاز، والخواص الحركية، ومتساويات التوازن المرتبطة بإزالة الميثيل البرتقالي. أظهرت النتائج أن فحم البرتقال المنشط كيميائيا بحامض الهيدروكلوريك HCL كان مادة مازة فعالة لإزالة الميثيل البرتقالي، وقد تم تحديد قدرة الامتصاص للممتز باستخدام متساوي درجة حرارة Freundlich. خلصت الدراسة إلى أن نموذج الدرجة الثانية الزائف كان مناسبًا جيدًا للبيانات التجريبية. باختصار تشير هذه الدراسة إلى أن فحم البرتقال المنشط كيميائيا بواسطة حامض الهيدروكلوريك HCL يمكن أن يكون مادة مفيدة وفعالة من حيث التكلفة لإزالة الميثيل البرتقالي من المحاليل المائية.

الكلمات الدالة: الميثيل البرتقالي، فحم البرتقال، أيزوثيرمات الإمتزاز، حركية الإمتزاز، الإمتزاز، التنشيط الكيميائي.

1. Introduction

Dyes pose environmental hazards by resisting biodegradation and persisting in the environment due to their high thermal and photo stability (Mehra et al., 2021). Dyes, as specialty chemicals, are extensively used in industries including textiles, food, pharmaceuticals, leather, and paper, serving various purposes (Aruna Janani et al., 2023). International environmental issues related to the textile industry are specially related to water pollutants caused by the discharge of untreated effluent, as well as the ones because of the use of doubtlessly poisonous substances, in particular at some point of processing (Rovira and Doming, 2019). Textile effluent is the cause of an exquisite deal of environmental degradation and human ailment (Parvin et al., 2020). Dyes are categorized based on their uses or chemical compositions. Globally, more than 10,000 distinct synthetic dyes and pigments are manufactured annually, amounting to 7×10^5 Tons. 200,000 tons of colors used in the textile sector are released into effluents annually. Unfortunately, because of their remarkable stability against a variety of impacts, these dyes end up in typical wastewater and get trapped in the environment. Dyes' intricate structures enhance their biodegradation resistance (Hosny et al., 2023). Dyes are generally classified into anionic (direct, acid, and reactive dyes), cationic (basic dyes), and non-ionic (disperse and vat dyes) categories (Zheng et al., 2015). Azo dyes specifically fall under the anionic category (Salleh et al., 2011). Azo dyes hold the position of being the most extensively employed dyes, making up more than 60% of the overall dye market. Roughly 70% of the dyes utilized in various industries are categorized as azo dyes (Benkhaya et al., 2020). The distinctive feature of these dyes is the existence of one or more azo groups (-"N"="N"-) attached to aromatic rings. Azo dyes are composed of a nitrogen-nitrogen double bond (N=N), but they can have various structures. Monoazo dyes consist of a single N=N double bond, whereas diazo and triazo dyes have two and three N=N double bonds, respectively. Azo groups are commonly found attached to benzene and

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naphthalene rings. However, they can also form bonds with aromatic heterocycles or enolizable aliphatic groups. These additional side groups are essential in defining the color of the dye and contribute to a diverse spectrum of shades and intensities that can be achieved (Al-Rubaie and Mhessn, 2012). Azo dyes, pose a threat to human and aquatic life when they are discharged into the environment through textile effluents. Their tenacity and release into the environment are causing worry on a worldwide scale (Ajaz et al., 2020). Azo dyes pose challenges for biodegradation due to their intricate structure and synthetic origin (Popli and Patel, 2015). Among these dyes, methyl orange is particularly notable for its high stability and widespread use in the textile industry, making it resistant to biodegradation (Sha et al., 2016). A variety of treatment methods, including physical, biological, and chemical techniques, have been utilized to eliminate dyes from wastewater. These techniques comprise various strategies including biological degradation, photochemical degradation, chemical oxidation, coagulation, reverse osmosis, flotation, and adsorption. Activated carbon adsorption is a frequently employed method among these approaches due to its notable efficacy in the separation of diverse chemical compounds. This choice is favored for its uncomplicated design, economic viability, and exceptional efficiency, which has been considered by many researchers (Chen et al., 2010; and Moradi et al., 2022). So, this study focused on utilizing Orange Charcoal as an adsorbent to remove methyl orange (MO) from simulated wastewater.

