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Research Article

Assessment of the Efficiency of Crushed Ceramics in Adsorbing Methyl Orange Dye from Aqueous Solutions

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Abstract

This study evaluates the efficiency of crushed ceramic as an adsorbent for the removal of methyl orange dye from aqueous solutions. Various parameters influencing the adsorption process were examined, including pH, contact time, adsorbent dosage, initial dye concentration, and temperature. The adsorption process was optimized at a pH of 2.0, achieving a maximum removal efficiency of 99.31%. Adsorption equilibrium was reached after 90 minutes, with the adsorption capacity increasing with higher initial dye concentrations. The adsorption isotherms were analyzed using Langmuir and Freundlich models, with the Freundlich model providing a better fit for the equilibrium data (N = 2.724). Kinetic studies indicated that the adsorption followed a pseudo-second-order model, suggesting that the rate-limiting step involves chemisorption. The Langmuir maximum adsorption capacity (Q_{max}) was determined to be 22.31 mg/g, while the Freundlich constant (KF) was 2.026. The results demonstrate that crushed ceramics are a promising low-cost adsorbent for the efficient removal of methyl orange from contaminated water, offering potential applications in wastewater treatment. The study also underscores the importance of utilizing appropriate kinetic and isotherm models for accurately predicting adsorption behavior and optimizing operational conditions.

Keywords: Methyl Orange Dye, Isotherm Models, Adsorption, Crushed Ceramics, Kinetic Studies

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1. Introduction

Good health can't be assured because of pollution everywhere. Pollution happens when harmful substances contaminate the air, water, and soil, which disrupts natural processes and harms the environment [1]. Environmental pollution has been a major problem for the world and has caused a decrease in the population [2]. Most pollution comes from human activities and can be classified into six types: air, land, noise, thermal, visual, and water pollution. Water is a key natural resource that helps manage global ecosystems by connecting the earth's land, air, and living things. It moves substances between these parts and supports important chemical reactions [3]. Water is crucial for both nature and human progress, but pollution has made it a serious threat to the health of the environment [4].

Water pollution is mainly caused by industries that often ignore the environment when they release their waste into water bodies. Industries that produce dyes, like those in textiles, paper, and plastics, use large amounts of water and create a lot of colored wastewater. Even small amounts of dye in the water make it unappealing. Dyes can be toxic, cancer-causing, and dangerous to living organisms [5]. Living organisms need water for their biochemical processes, so it's important to remove dyes from wastewater before it is released into water bodies. Environmental issues often occur because of poor management of wastewater. The dye manufacturing industry, textile industry, and fabric users are major contributors to the discharge of dye-laden wastewater into water sources [6]. It's estimated that the dye manufacturing industry releases about 2% of the dye it produces each year in its wastewater. In the textile industry, 10-15% of the dye is lost during the dyeing process and ends up in the wastewater. Dyes are also used in other industries, such as those making laboratory equipment, paper, plastic, fertilizer, detergent, leather, cosmetics, and pharmaceuticals [7]. There are over 10,000 dyes available for commercial use, and many of them have harmful effects on living organisms [7]. Synthetic dyes are more stable and harder to break down because of their complex structures. When living organisms are exposed to these dyes, they can suffer from health problems because the dyes are toxic and can cause cancer [8]. Organic dyes are also dangerous and can harm aquatic life and disrupt the food chain. To remove dyes from water, various methods and technologies are used, including physical, biological, and chemical approaches [9]. However, some of these methods may not be practical due to high costs, inability to meet strict regulations, or the creation of hazardous byproducts that are hard to treat [9]. Among all these methods, adsorption is the most effective and non-destructive technique for removing dyes from water. It is popular because it is cost-effective, simple to design and operate, and works well even with toxic substances. Adsorption is widely used in industry for separating and purifying wastewater [10]. Activated carbon is a popular adsorbent for dye removal because it has a high capacity for adsorption. However, its use is limited due to its high cost and a 10-15% loss of effectiveness during regeneration [10]. Researchers are currently exploring alternative adsorbents for dye removal. The key requirements for these alternatives are reusability, low operating cost, better selectivity for specific dyes, effective removal of dyes regardless of their toxicity, short operation time, and no creation of harmful byproducts. Some materials being studied for dye removal include various biosorbents like mango peel, sugarcane bagasse, corn cob, and coconut shell, as well as materials like clay and silica [11].

