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# Batch adsorption of an anionic dye from aqueous solution onto acid and basic activated carbon synthesized from almond shell by chemical activation method

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**Abstract:** The aim of this study is to assess the impact of activating agents on the removal of methyl orange (an anionic dye) through the adsorption process in a batch system, utilizing acid and basic-activated carbons derived from almond shell grains. The acid and basic activated carbons (AAC and BAC) were prepared via chemical activation using phosphoric acid ($H_3PO_4$) and potassium hydroxide (KOH), respectively. Both types exhibited a high yield in adsorption capacity. The investigation into the effect of various operational parameters on adsorption capacity involved varying time, pH, temperature, and the amount of activated carbon. The results from the adsorption kinetics revealed that the equilibrium time is 40 minutes for both types of carbons. Additionally, at a temperature of 25°C, using 0.2 g of activated carbon, at pH 2 for AAC and pH 6 for BAC, the maximum adsorption efficiencies were 90% and 85%, respectively. The pseudo-first-order and pseudo-second-order kinetic models were applied to test the experimental data. The results showed that the dye uptake process onto acid-activated carbon followed the pseudo-second-order rate expression, and the uptake using basic-activated carbon followed the pseudo-first-order model. These results suggest that it is feasible to convert wastes into porous materials for the effective removal of dyes from aqueous environments.

***Keywords:*** *Activated carbon, Adsorption, Almond shell, Anionic dye, Chemical activation.*

## 1. Introduction

Water contamination is worsening as the population continues to grow and development accelerates. Numerous novel contaminants have been identified in aquatic ecosystems, including plastics, solvents, dyes, paints, persistent organic pollutants, and more[1, 2].Wastewater from a variety of industries, including food processing, cosmetics, plastics, and pharmaceuticals, was examined for synthetic dyes[3-6].Synthetic dyes are often very robust against light, oxidation, and biodegradation and have a rigid chemical structure [7, 8].They rank among the most pressing environmental issues of our day. These substances are extremely poisonous and insoluble. They are hazardous contaminants that are poisonous to aquatic life and present a risk to the environment if they are drained straight into the receiving waters[9].A pressing issue that requires attention is how to efficiently remove dyes from the water system while lowering the risk they pose to human health[10-12].

As per the allegations, the MO dye is poisonous and irritates the skin when it comes into contact with it. It may also be ingested or inhaled[13].

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There are several ways to deal with this problem and eliminate dyes in an aqueous solution. One technique that uses varying molecule sizes as a key to separation is membrane filtration. The expensive membrane and maintenance costs of this chemical-free, effective pollution removal technology are its drawbacks[14]. Ion exchange resin is an additional strategy that provides simplicity and reusability. The consumption of acids and alkalis is frequently three to four times greater than the necessary stoichiometric quantities, which is a major disadvantage in their regeneration process[15]. This causes residual ions to remain in the treated water and raises operational expenses. At the anode, oxygen gas is created while metal ions are reduced to their elemental form at the cathode in electrolytic recovery. Although this process uses a lot of energy and produces little sludge, it doesn't require any chemicals[16].

Recently, the adsorption method has emerged as an appealing alternative for decolorizing dye wastewaters, with low-cost and easily available adsorbents being used in a wide range of applications. There have been reports of renewable and cheaper adsorbent precursors derived from forestry and agricultural residues for the removal of hazardous chemicals from aqueous media[4, 17-19].

Carbon-based adsorbents, clay minerals, zeolites, siliceous materials, and polymers are among the materials that have been investigated for adsorption from water[20, 21]. Because of its wide surface area and abundance of oxygen-rich functional groups, activated carbon, a carbonaceous adsorbent, has garnered significant interest. These factors enhance its capacity to absorb pharmaceutical pollutants from aqueous solutions[22]. Its extensive use in water treatment demonstrates how well it works to eliminate contaminants and enhance water quality[23].

A variety of agricultural waste products, including bagasse pith, almond husk, coffee husk, and coconut husk, can be used to produce activated carbon. These materials' high specific surface areas and enhanced ability to absorb heavy metals result from their high porosity and wide range of pore sizes. The two primary processes used to manufacture activated carbon are chemical activation, which employs substances like KOH, $H_3PO_4$, or $ZnCl_2$ to improve pore structure and adsorption qualities, and heat activation (carbonization), which converts biomass into biochar. The structure and surface area of the activated carbon are significantly influenced by the choice of chemical activator[24, 25].

Numerous investigations have looked into the adsorption of MO dye from water. Al-Rub et al. [26] studied the adsorption properties of Methyl Orange dye (MO) onto Commercial Activated Carbon (CAC) in aqueous solution Al-Rub et al. [26]. Dehmani et al. [27]investigated the use of commercially available activated carbon to remove methyl orange from aqueous solutions[27, 28] in their work, they investigated the adsorption of methyl orange on activated coffee grounds, while Khaniabadiet al. [29] investigated the removal of aniline and methyl orange from liquid-phase aloe vera leaf wastes Khaniabadiet al. [29].Islam et al. [30],their work aims to valorize the activated carbon (AC) derived from coconut shells in order to remove the molybdenum (Mo) from an aqueous solution.

In this work, two (02) types of activated carbons (acidic and basic activated carbons) were produced from almond shells using a chemical activation process for the removal of methyl orange. The chemical agents used for this purpose are phosphoric acid and potassium hydroxide for acidic and basic chemical activation, respectively. Methyl orange, the pollutant to be removed, is an anionic dye primarily used for dyeing textiles. The objective is to determine the influence of certain operating conditions using the two types of activated carbons mentioned on the adsorption capacity of methyl orange. Therefore, the adsorption kinetics were studied by applying two kinetic models to analyze the uptake process of the experimental data points.

## 2. Materials and Methods

### *2.1. Preparation of Activated Carbon*

#### *2.1.1. Preparation of Raw Matter*

Raw activated carbon is prepared from almond shells that have been washed thoroughly with distilled water and then dried in an oven at 105°C for 24 hours. The shells are then crushed and sieved to retain only the fraction between 0.315 and 0.63 mm (Figure 1) and (Figure 2).