2. Materials and Methods

2.1 Materials

2.1.1. Chemicals:

All experiments utilized analytical-grade chemicals obtained from Sigma-Aldrich (USA) without requiring any additional purification. Double-distilled water was used for all experiments. The following chemicals were used:

- Methyl orange (purity: >99%)
- Hydrochloric acid (HCl) solution (concentration: 0.1 M)
- Sodium hydroxide (NaOH) solution (concentration: 0.1 M)

2.2. Preparation of OCAC

To acquire orange charcoal, we can obtain it from local markets in Libya, ensuring that it is made from orange wood to contain the necessary carbon material. Before use, it is important to eliminate any impurities or foreign particles present in the charcoal by sifting or sieving it manually. This process significantly enhances the quality of the final activated carbon.

The chemical activation of the orange charcoal involves the following steps:

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a. Impregnation: A specific amount of orange charcoal is mixed with a HCl (2%) solution at a weight ratio of 1:3. The charcoal is then thoroughly soaked in the HCl solution for a defined period, (24 hrs), to ensure proper impregnation and activation.

b. Drying: After the impregnation, the charcoal is drained and dried at an appropriate temperature, 80°C, until it reaches a constant weight. This drying phase eliminates excess moisture and prepares the charcoal for the activation process.

c. Activation: The dried and impregnated orange charcoal is subjected to a high-temperature activation process. The activation temperature and duration are determined through preliminary experiments. This process typically involves heating the impregnated charcoal in a controlled environment, such as an oven, under specific conditions like heating rate and final temperature for a duration of 2 hrs. These conditions induce both chemical and physical changes, ultimately resulting in the formation of chemically activated carbon.

d. Washing and Drying: Following the activation process, the chemically activated carbon derived from orange charcoal undergoes thorough washing with distilled water to eliminate any remaining activating agent, such as HCl. The washed activated carbon is then dried at an appropriate temperature and stored in a desiccator to prevent moisture absorption.

2.3. Adsorption Experiments

To prepare a stock solution of methyl orange (MO) with a concentration of 1000 mg/L, dissolve 1 g of the dye powder in distilled water. Then, to obtain working solutions with desired concentrations (2 mg/L, 4 mg/L, 8 mg/L, 12 mg/L, 16 mg/L, and 20 mg/L), dilute the stock solution accordingly.

In this study, several equations are commonly used to describe and analyze adsorption processes. Here are some of the equations frequently employed in adsorption:

$$q_e = \frac{(C_O - C_e)}{m_s} \times V \qquad \qquad \text{...... (1)}$$

$$R(\%) = \frac{(C_O - C_e)}{C_O} \times 100 \qquad \qquad \text{...... (2)}$$

where C_o and C_e are the initial and equilibrium concentrations of MO in mg/L, respectively. q_e represents the equilibrium adsorption of MO in mg/g, while V and m_s represent the volume of the solution in liters and the mass of the adsorbent in grams, respectively.

2.4. Kinetics of the adsorption process

In the adsorption kinetic studies, 0.6 g of orange charcoal activated carbon (OCAC) with a particle size of 100 mm was added to a 50 mL solution of MO dye with an initial concentration of 4 mg/L. The experiments were conducted at optimal pH 10, with an agitation speed of 200 rpm at ambient temperature (25 °C). Samples were taken from the solution at specific time intervals (1, 2, 3, 5, 7, 10, 30, & 60 min), filtered, and analyzed.