To protect health and meet strict environmental regulations, it is crucial to treat wastewater contaminated with heavy metals and dyes before its release into the environment. This research aims to assess the effectiveness of crushed ceramics as an adsorbent for removing methyl orange dye from water and to determine the optimal conditions for this process. The study involves preparing and characterizing the adsorbent made from broken ceramics, analyzing the impact of factors such as pH, contact time, adsorbent dosage, and initial dye concentration on dye removal efficiency, and evaluating adsorption equilibrium using Langmuir, Freundlich, and Brunauer-Emmett-Teller (BET) isotherm models. Additionally, the research investigates the kinetics of dye removal by fitting the data to pseudo-first-order, pseudo-second-order, and intra-particle diffusion models.

2. Methods Adsorbent Preparation

Ceramics were purchased from Oja-Oba market in Akure, Nigeria, then crushed using a grinding stone and sun-dried for two days. After drying, the crushed ceramics were sieved through a 63μm laboratory test sieve to achieve a uniform particle size and stored in an airtight container until use.

2.1. Preparation of Reagents

2.1.1. Preparation of Methyl Orange Dye Stock Solution

A 100 mg/L methyl orange dye solution was prepared by adding 100 mL of a standard methyl orange dye stock solution to a 1000 mL flask and then filling the flask with distilled water up to the mark [13].

2.1.2. Preparation of Working Standard Solutions for UV-visible Spectrophotometer

Ten working solutions were prepared from the stock solution by using the dilution formula $C_1V_1 = C_2V_2$ where C_1 is the concentration of the standard solution (100 mg/L), V_1 is the required volume of the standard, C_2 is the required working standard concentrations (10 mg/L to 100 mg/L) and V_2 is 10 mL.

2.2. Determination of the Effect of pH

To test the effect of pH on the adsorption process, 1.0 g of the adsorbent was placed into seven pre-labeled beakers, and 25 mL of a 100 mg/L methyl orange dye solution was added to each beaker. The pH of the mixtures was then adjusted to 1.0, 2.0, 3.0, 4.5, 6.0, 7.0, and 8.0 in the seven beakers using

0.1M HCl and 0.1M NaOH solutions. The samples were agitated on an orbital shaker at 100 rpm for 15 minutes. After shaking, the samples were centrifuged, and the dye solution was separated from the adsorbent by decantation. The absorbance of the supernatant solution was measured using a UV-VIS Spectrophotometer at a wavelength of 464 nm [14].

2.3. Effect of Contact Time

The beakers were labeled, and 1.0 g of the adsorbent was placed into each of the 10 beakers. Then, 25 mL of a 100 mg/L methyl orange dye solution was added to each beaker. Each sample was adjusted to the optimum pH and shaken on an orbital shaker at various time intervals: 2, 5, 10, 15, 20, 25, 30, 60, 90, and 120 minutes. At each interval, a sample was withdrawn, centrifuged, and the dye solution was separated from the adsorbent by decantation. The absorbance of the supernatant solution was measured using a UV-VIS Spectrophotometer at a wavelength of 464 nm [15].

2.4. Effect of Adsorbent Dosage

The beakers were labeled, and different amounts of adsorbent (0.1, 0.5, 1.0, 1.5, 2.0, and 2.5 g) were placed into them. Then, 25 mL of a 100 mg/L methyl orange dye solution was added to each beaker. Each sample was adjusted to the optimum pH and agitated. After the optimum contact time, the samples were removed from the shaker, centrifuged, and the dye solution was separated from the adsorbent by decantation. The absorbance of the supernatant solution was measured using a UV-VIS Spectrophotometer at a wavelength of 464 nm [15].

2.5. Effect of the Initial Concentration

1.0 g of the crushed ceramic adsorbent was placed into labeled beakers. Then, 20 mL of dye solutions with different initial concentrations (50, 100, 150, 200, 250, and 300 mg/L) were pipetted into the beakers. The samples were adjusted to the optimum pH and agitated. After the optimum contact time, the samples were removed from the shaker, centrifuged, and the dye solution was separated from the adsorbent by decantation. The absorbance of the supernatant solution was measured using a UV-VIS Spectrophotometer at a wavelength of 464 nm [15].