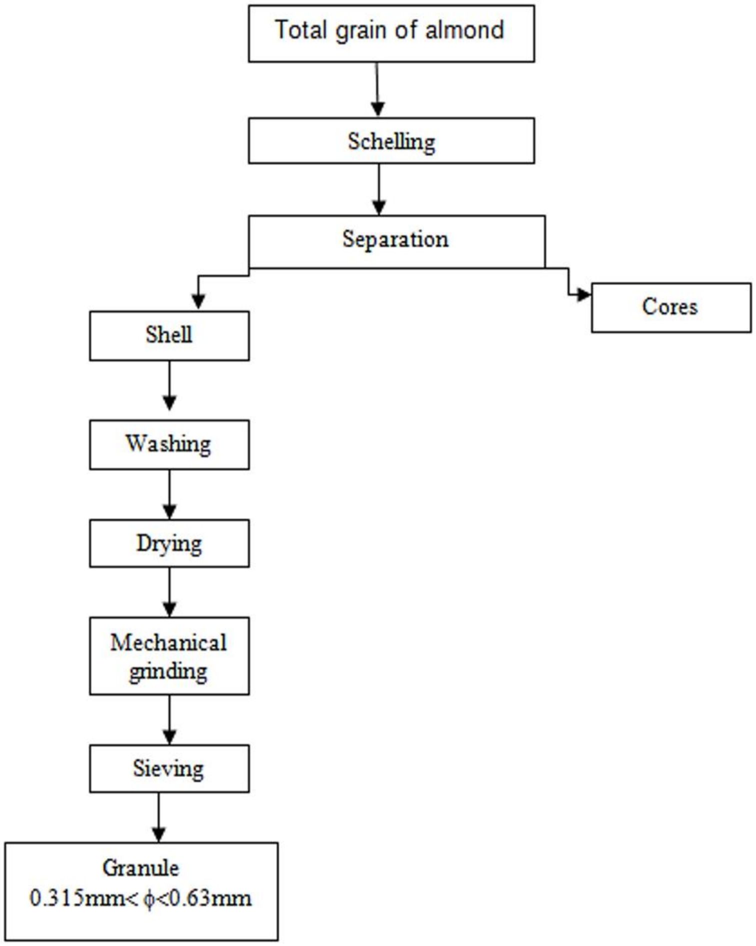


**Figure 1.**
Preparation of raw activated carbon.

**Figure 2.**
Grains of raw activated carbon.

### 2.1.2. *Acid Chemical Activation (Aca)*

- The activation was carried out using phosphoric acid ($H_3PO_4$): a mass of 25 g of almond shell seed was placed in a beaker containing 25 mL of 85% phosphoric acid.

- The solution was dried in an oven at 105°C for 24 hours. After heating, the acid-activated shell took the form of a disc. The disc was then ground using a manual mortar and washed with water, followed by neutralization with a 0.5N $NaHCO_3$ solution.
- The activated carbon neutralized acid activation was washed again with water until the residual water reached a pH of 6. Finally, the acid-activated carbon (AAC) was dried in an oven at 105°C for 24 hours. The resulting (AAC) was stored in a tightly closed bottle. (Fig. 3) shows the final state of the prepared (AAC).

**Figure 3.**
Prepared acid-activated carbon (AAC).

### *2.1.3. Basic Chemical Activation (Bca)*

- Chemical activation with $H_2SO_4$: A 25 g mass of almond shell seeds was placed in an $H_2SO_4$ solution (97%) for 24 hours and then washed several times with water until the residual liquid reached a pH of 6.
- The mixture was then immersed in 100 ml of KOH (2N) while stirring at 85°C for 2 hours. To obtain the basic activated carbon (BAC), the liquid was filtered to separate the activated shell grains, which were then dried at 120°C for 24 hours. The resulting BAC was stored in a tightly closed bottle. The final state of the prepared BAC is shown in Fig. 4.

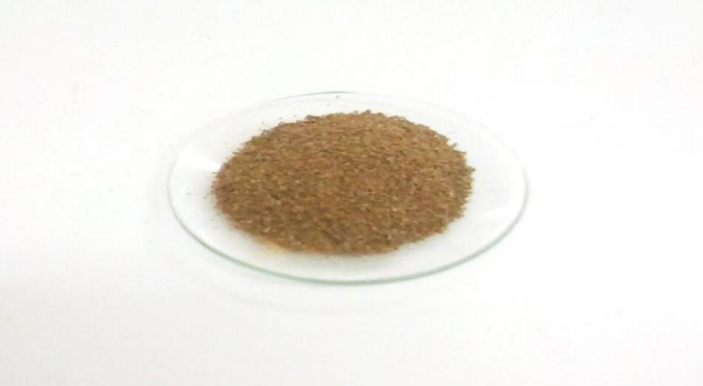


**Figure 4.**
Prepared basic activated carbon (BAC).

The surface functional groups of the acid- and basic-activated carbon were detected by Fourier transform.

infrared (FT-IR) spectroscope (SHIMADZU FT-IR- 8400). The spectra were recorded from 4,000 to 400 $cm^{-1}$.

### *2.2. Chemicals*

Methyl orange ($C_{14}H_{14}N_3NaO_3S$) is an azo dye synthesized through a diazo coupling reaction. It is produced from sulfamic acid and dimethylaniline. Since sulfamic acid is insoluble in acidic solutions, it is first alkalinized. Afterwards, it undergoes diazotization and reacts with dimethylaniline. The initial product formed is helianthin, which is the acidic form of methyl orange. In basic solutions, helianthin is converted into the sodium form, known as methyl orange.

### *2.3. Analysis*

The methyl orange concentration in the supernatant solution was analyzed using a UV spectrophotometer.

(Shimadzu UV Mini-1240) by monitoring the absorbance changes at a wavelength of maximum absorbance of 465 nm[31].

### *2.4. Process of Adsorption*

All experiments were conducted in a batch system within an electrochemical cell, where adsorbent samples were brought into contact with 100 ml of aqueous methyl solutions at a specific pH. The mixtures were subjected to mechanical stirring. The samples were then analyzed by ultraviolet-visible spectroscopy. The initial and final pH values of the aqueous phase were measured using a digital pH meter.

The adsorbed quantity of aqueous solution (q) per unit mass of the solid support is calculated by the following formula:

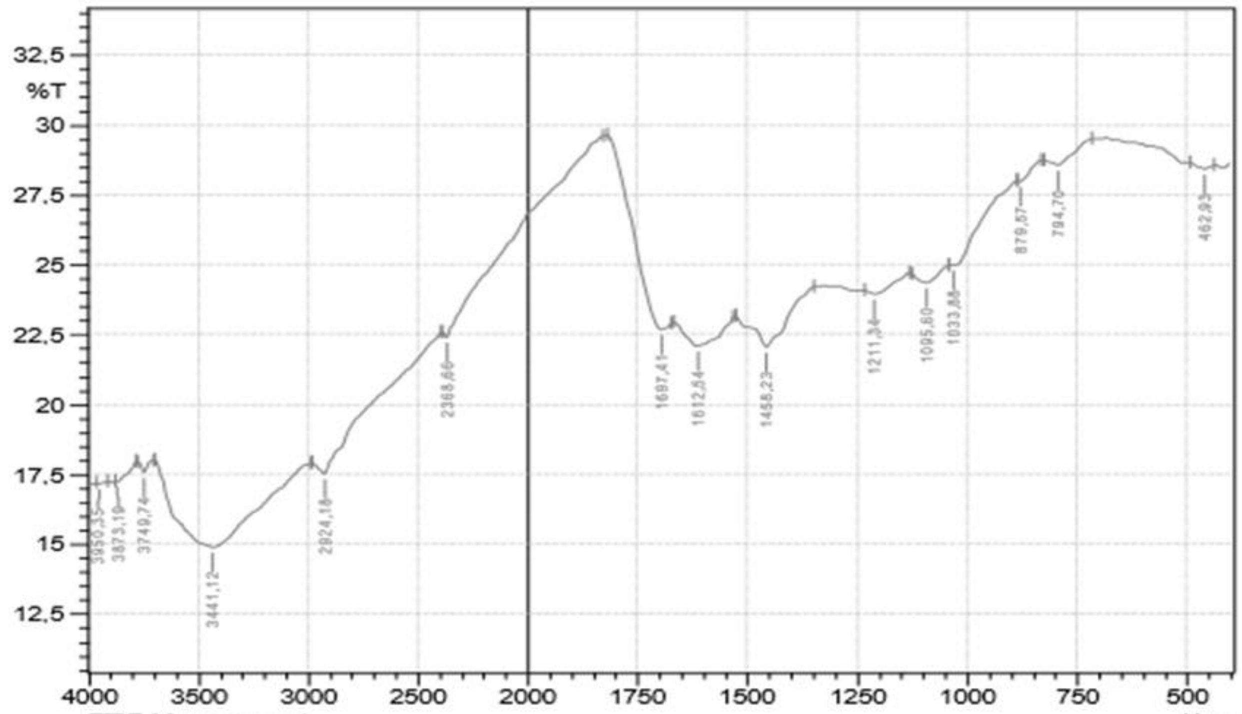
$$q = (C_0 - C_e)\frac{V}{m}(1)$$

Where q is the adsorbed quantity of MO per gram of adsorbent at time t (mg/g); $C_e$ is the concentration of the solution at equilibrium (mg/L); $C_0$ is the initial MO concentration (mg/L); V is the volume of MO solution (L); m is the mass of biosorbents (g). Thus, the curve of the isotherms of adsorption is: q (mg) = *f*($C_e$ (mg/L)).

## 3. Results and Discussion

### *3.1. Textural Characterization of Prepared Activated Carbon*

The FT-IR spectra of acid, basic, and raw activated carbon (Fig. 5) showed the presence of a band around 3400 $cm^{-1}$, corresponding to the presence of O-H groups. Another band around 2924 $cm^{-1}$ relates to C–H groups, which were observed in the raw charcoal and after acid activation but were absent in basic activation. A band at 1612.79 $cm^{-1}$ indicates the presence of the C=O group, and a band at 1913.45 $cm^{-1}$ is likely associated with the characteristics of C=C olefins and aromatic bonds. The bands around 1030.00 and 1040.00 $cm^{-1}$ correspond to the vibration of C–O–C in polysaccharides. These bands result from the presence of cellulose, hemicelluloses, and lignin, which are the major constituents of the almond shell.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1211,34
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1033,88
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FTIR Measurement
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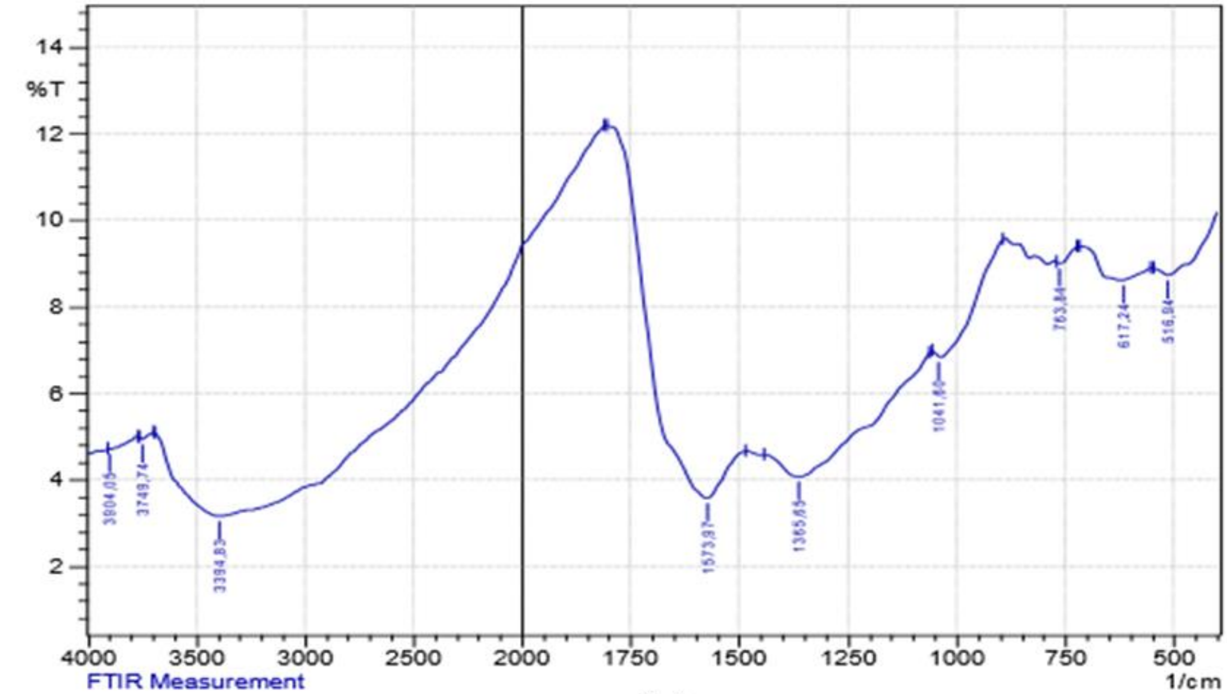
(a)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1041,80
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FTIR Measurement
1/cm

(b)