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The adsorption kinetics focused on the rate of MO adsorption, the time required for complete adsorption, and the mechanism of the reaction. The kinetics behavior was determined using Ho and McKay's pseudo-second-order model and Lagergren's pseudo-first-order model. Then, calculate the rate constants or reaction rate coefficients associated with the adsorption process. By examining the kinetics of adsorption, we gain valuable insights into the adsorption mechanism, identify the rate-controlling step, and assess the efficiency of the adsorbent in removing methyl orange.

2.4.1. Utilizing the Lagergren pseudo-first-order model for studying dye adsorption:

The Pseudo-first-order Model can be represented in its integral form as follows:

$$\log(q_{e-}q_t) = \log q_e \times k_1 \times 2.303t \qquad \ldots\ldots$$

(3)

In order to determine the adsorption capacity, q_t and q_e, at different times t and at equilibrium, respectively (measured in mg/g), the rate constant k_1 for pseudo-first-order adsorption (measured in min^{-1}) and the contact time t (measured in min). By plotting the logarithm of (q_e - q_t) against t, then obtain a linear relationship. The slope and intercept of this plot allow us to determine the values of k_1 and the predicted q_e, respectively. (McBride ,1997)

2.4.2 Utilizing the Lagergren pseudo-second-order model for studying dye adsorption:

The adsorption kinetics can be described by the pseudo-second-order model, as mentioned by Elmorsi (2011). To simplify and linearize the model, it can be expressed as follows:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \qquad \ldots\ldots (4)$$

In Eqn. (4), k_2 represents the second-order rate constant of adsorption, measured in grams per milligram per minute (g/(mg.min^{-1})). The second-order rate constant is utilized to determine the initial sorption rate, which can be calculated using the following equation:

$$h = k_2 q_e^2 \qquad \ldots\ldots (5)$$

When plotting t/q_t against t, a linear relationship is observed. The values of k_2 and the equilibrium adsorption capacity (q_e) can be calculated by analyzing the intercept and slope of the plot according to Eqn. (4).

2.5. Applying adsorption isotherm models to investigate dye adsorption

The adsorption isotherm is crucial in understanding how the adsorbent and the adsorbate interact and provides insights into the adsorption capacity. It plays a significant role in comprehending the adsorption mechanism. The surface phase can be regarded as either a monolayer or a multilayer. Various isotherm models have been proposed in the literature, but the Langmuir and Freundlich models are the most commonly employed ones for describing the adsorption isotherm (McBride, 1997).

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2.5.1. Utilizing the Langmuir adsorption isotherm model for studying dye adsorption:

The Langmuir adsorption isotherm model suggests that adsorption occurs at specific, uniform sites within the adsorbent material. This model has been widely applied to describe monolayer adsorption processes and has demonstrated success in doing so. The linearized version of the Langmuir isotherm can be represented as follows, according to (McBride, 1997).

$$\frac{C_e}{q_e} = \frac{C_e}{q_m} + \frac{1}{k_a q_m}$$ ...... (6)

Where, k_a is the equilibrium concentration of MO in solution (mg/L), q_e is the amount of MO adsorbed at equilibrium time (mg/g), q_m is maximum adsorption capacity (mg/g) and is isotherm constants for Langmuir (L/mg). The slop and intercept of plot between C_e/q_e vs. C_e will give q_m and k_a respectively

2.5.2. Utilizing the Freundlich adsorption isotherm model for investigating dye adsorption:

The Freundlich adsorption isotherm model proposes that the adsorption surface is heterogeneous, containing sites with varying energies of adsorption and availability for adsorption. The linearized form of the Freundlich isotherm can be expressed as shown in Eqn. (7) according to Ayawei et al. (2015):

$$\ln q_e = \ln k_f + \frac{1}{n}(\ln C_e)$$ ...... (7)

In Eqn. (7), q_e represents the amount of adsorbate (in this case, MO) adsorbed at equilibrium time, measured in milligrams per gram (mg/g). C_e is the equilibrium concentration of the dye in the solution, measured in milligrams per liter (mg/L). K_f represents the adsorbent's capacity, and n is the intensity of the adsorption constant for the Freundlich model. To determine the values of K_f and n, a plot of $\ln(q_e)$ versus $\ln(C_e)$ is created. The intercept and slope of the plot provide the values of K_f and n, respectively. Additionally, the linear regression correlation coefficient R^2 can be used to evaluate the goodness of fit of the model, indicating the level of agreement between the experimental data and the model predictions.