2.6. Effect of Temperature

1.0 g of crushed ceramic adsorbent was placed into labeled beakers. Then, 20 mL of a 50 mg/L methyl orange dye solution was added to each beaker at different temperatures (room temperature, 35°C, 45°C, 55°C, and 65°C). The samples were kept at a constant pH and shaken in a water bath shaker for the same contact time. After shaking, the supernatant was collected by centrifugation and decantation, and its absorbance was measured using a UV-VIS Spectrophotometer at a wavelength of 464 nm. This procedure was repeated for dye solutions with concentrations of 100, 150, 200, 250, and 300 mg/L to analyze the kinetic characteristics of the adsorption.

2.7. Statistical Analysis

All experiments were conducted in duplicate, and the data were presented as mean values ± standard deviation of the duplicate measurements. The results were also illustrated using graphs and curves created with Microsoft Excel 2016.

3. Results and Discussion

Figures **1** and **2** display the FT-IR spectra of the adsorbent before and after adsorption. Both spectra show similar peaks, though the second spectrum has a slight shift to the left. The peak at 3649.1 cm^{-1} in the pre-adsorption spectrum is attributed to the stretching of the O-H group, which results from intermolecular and intramolecular hydrogen bonding in polymeric compounds like alcohols, pectin, hemicelluloses, cellulose, and lignin, as supported by Meda et al., 2022 [16]. The peak around 2940.9 cm^{-1} in the post-adsorption spectrum corresponds to the stretching vibrations of the C-H bonds in methyl (CH_3), methylene ($=CH_2$), and methoxy (OCH_3) groups. The intense peak at 1051.1 cm^{-1} is due to the C-O stretching in alcohols or carboxylic acids. Additionally, the peak at 775 cm^{-1} is related to C-H stretching in the aromatic ring.

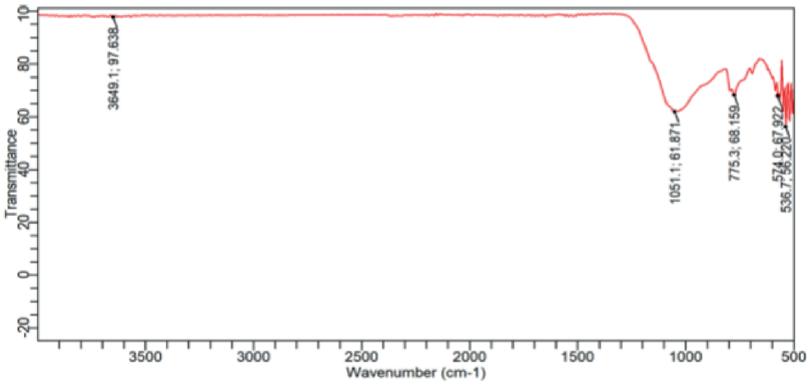

Figure 1: FTIR spectra of adsorbent before adsorption.

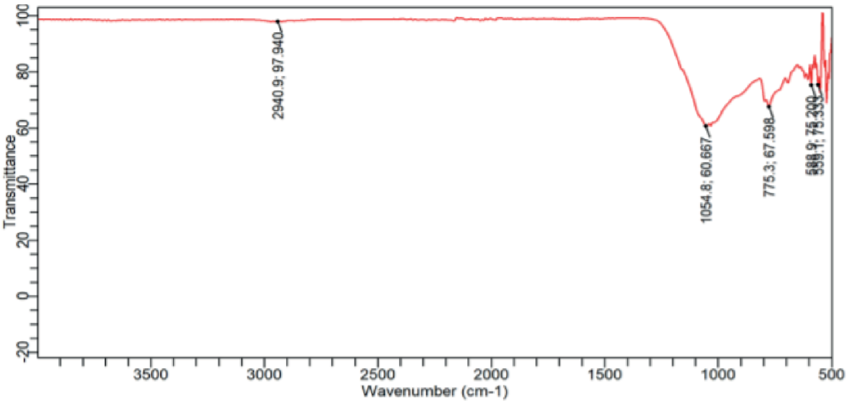

Figure 2: FTIR spectra of adsorbent after adsorption.