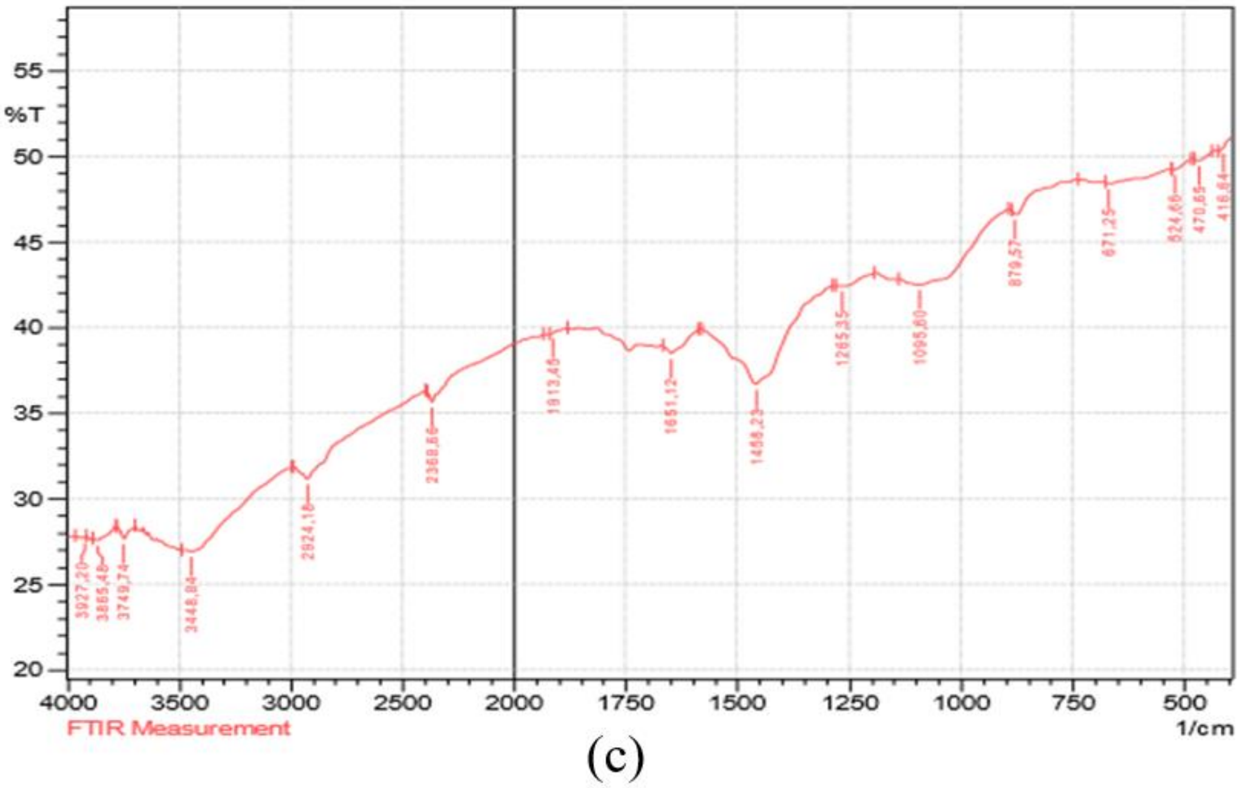


**Figure 5.**
FT-IR spectra (a): Acid-activated carbon, (b): Basic activated carbon, and (c): raw activated carbon.

Biosorption data for the uptake of M O onto activated carbon were investigated at various contact times, pH levels, temperatures, and masses of adsorbent. The results are presented as the adsorption yield ($\Upsilon$) of MO per percent.

Adsorption yield is given as:

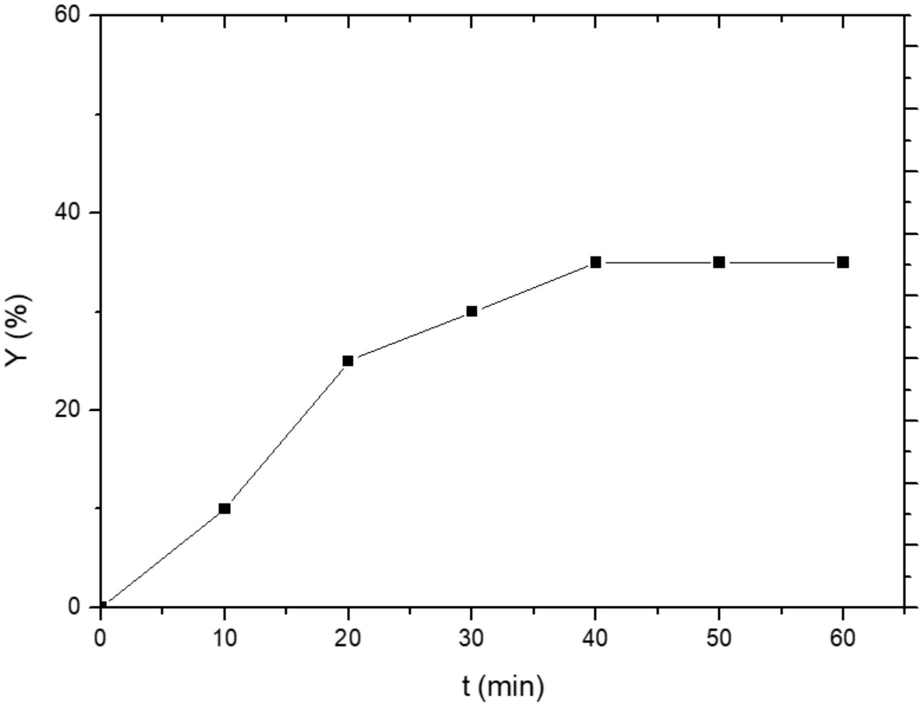
$$(\Upsilon.\%)=\frac{C_0-C_1}{C_0}*100 \qquad (2)$$

Where $C_0$ is the initial dye concentration in (mg $L^{-1}$) and '$C_1$' is the concentration of dye in solution at time *t* in (mg $L^{-1}$).

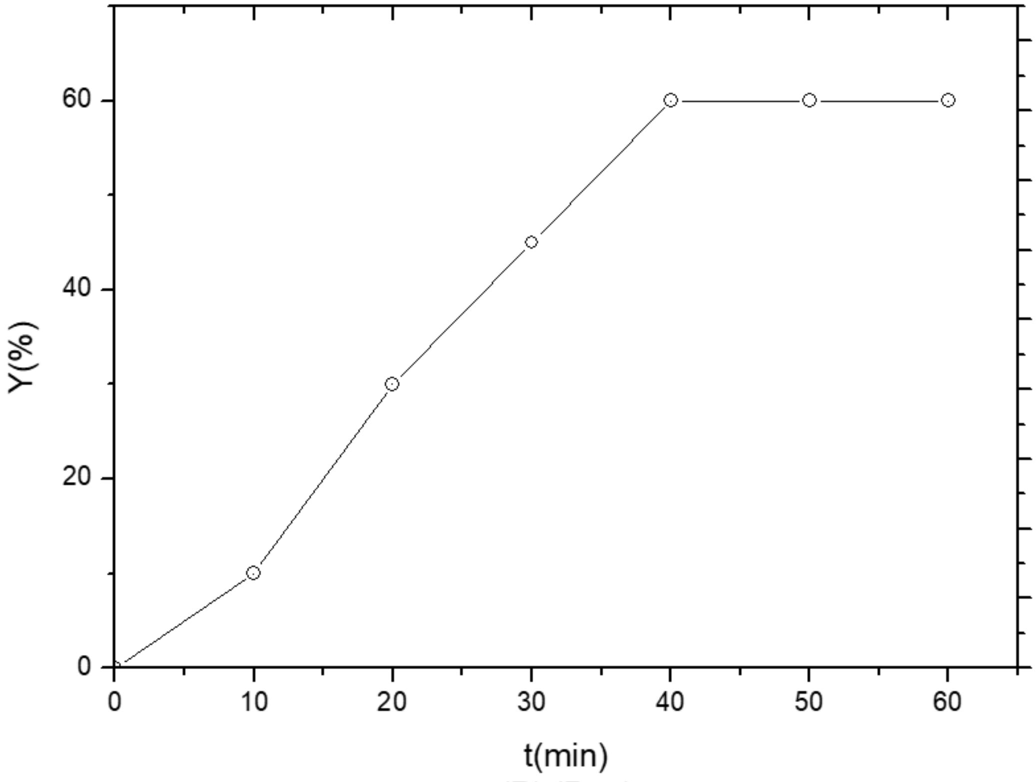
### *3.2. Effect of Operating Conditions*

#### *3.2.1. Effect of Contact Time*

The relative tests to determine the effect of contact time were carried out at a temperature (T) of 25°C and at pH = 7 of the solution. The initial dye concentration was 10 ppm. The mass of adsorbent (AAC and BAC) was 0.2 g. The mixture was stirred at an average speed of 250 rpm. Samples of the aqueous phase were taken over time to measure the adsorption yield ($\Upsilon$ (%)). We then determined the variation in the residual concentration of MO in the aqueous solution using UV spectrophotometry.