3. Results and Discussions

The calibration curve, as depicted in Figure (1), represents the relationship between the concentration of the standard solutions of methyl orange (MO) and the corresponding remaining concentration of MO obtained in each adsorption experiment. The purpose of this calibration curve is to establish a quantitative relationship between the initial concentration of MO and the amount that remains after it undergoes adsorption onto a specific material or surface. Eqn. (8) represents the regression equation used in this context. The coefficient of determination (R^2) associated with this equation is 0.997, indicating a strong level of linearity. This high R^2 value suggests that the equation provides a good fit to the data points on the calibration curve. Therefore, it is statistically acceptable to utilize this equation for accurately calculating the remaining concentration of MO.

A=0.0871 C_e -0.0121 ...... (8)

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Where A is the absorbance of each MO solution, and C_e is MO concentration in mg/L
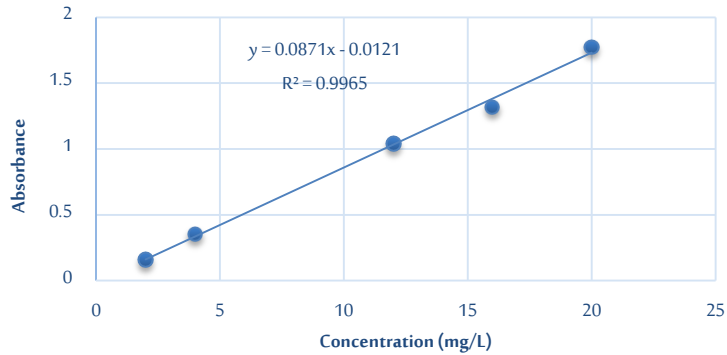

Figure1. Calibration curve

3.2. Assessing the adsorption properties of activated carbon

3.2.1. Experiments investigating the process of adsorption

The pH of the solution is a crucial factor that significantly affects the adsorption of dyes as it controls the interactions between the adsorbent and the adsorbate, as mentioned by Baloo et al. (2021). Therefore, it is important to optimize and determine the ideal pH conditions for effective dye adsorption. In Figure 2(a), the removal efficiencies of the MO dye on OCAC are shown at varying pH values ranging from 4 to 12. It is evident from the graphs that the highest removal efficiency of MO was achieved at pH 10, reaching 88%. However, as the pH increased, the removal efficiency decreased significantly from 88% to 77%. This phenomenon can be attributed to the transfer and removal of protons from the functional groups on the outer part of the adsorbent under different pH conditions. The presence of functional groups allows for interactions with the dye molecules. Additionally, the anionic or cationic nature of the target dye can also influence its adsorption behavior at different pH levels. The optimum pH condition of 10 for MO dye was determined in this study and was subsequently used in further experimental investigations. Figure 2(b) demonstrates the effect of varying initial concentration. The effect of initial concentration on the adsorption of methyl orange (MO) on orange charcoal activated carbon was studied using a concentration range of 2, 4, 8, 12, 16, and 20 mg/L. Among these concentrations, it was observed that the highest removal percentage of 76% was achieved at an initial concentration of 4 mg/L. At lower initial concentrations, such as 2 mg/L, the adsorption capacity of the orange charcoal activated carbon may not be fully utilized. This means that there are available surface sites on the charcoal that are not completely occupied, resulting in a lower removal percentage. At an initial concentration of 4 mg/L, the highest removal percentage of 76% was achieved. This indicates that the adsorption sites on the activated carbon were effectively utilized, and a significant amount of methyl orange