3.1. Effect of pH on the Removal Efficiency of Methyl Orange

The effect of pH on the removal of methyl orange in a solution by crushed ceramics adsorbent was studied at a pH of 2 to 8. Figure **3** shows the removal efficiency (%) with their corresponding pH values. The removal efficiency (%) of methyl orange by the adsorbent was generally higher at the lower pH of 1 (98.29 %) and 2 (99.31 %) compared to the other pH. This may be because at lower pH, the surface of the adsorbent becomes more protonated, having more H^+ from the 0.1M HCl and other positive ions that may be there as impurities. This in turn makes methyl orange dye, which is an anionic dye, bind with the surface of the adsorbent easily [17]. With the increase in pH, the hydroxyl ion (OH^-) from the 0.1M NaOH competes with the anionic dye for sorption sites, thereby reducing its adsorption efficiency [18]. The optimum pH for the rest of the experiments was thus selected as pH 2 with a removal efficiency of 99.31% and was chosen as one of the parameters in the next optimization factors.

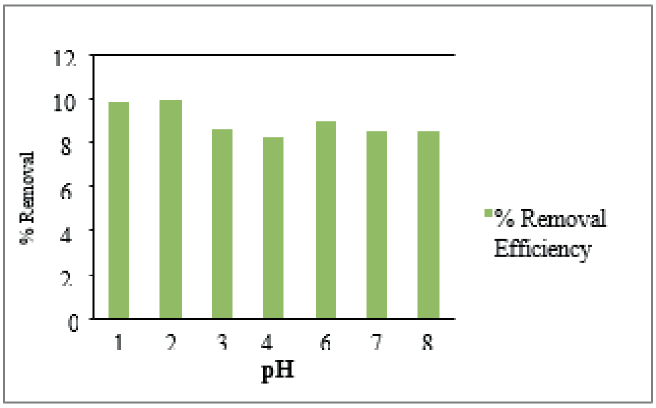

Figure 3: Effect of pH on removal of methyl orange with crushed ceramics. Initial concentration = 100mg/L, Agitation speed = 100rpm, Contact time = 2hours, Adsorbent dosage = 1.0g.

3.2. Effect of Contact Time on the Removal Efficiency of Methyl Orange

Contact time plays a key role in the adsorption process and it often determines the amount of dyes adsorbed on an adsorbent. It is an important parameter for successful use of the adsorbents for practical applications. Rapid adsorption is among the desirable parameters in the choice of a suitable adsorbent. The effect of contact time on the adsorption of methyl orange dye was investigated and the results are displayed in Figure **4**. The rate of sorption of methyl orange dye generally increases with increasing the contact time. At 90 minutes, the equilibrium is achieved as a result of the binding sites that became exhausted. The percentage removal gradually slowed down due to decreasing availability of active sites by methyl orange [19].

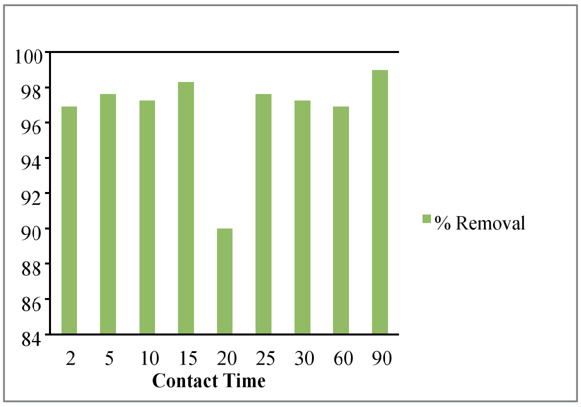

Figure 4: Effect of contact time on removal of methyl orange dye. pH = 2.0, Initial concentration = 100mg/L, Agitation speed = 100rpm, Adsorbent dosage = 1.0 g.

3.3. Effect of Adsorbent Dosages on the Removal Efficiency of Methyl Orange

The effect of adsorbent dosages ranging from 0.1g to 2.5g on the adsorption of Methyl orange is presented in Figure **5**. The adsorption of the Methyl orange dye increased from 89% to 94% with an increase in adsorbent dosage from 0.1 to 1g. At an adsorbent dosage of 2g, the percentage removal dropped which was a result of the overlapping of the adsorption sites. The results show that as the adsorbent dosage increases the affinity for methyl orange to bind with the binding sites decreased which was observed to be due to the overlapping of the negatively charged ions at the surface of the adsorbent [20]. An increase in the adsorbent dosage later increased with the percentage removal and reached its equilibrium again at 2.5g with % removal 91%, which was a result of the binding sites available having become exhausted.