(A): (Aac)



(B):(Baa)

**Figure 6.**
Effect of contact time on the removal of MO (m=0.2g, w=250 tr/min, pH=7, [MO] =10ppm, T25(C°)).

The dye yield on activated carbon increases with contact time (Fig.6). Equilibrium is reached after approximately 40 minutes, with yields of 35% and 60% for both acid and basic activated carbon, respectively. The results indicate the existence of two phases: the first is rapid, and the second is slow. This is related to the high availability of free active sites on the adsorbent at the beginning of the experiment, which decreases as the experiment progresses.

### *3.2.2. Effect of pH*

In our study, we monitored the effect of pH on adsorption. The initial concentration of the methyl orange solution was 10 ppm. The aqueous solution was adjusted to different pH values (2, 4, 6, 8, 10, 12) using 0.2 g of adsorbent (AAC and BAC) at a temperature of 25°C. The mixtures were maintained under constant stirring at 250 rpm for 40 minutes. The pH adjustment was carried out by adding a few drops of acid (HCl) 0.1 N or base (NaOH) 0.1 N. Subsequently, the samples were analyzed by a UV-visible spectrophotometer.

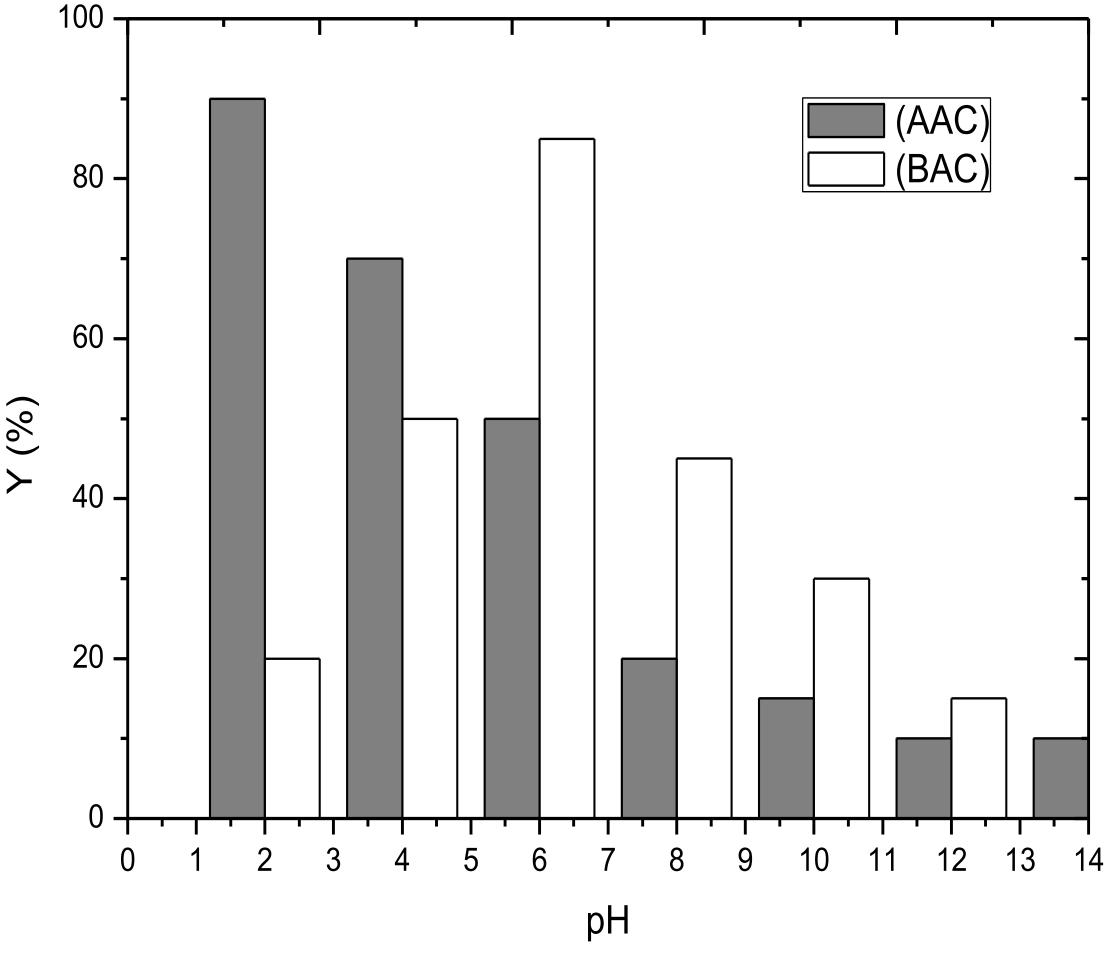


**Figure 7.**
Effect of pH on the removal of MO (m=0.2g, w=250 tr/min, t=40min, [MO] =10ppm, T25(C°)).

The results presented in (Fig.7) demonstrate that for adsorption using acid-activated carbon (AAC), the maximum yield was approximately 90%, occurring at a pH of 2. This can be explained by the presence of positive charges on the surface of the activated carbon. At acidic pH values, the number of positively charged sites increases, promoting the adsorption of anions through electrostatic attraction.

This is likely the case for methyl orange, which is an anionic dye. The decrease in adsorption yield with increasing pH in alkaline medium can be attributed to an excess of $OH^-$ ions, which compete with the dye for adsorption sites on the activated carbon. The high concentration and mobility of $OH^-$ ions favor their adsorption over that of the dye. Therefore, the optimal pH for maximum methyl orange adsorption on acid-activated carbon under the study conditions is pH 2. For adsorption using basic-activated carbon (BAC), the results show that the maximum yield was approximately 85%, occurring at a pH of 6.

These phenomena can be explained by the fact that at high initial pH values, the surface of the adsorbent is negatively charged and is surrounded by ions from the alkaline medium. As a result, the negative charge predominates, which reduces the interaction between methyl orange ions (anionic pollutant) and the adsorbent sites. However, at acidic pH levels, the concentration of negative charges decreases, and the number of positive charges increases, leading to enhanced interactions between the dye ions and the surface sites.

### *3.2.3. Effect of Temperature*

Temperature is an important factor that influences the adsorption capacity of an adsorbent. Additionally, the solution's characteristics are also affected by ambient temperature. To illustrate the influence of this parameter on adsorption, 0.2 grams of support (AAC) and (BAC) were placed in contact with solutions of 10 ppm of MO for 40 minutes, at pH=2 for (AAC) and pH=6 for (BAC), at a stirring rate of 250 rpm.