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molecules were adsorbed onto the surface. The concentration gradient between the solution and the activated carbon favored the adsorption process, leading to a higher removal percentage. As the initial concentration increased further (8, 12, 16, and 20 mg/L), the adsorption capacity of the charcoal became saturated. This means that the available surface sites became occupied, and the rate of adsorption gradually decreased. Consequently, the removal percentage of the dye also decreased as the concentration increased. The optimum initial concentration condition of 4 mg/L for MO dye was determined in this study and was subsequently used in further experimental investigations. Another important parameter for dye removal is the mass of the adsorbent. Figure 2(c) demonstrates the effect of varying adsorbent mass (ranging from 0.1g to 1g per 50 mL) on the removal efficiencies of the MO dye on OCAC. Increasing the adsorbent mass from 0.1g to 0.5g leads to a notable improvement in removal efficiency. This can be attributed to the enhanced surface area and increased availability of adsorption sites on the adsorbent, as previously observed. Zhao et al. (2022). The equilibrium and maximum adsorption efficiency were obtained at 0.6g of adsorbent, and thus, this condition was considered as the optimum adsorbent mass for subsequent studies. Additionally, at this optimum mass, a remarkable removal percentage of 94% was achieved. The equilibrium time is another significant parameter in the adsorption of MO dye. In Figure 2(d), the graph illustrates the correlation between the equilibrium adsorption capacity (q_e) of the MO dye and the contact time. It demonstrates how the quantity of MO dye adsorbed onto the adsorbent (OCAC) alters as the contact time progresses. As the contact time increases, the adsorption capacity eventually stabilizes at a value of 0.140 mg/g, indicating the attainment of equilibrium. It shows that the adsorption efficiency increased with increasing contact time and reached equilibrium after 30 minutes. The rapid initial adsorption is attributed to the availability of more pores on the OCAC structure, enabling quicker mass transfer of the dye from the solution onto the adsorbent. This indicates good accessibility to the binding sites for the dye on the adsorbent, which is advantageous in practical applications as it reduces residence time (Hashemian et al., 2013; and Zhao et al., 2022). The optimum contact time condition was determined to be 30 minutes. In summary, the pH, initial concentration, adsorbent mass and contact time are significant parameters in the adsorption of MO dye onto OCAC. Optimizing these parameters can enhance the removal efficiency of the dye and improve the adsorption process.

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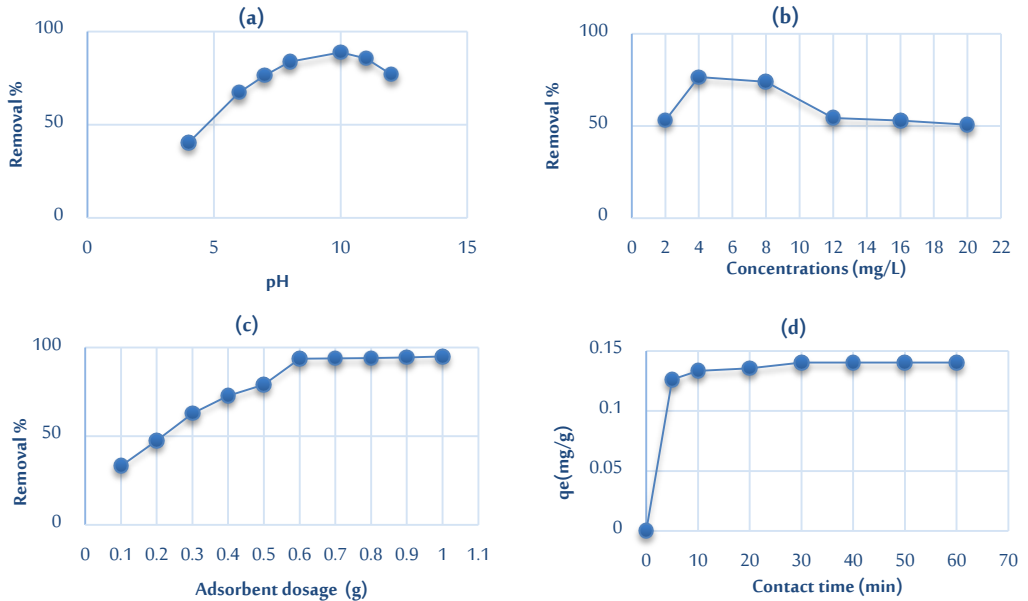
Figure 2. Multi-Parameter Plot: a) Influence of pH, b) Influence of initial concentrations, c) Influence of adsorbent dosage, and d) Influence of Contact Time; on Adsorption Process.