3.4. Effect of Temperature on the Removal Efficiency of Methyl Orange

Figure **7** shows the effect of temperature on the adsorption efficiency. Starting from the room temperature, it was observed that with an increase in the temperature by a 10-degree interval, the percentage removal increased gradually. The increase in temperature affects the ions' kinetic energy by increasing it and giving room for the dye concentration to be attached to the adsorbent surface [21].

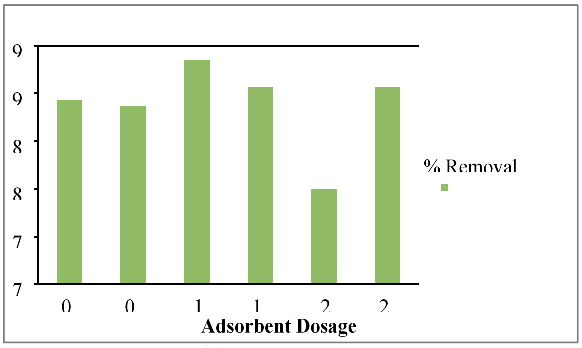

Figure 5: Effect of the adsorbent dosage on removal of methyl orange dye. pH = 2.0, Initial concentration = 100 mg/L, Agitation speed = 100rpm, Contact time = 90 Minutes.

3.5. Effect of the Initial Concentration on the Removal Efficiency

The effect of the initial concentration ranges from 50, 100, 150, 200, 250, and 300mg/L on the amount of methyl orange adsorbed is presented in Figure **6**. It was observed that with an increase in the initial Congo red concentration, the percentage removal first increased and then decreased. The percentage removal decreased because at higher concentrations the available sites on the adsorbent become limited. The adsorption capacity increased as dye concentration increased as a result of utilizing all available adsorption sites at higher concentrations [21].

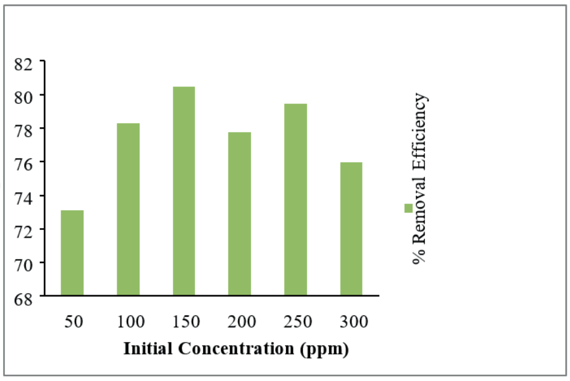

Figure 6: Effect of the initial concentration on removal of Methyl orange. pH = 2.0, Agitation speed = 100rpm, Contact time = 90 Minutes, Adsorbent dosage = 1.0gs.

3.6. Langmuir Adsorption Isotherm for the Sorption of Adsorbent Methyl Orange

The Langmuir isotherm model is a straight-line graph which is a plot of C_e/q_e against C_e. For the sorption of methyl orange onto crushed ceramic adsorbent, the Langmuir isotherm model graph is presented in Figure **8**.

Details of the Langmuir isotherm parameters of adsorbent methyl orange are presented in Tables **1** and **2**.

Details about pseudo first order kinetic parameters for methyl orange are presented in Table **4**.

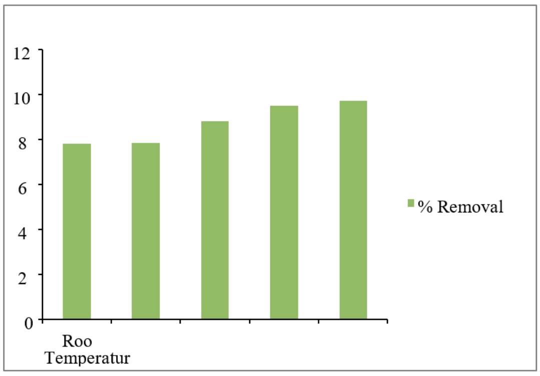

Figure 7: Effect of the Temperature on the removal of Methyl orange dyes, Eriochrome Black T, and their binary mixture using untreated pulverized elephant grass. pH = 2.0, Agitation speed = 100rpm, Contact time = 90 minutes, Initial concentration = 30mg/L, Adsorbent dosage = 1.0g.