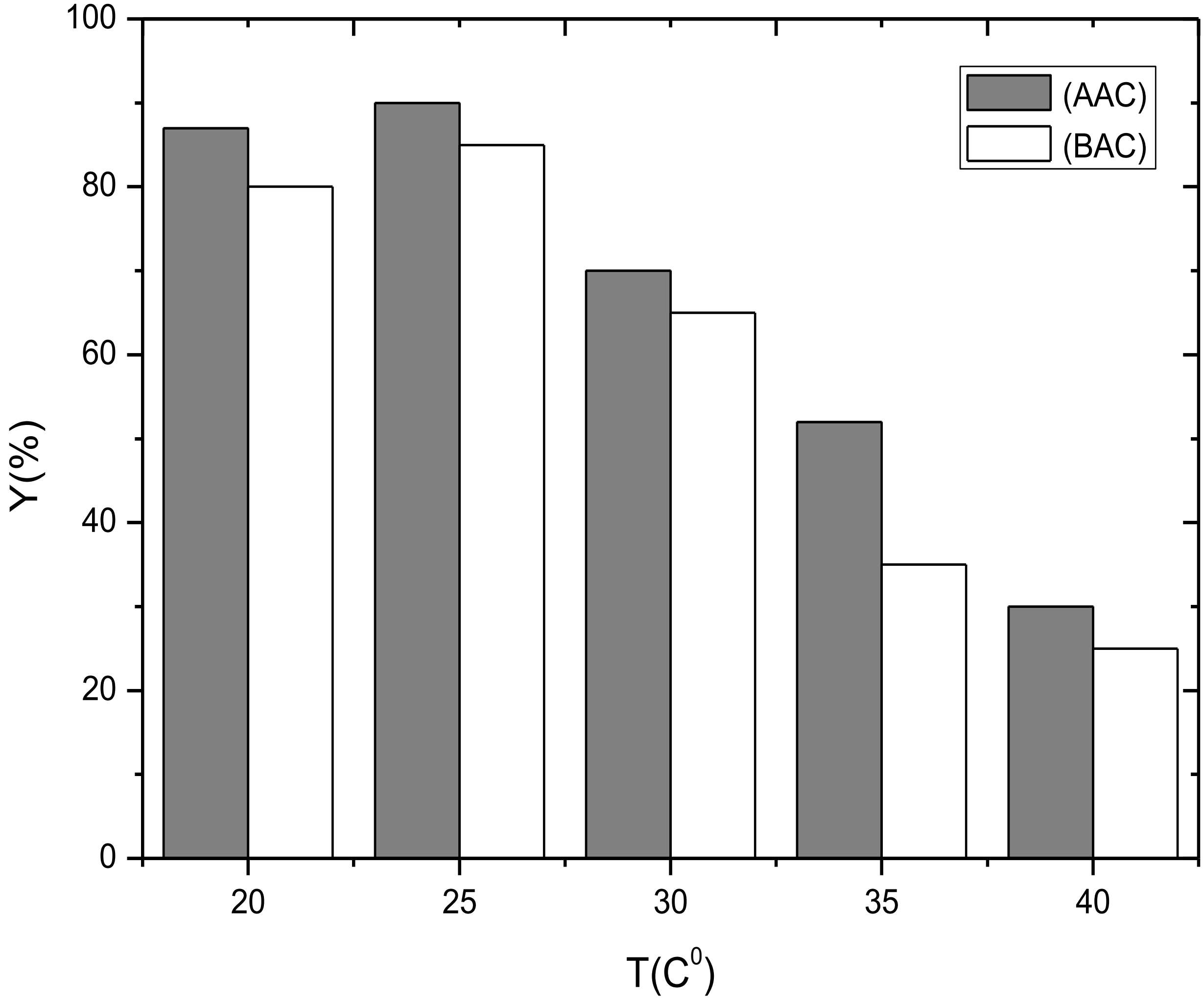


**Figure 8.**
Effect of temperature on the removal of MO (m=0.2g, w=250 tr/min, t=40min, [MO] =10ppm, pH =2 for (AAC), pH=6 for (BAC)).

From the results shown in (Fig.8), we observed that temperatures between 20 and 30°C do not have a significant influence on the yield of MO adsorbed onto both AAC and BAC. However, above 30°C, the adsorption yield (*Y*) decreases as the temperature increases, which may be attributed to the desorption phenomenon that could occur during the reaction. Therefore, it is advisable to conduct the experiments at room temperature.

### *3.2.4. Effect of Adsorbent Mass*

The adsorption of methyl orange onto the two types of activated carbon (AAC and BAC) was carried out at 25°C with varying adsorbent masses from 0.1 to 0.5 g. The pH was 2 for AAC and 6 for BAC, respectively.

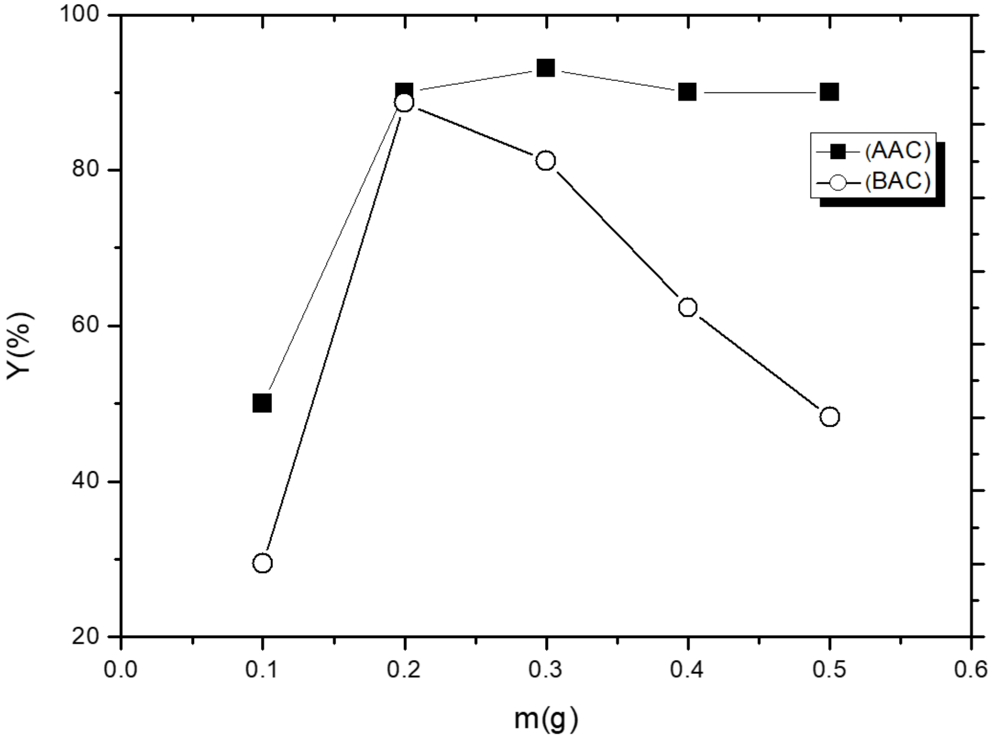


**Figure 9.**
Effect of adsorbent mass on the removal of MO (w=250 tr/min, t=40min, [MO] =10ppm, pH =2 for (AAC), pH=6 for (BAC), T=25(C°)).

For the acid-activated carbon (AAC) (Fig.9), we noted that as the mass of the carbon increased, the adsorption yield rose from 50% for 0.1 g to 90% for 0.2 g and then stabilized. This indicates a larger surface area of the carbon and a greater number of active sites available for the retention of OM molecules. The stabilization occurs due to the saturation of the available active sites. Therefore, 0.2 g is the optimal mass chosen.