3.2.2. Kinetics Study:

The study aimed to gain a deeper understanding of the adsorption kinetics of methyl orange (MO) dye onto orange charcoal activated carbon(OCAC). Various adsorption kinetic models were utilized to analyze the adsorptive behavior of the process. These models include the pseudo-first-order and pseudo-second-order, including the equations used to analyze the kinetics and diffusion of MO adsorption onto OCAC. The experimental procedures for the kinetics models are illustrated in Figure (3).
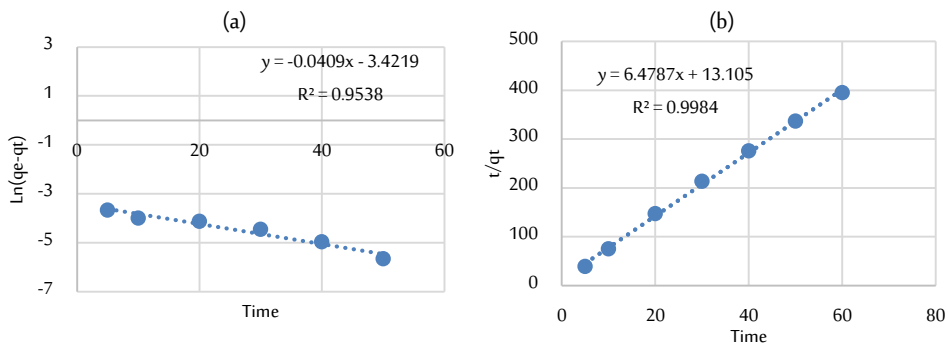

Figure 3. a) Pseudo 1st order Kinetic study and *b)* Pseudo 2nd order Kinetic study;

at (dosage of 0.6 g, initial concentration of 4 mg/L, and pH of 10).

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3.2.3. Adsorption isotherms:

To investigate the isotherm of methyl orange using the Freundlich and Langmuir models, we prepared different concentrations of methyl orange (2, 4, 8, 12, 16, and 20 mg/L) by diluting a stock solution. Each solution had a volume of 50 mL. In the adsorption experiments, we used identical containers and added 50 mL of a specific methyl orange solution to each container. We introduced a fixed amount of absorbent material weighing 0.6 g into each container and thoroughly mixed the solution and absorbent material. The mixtures were allowed to reach equilibrium for 30 minutes under controlled conditions, including constant temperature and agitation. After the equilibration period, we measured the equilibrium concentration of methyl orange in each solution using analytical techniques UV-Vis spectrophotometry. To calculate the amount of methyl orange adsorbed, we subtracted the equilibrium concentration from the initial concentration for each solution. The adsorbed amount (mg/g) was determined using Eqn. (1). To analyze the adsorption behavior, we plotted the amount of methyl orange adsorbed (mg/g) on the y-axis against the equilibrium concentration of methyl orange (mg/L) on the x-axis for each concentration. We then applied the Freundlich and Langmuir isotherm models to the experimental data. Figs. (4 & 5) shows the linear plot of, Langmuir and Freundlich isotherms respectively, the R-squared correlation coefficient (R^2) is a statistical measure that indicates the goodness of fit of a model to the observed data. It represents the proportion of the variation in the dependent variable (in this case, the adsorption data) that can be explained by the independent variable (the Langmuir or Freundlich model). In this study, the R^2 correlation coefficient for the Langmuir model was 0.8818, while the R^2 correlation coefficient for the Freundlich model was 0.9235. An R^2 value of 0.8818 for the Langmuir model suggests that approximately 88.18% of the variation in the adsorption data can be explained by the Langmuir model. Similarly, an R^2 value of 0.9235 for the Freundlich model indicates that approximately 92.35% of the variation in the adsorption data can be explained by the Freundlich model. Based on these R^2 values, both the Langmuir and Freundlich models provide reasonably good fits to the adsorption data. However, the Freundlich model, with a higher R^2 value of 0.9235, demonstrates a slightly better fit to the data compared to the Langmuir model with R^2 value of 0.8818. This suggests that the Freundlich model may provide a more accurate description of the adsorption behavior of methyl orange onto orange charcoal activated carbon in this particular case.
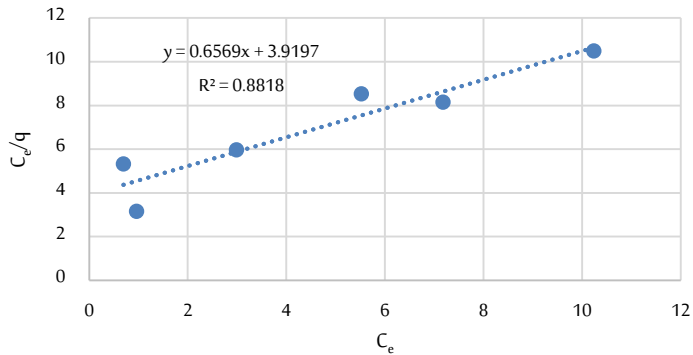
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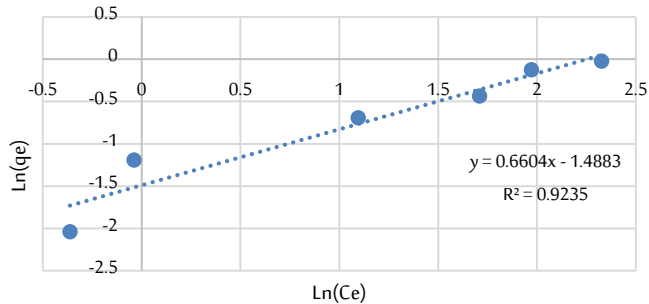**Figure 4. Shows the linear plot of Langmuir isotherm**

Figure 5. Shows the linear plot of Freundlich isotherm.

4. Conclusion

The findings of this study indicate that activated carbon could be a promising and effective option for eliminating methyl orange from wastewater. The study determined that the optimal conditions for adsorption, including pH, dosage of adsorbent, contact time, and initial concentration, were pH 10, 0.6 g of adsorbent per 50 ml of solution, 30 minutes of contact time, and an initial concentration of 4 mg/L. It's worth noting, however, that the optimal conditions for adsorption may differ based on the particular adsorbent and the characteristics of the pollutant being targeted. The study found that the Freundlich isotherm and the pseudo-second order model were both appropriate for describing the adsorption behavior of methyl orange onto activated carbon under the given conditions. These findings could help to develop effective and sustainable methods for treating wastewater, which is crucial for environmental protection and public health.

5. Future Work

I intend to conduct a review paper on the use of orange charcoal chemically activated with hydrochloric acid to remove multiple pollutants, as it achieved high results for removing methyl orange in the current study. It

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also achieved previous high results with copper (II) and methylene blue dye, with another research groups and these researches is under publication.

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