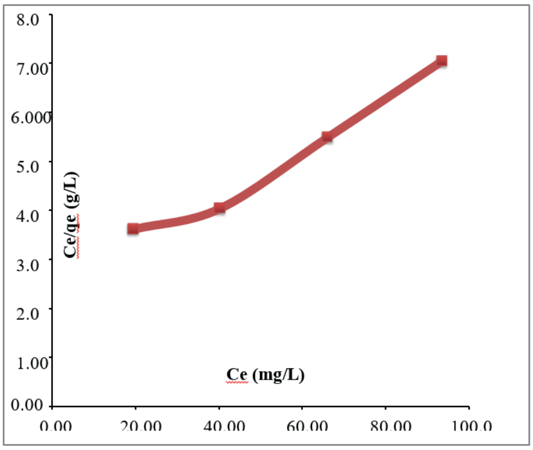

Figure 8: Langmuir Isotherm for methyl orange adsorption.

Table 1: Langmuir isotherm parameters of adsorbent methyl orange.

Methyl orange	
Qmax	**KL**
22.31	0.020

Table 2: R_L values for the adsorption of Methyl orange mixture.

Initial Concentration	R_L
50	0.49
100	0.41
150	0.35
200	0.2

3.7. Freundlich Adsorption Isotherm of Adsorbent Methyl Orange

The Freundlich isotherm assumes a heterogeneous surface with a non-uniform distribution of heat of biosorption over the surface and a multilayer biosorption can be expressed (Freundlich, 1906). The Freundlich model was expressed as:

$$\log q_e = \log K_F + 1/n \log C_e \tag{1}$$

Freundlich isotherm model is a straight line graph and a plot of log q_e against log C_e. For the sorption of methyl orange onto crushed ceramic adsorbent, the Freundlich isotherm model graph is presented in Figure **9**.

The details about Freundlich isotherm parameters of adsorbent methyl orange are presented in Table **3**.

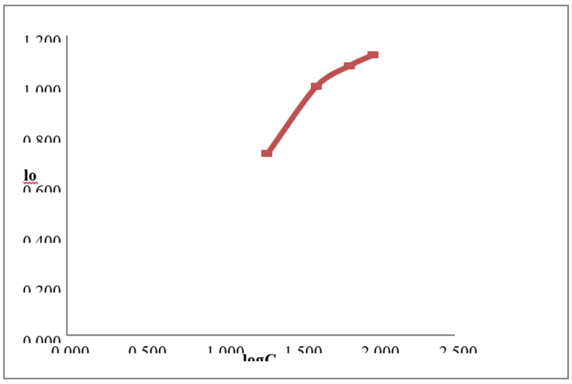

Figure 9: Freundlich isotherm plot for the sorption of methyl orange onto crushed ceramics.

Table 3: Freundlich isotherm parameters of adsorbent methyl orange.

N	KF
2.724	2.026

Table 4: Pseudo First order Kinetic Parameters for methyl orange.

$q_e$exp (mg/g)	$q_e$cal (mg/g)	K_1 (min-1)
76.87	84.92	0.0074

Results obtained show that the equilibrium data for the adsorption of methyl orange are best fitted for Freundlich isotherm because the N-value and their R^2 values are close to one. Chen *et al*. (2010), however, proposed that the values of N in the range of 2 to 10 are good, 1 to 2 values are moderate, and less than 1 are poor sorption characteristics [21]. For the sorption of methyl orange, the multi-layer is formed which is the physic-sorption type of adsorption. The absorption kinetics provides information about the mechanism of adsorption which is important for the efficiency of the adsorption process. A pseudo-first-order model of log (q_e-q_t) against t in which K_1 and q_e are calculated from the slope and intercept respectively from the plot. Figure **10** shows the plot of the pseudo-first-order model at a concentration of 100mg/L and at varied times (10, 15, 20, and 25 minutes).

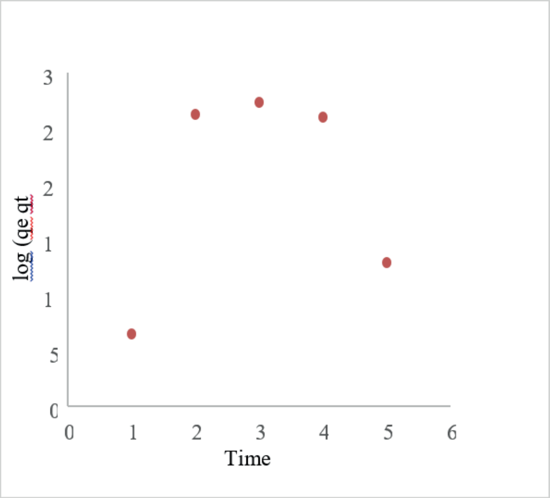

Figure 10: Plot of the pseudo-first-order model of the adsorption of methyl orange onto modified mango leaves. pH = 2.0, Initial concentration = 100mg/L, Agitation speed = 100rpm, Adsorbent dosage = 1.0g.