For the utilization of basic activation carbon (Fig. 9), and according to the obtained results, which indicate that the adsorbent masses between 0.1 and 0.2 g cause the adsorption rate to increase from 30% to 85%, but beyond 0.2 g, the yield decreases. This can be explained by the contamination of our BAC.

### *3.3. Modeling of Adsorption Kinetics*

In order to study the mechanism of the adsorption process, two simplified kinetic models were applied. The first was the pseudo-first-order equation given by Redlich and Peterson [32] as:

$$\log(q_e - q_t)=\log(q_e)-\frac{K_1}{2.303}t \qquad (3)$$

Where $q_e$ and $q_t$ are the amounts of MO adsorbed (mg/g) at equilibrium and at time t (min), respectively, and $k_1$ ($min^{-1}$) is the rate constant of adsorption.

On the other hand, the pseudo-second-order equation based on equilibrium adsorption[33] is expressed as:

$$\frac{t}{q_t} = \frac{1}{K_2\,q_e^2}+\frac{1}{q_e}t \qquad (4)$$

Where $k_2$ (g/mg min) is the rate constant of second-order adsorption.

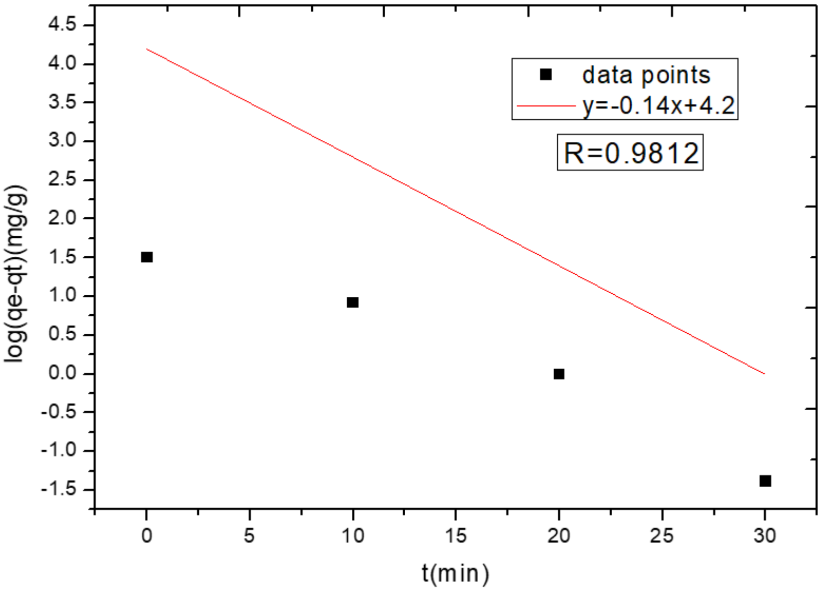
*3.4. Validity of kinetic models*

The validity of each model was determined by the root sum of squareerror (RMSE, %) given by the following equation[34]:

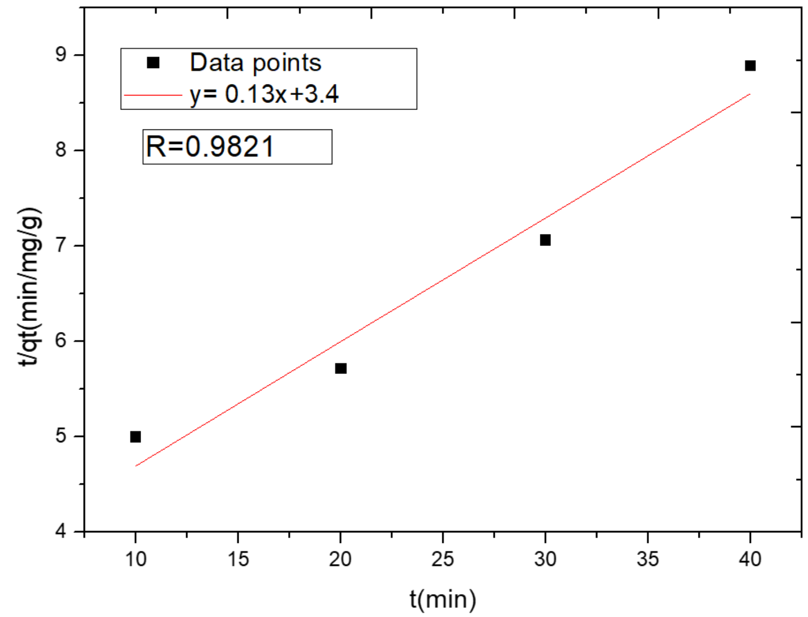
$$RMSE = \sqrt{\frac{1}{N}\sum_{q=1}^{N}\left[(Q)_N^{exp} - (Q)_N^{predict}\right]^2} \quad (5)$$

Where N is the number of data points, and Q are $\log(q_e - q_t)$and$(\frac{t}{q_t})$ experimental and predicted data for each kinetic model.

The graphical representations of the pseudo-first-order and pseudo-second-order equations are given, respectively, by figures (Fig.10a and Fig.10b) for acid-activated carbon and figures (Fig.11a and Fig.11b) for basic-activated carbon.

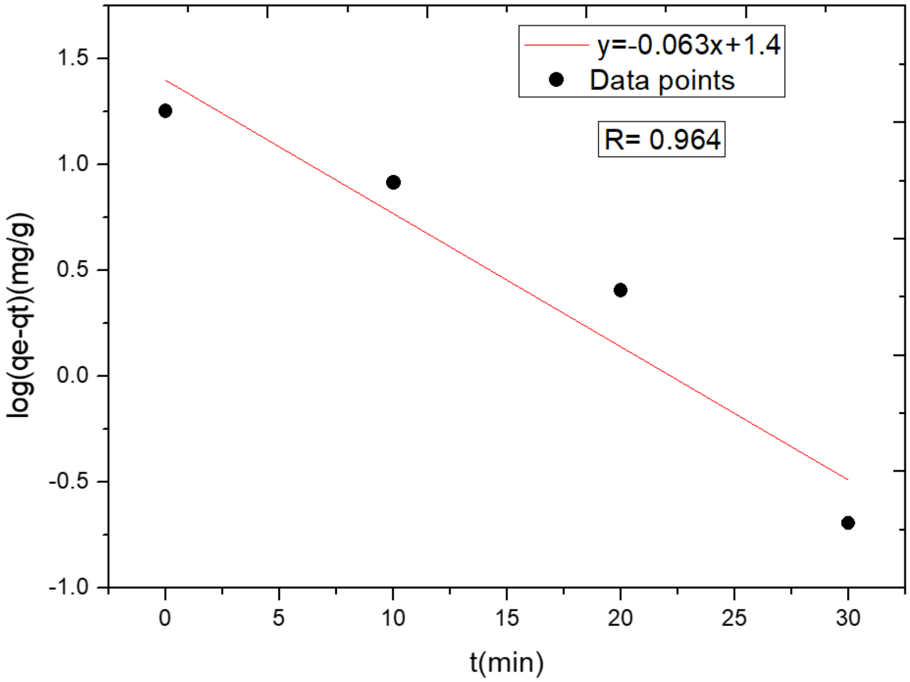


(a)