The pseudo-second-order kinetic rate equation was expressed according to Ho and Mckay, 1998:

$$d_{qt}/d_{qt} = k_2(q_e - q_t)^2 \tag{2}$$

$$t/q_t = 1/k_2 q_e^2 + 1/q_{et} \tag{3}$$

This is the plot of t/q_t against t in which q_e and K_2 are calculated from the slope and intercept respectively from the plot. Figure **11** shows the plot of the pseudo-second-order model at a concentration of 100mg/L and at varied times (10, 15, 20, and 25 minutes).

The constant coefficient of pseudo-second-order kinetic for the adsorption of mercury (II) ions, Congo red dye, and their binary mixture by modified mango leaves are presented in Table **5**.

By comparing Tables **4** and **5**, Pseudo Second Order kinetics was seen as the best fitted for the adsorption of methyl orange onto crushed ceramics than Pseudo First Order kinetics since the calculated equilibrium adsorption capacity value of methyl orange was close to the experimental equilibrium adsorption capacity value in Pseudo Second Order kinetics than in Pseudo First Order kinetics.

4. Conclusions

Powdered ceramics were investigated for their performance in removing methyl orange from contaminated aqueous solutions. The FTIR spectra of ceramics powder revealed that OH, C-H, C=C, and C-O stretching

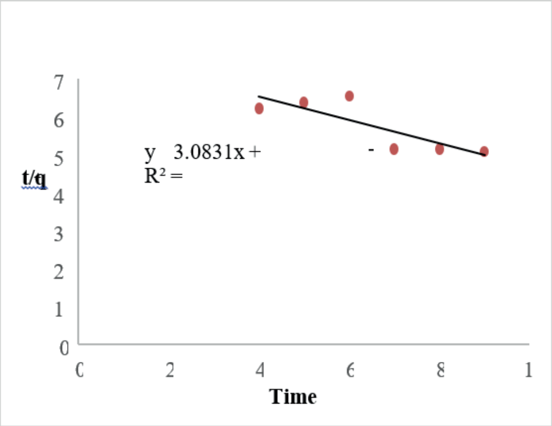

Figure 11: Plot of pseudo-second-order model of the adsorption of methyl orange on crushed ceramic pH = 2.0, Initial concentration = 100mg/L, Agitation speed = 100rpm.

Table 5: Pseudo-second-order Kinetic Parameters for the adsorption of mercury (II) ions, Congo red dye, and their binary mixture by modified mango leaves.

Methyl orange		
$q_e$exp (mg/g)	**$q_e$cal (mg/g)**	**K_2 (min-1)**
-0.0017	45.455	71.45

were responsible for the adsorption. Effects of the different experimental parameters that influenced the efficiencies of the adsorbent have been evaluated and optimized. Since wastewater from the textile and dye industries contains a lot of colored organic compounds, this work was geared towards removing methyl orange dye at once from the contaminated aqueous solution. The investigation revealed that the adsorption capacity of the powdered ceramics on removal of high enough to be comparable to observed values in the literature. Kinetic data obtained for the adsorption of methyl orange dye was fitted well with a pseudo-second-order model. To improve the adsorption capacity of the powdered ceramics on the removal of dyes from the contaminated aqueous solutions; it is recommended that; the binding sites of the powdered ceramics be increased by treating it with other chemicals that could open up pore sites for more adsorption and more studies be conducted on the mechanisms of adsorption by use of Scanning Electron Microscopy (SEM) and Energy dispersive Spectroscopy (EDS) to understand how the adsorption takes place.

Competing Interests

There are no competing interests to declare.

Ethical Approval

Not applicable

Author’s Contributions

E.O. supervised the work, H.T. wrote the manuscript, and O.A. worked on the analysis that was used to prepare the figures.

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Availability of Data and Materials

The authors declare that the data supporting the findings of this study are available within the paper. Should any raw data files be needed in another format they are available from the corresponding author upon reasonable request.

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