(b)

**Figure 10.**
Modeling of kinetic adsorption of MO onto (AAC)



(a)

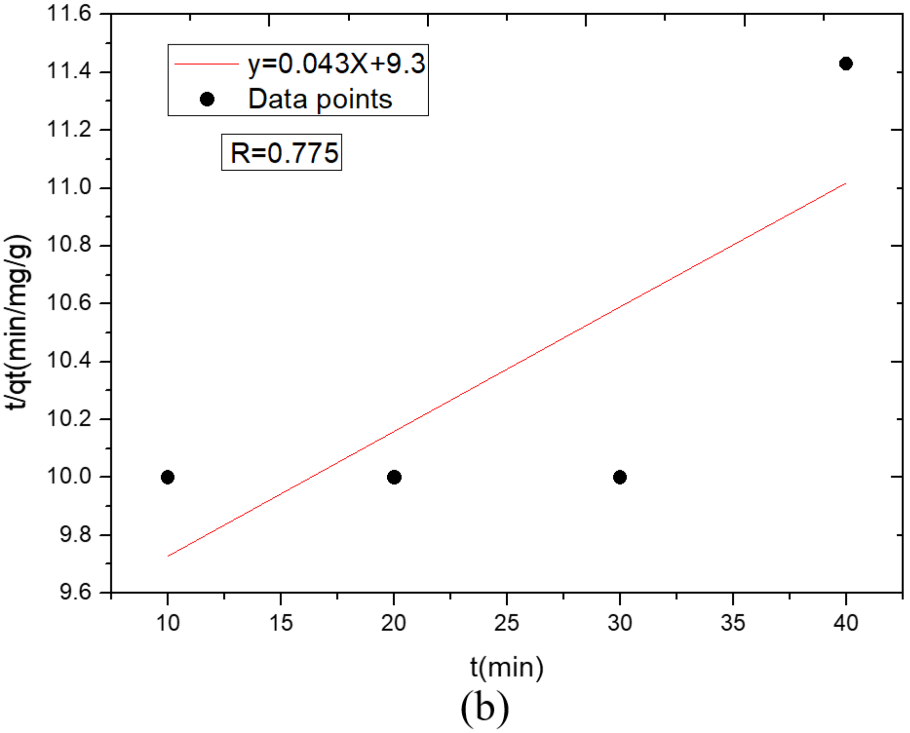


**Figure 11.**
Modeling of kinetic adsorption of MO onto (BAC).

For the acid-activated carbon (AAC), the obtained results show a high correlation coefficient (R) > 0.98 for the pseudo-first and second-order models. Nevertheless, for the pseudo-second order model, the correlation coefficient (R) is slightly higher than that obtained by the pseudo-first order model. Therefore, the pseudo-second order is considered the best model. For the basic activation carbon (BAC), the correlation coefficient obtained by the pseudo-first order model (R=0.964) is higher than that obtained by the pseudo-second order (R=0.775). Consequently, the pseudo-first-order kinetic model is well-suited to simulate the time evolution of the dye concentration in the solution. The comparison between the pseudo-first-order and pseudo-second-order performances on acid and basic activation carbon adsorbents is given in Table 1.

According to the results in Table 1, and in addition to the correlation coefficient values, the lowest values of (RMSE) indicate the better kinetic model. It was confirmed that, for the adsorption using (AAC), the pseudo-second-order kinetic model is appropriate to describe the removal of MO onto acid-activated carbon. For the adsorption using (BAC), the pseudo-first-order kinetic model yielded the lowest (RMSE) values, so it is considered the suitable model in describing the treatment of water from MO onto basic-activated carbon.

**Table 1.**
Comparison of the pseudo-first-order and pseudo-second-order performances on acid and basic activation carbon adsorbent.

| Adsorbent | Operating conditions | | Pseudo- first- order kinetic model | | | Pseudo-second-order kinetic model | | |
|---|---|---|---|---|---|---|---|---|
| | $C_0$ (ppm) | $q_e$(mg/g) | $K_1$ (1/min) | R | RMSE (%) | $K_2$ (g/mg min) | R | RMSE (%) |
| (AAC) | 10 | 4.5 | 0.3224 | 0.9812 | 191.05 | 0.024 | 0.9821 | 27.22 |
| (BAC) | 10 | 4.25 | 0.1451 | 0.964 | 19.23 | 0.009 | 0.775 | 39.55 |

Table 2 compares the adsorption capacity of various adsorbents for methyl orange reported in the literature. It is reasonable to expect that variations in qualities and operating conditions will produce different results. These changes are evident in the results presented in Table 2, indicating that the properties of the adsorbent have a significant impact on its efficiency.

**Table 2.**
Comparison between the obtained adsorption performances and various adsorbents in the literature works.

| **Adsorbent name** | **R%** | ***q(mg/g)*** | **Reference** |
|---|---|---|---|
| AC from coffee waste | - | 1017 | Rattanapanet al. [28] |
| AC from aloe vera | - | 196.1 | Khaniabadiet al. [29] |
| AC from coconutshell | 100 | 3.00 | Islam et al. [30] |
| ACfrom waste tire rubber | 80 | - | Islam et al. [35] |
| AC from popcorn | 48.5 | - | Yu et al. [36] |
| AAC from almond shell | 90 | 4.5 | This work |
| BAC from almond shell | 85 | 4.25 | This work |

## 4. Conclusion

This study, which falls within the framework of wastewater treatment, aims to investigate the reactivity and effectiveness of a bio-support (almond shells) in the adsorption process of an anionic dye (methyl orange). The results obtained in this study showed:

- The possibility of producing activated carbons from almond shells through basic chemical activation using potassium hydroxide (KOH) and acid chemical activation using ($H_3PO_4$).
- The almond shells were characterized by FTIR. The bands obtained in the spectrum of the almond shell grains before activation were either absent or less intense compared to those observed after chemical treatment, indicating that the grains were well activated.
- The retention of the dye by the support was rapid during the first minute of contact, reaching a plateau that corresponds to equilibrium after 40 minutes, which is related to the availability of active sites on the support.
- pH and temperature are parameters to consider. At room temperature, the adsorption capacity was maximized at approximately 90% at pH=2 for the (AAC) and 85% at pH=6 for the (BAC). The effect of the mass of activated carbon shows that the removal efficiency of MO increases with the increase in the quantity of activated carbon. This result can be explained by the increase in the contact surface area provided by the adsorbent.
- The appropriateness of the first- and second-order kinetic models for the sorption of dye onto adsorbents is also discussed. It was concluded that the biosorption kinetics of MO onto acid and basic activated carbon follow, respectively, the second-order and first-order adsorption kinetics.
- The results derived from this work confirm the possibility of valorizing almond shells as adsorbents for treating wastewater from dyes.

## Transparency:

The authors confirm that the manuscript is an honest, accurate, and transparent account of the study; that no vital features of the study have been omitted; and that any discrepancies from the study as planned have been explained. This study followed all ethical practices during writing.

## Copyright:

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