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# Mechanism Study of Indigo Carmine (IC) Dye removal by adsorption using Nano Charcoal based on Walnut shells

**Noor S. Abd AL-hadi [1,a],Laith A. Hameed [2,b] and Huda Mutar Ibrahim[3,c]**

[1]Department of Chemistry, College of Education, University of Samarra, Samarra, Iraq, [a] noor.saddam18@gmail.com

[2] Department of civil, College of engineering, University of Samarra, Samarra, Iraq,[b] laith.amjad@uosamarra.edu.iq

[3]Department of Chemistry, College of Education, University of Samarra, Samarra, Iraq, [c] huda.mutar@uosamarra.edu.iq

**Abstract**

Effluents from textile industries pose significant environmental and human health problems especially since some textile factories discharge highly colored wastewater into the River, affecting water quality and local activities. This study aims to develop an eco-friendly method to remove Indigo Carmine (IC) dye from the water using Walnut shells-based activated carbon (WSAC). Adsorption was conducted by mixing powdered WSAC with IC dye at 120 rpm. Parameters such as contact time, absorbent quantity, and dye concentration were examined. Samples were analyzed using UV-Vis spectrophotometry at 610 nm. Kinetic and isotherm models were used to describe adsorption properties. The study found that 98 % of IC dye could be removed using 750 mg of powdered activated carbon in 100 ml solution, with an equilibrium time of 40 minutes. The pseudo-second-order model best described the adsorption with a correlation coefficient of $R^2$ = 0.9905.

**Keywords**:-Textile effluent, Indigo Carmine, Adsorption, Walnut shells, Charcoal

## 1. Introduction

The textile industries have developed significantly in recent decades. Most industrial processes result in pollutant discharges due to the intensive use of additives and chemical dyes. Indigo Carmine is the most commonly used dye for denim fabrics. After washing and bleaching jeans, the colored effluents discharged into nature can alter the aquatic environment and ecological balance. Textile effluents containing organic dyes have high BOD and COD levels [1,17,18]. It's estimated that nearly 20% of the dyes used on fabrics do not adhere and end up in wastewater [2,19]. Synthetic dyes can be challenging to biodegrade and are often highly toxic. Their removal from wastewater is thus necessary. Several dye removal techniques have been conducted by various authors, including biological, chemical, and physical methods. Coagulation-flocculation [3], membrane filtration [4,20], ion exchange, activated sludge, and adsorption [5,6,7] are the most commonly used in conventional treatment systems. Adsorption is considered an effective and simple method for dye removal. Activated Charcoal is one of the most commonly used adsorbent materials [8,22]. From the perspective of industrial ecology and waste valorization, our research focuses on using an ecological absorbent, available locally, made from Walnut shell waste, this material is cheaper and renewable compared to commercial activated carbon [9,26]. In this study, we optimized the use of powdered activated carbon from Walnut shells for the removal of Indigo Carmine dye and aimed for better efficiency than other commonly used methods. We discussed kinetic and isotherm models based on the obtained results. This method is feasible for large-scale treatment without the need for specific or sophisticated installations.

## 2. Charcoal Preparation

The adsorbent used in this study was prepared from Walnut shells. It is a locally crafted Charcoal. After waste collection, the Walnut shells undergo preliminary treatments before carbonization and activation processes:

| Nomenclature | | m | adsorbent amount in the solution (g). |
|---|---|---|---|
| A | the solution absorbance | $K_2$ | the second-order reaction rate constant of IC adsorption Walnut shells Charcoal (L $mg^{-1}$ $min^{-1}$) |
| ε | molar absorption coefficient of the substance | $q_e$ | the adsorbed amount at equilibrium (mg/g) |
| L | optical path length | $q_t$ | the adsorbed amount at time t (mg/g) |
| C | concentration of the substance | t | the contact time (min) |
| $K_1$ | the first-order reaction rate constant of adsorption($min^{-1}$) | qe | the substance amount of adsorbed at equilibrium per unit weight of adsorbent – adsorption capacity (mg/g) |
| $q_e$ | the amount of adsorbed at equilibrium (mg/g) | $q_m$ | the capacity of adsorption at saturation (mg/g) |
| $q_t$ | the amount of adsorbed at time t (mg/g) | $C_e$ | the equilibrium concentration of the adsorbate (mg/L) |
| t | the contact time (min) | $K_L$ | the Langmuir constant |
| $C_0$ | the dye initial concentration (mg/L) | $q_e$ | the adsorbed amount per gram of solid |
| $C_e$ | the dye concentration at time t (mg/L) | $C_e$ | the equilibrium concentration of the adsorbate |
| $q_t$ | amount adsorbed at time t (mg/g) | v | the solution volume (ml) |
| $C_0$ | the dye initial concentration (mg/L) | | |
| $C_e$ | the dye concentration at time t (mg/L) | | |

- Sorting: removal of foreign elements.
- Drying: air drying for several days to reduce moisture and facilitate grinding.
- Grinding: reducing the size of the Walnut shells.
- Sieving: removing small residues.
- Weighing: measuring the amount of Walnut shells to be carbonized.

**Figure 1.** Charcoal powder from Walnut shells.

### 2.1. Carbonization

For this step, a furnace, an electric fan (used for blowing), a crucible (containing the product to be carbonized), and a thermometer (to control the temperature) are needed.

The procedure is as follows:

- Prepare the furnace.

- Place the products to be carbonized into the crucible and seal it.
- Place the crucible in preheated oven set to a temperature between 650°C and 700°C for 3 hours, utilizing charcoal combustion enhanced by an electric fan.
- After cooling, remove the carbonized product from the crucible.

### 2.1.2. Physical Activation

Activation of the material develops the porous structure of the carbon, enhancing its adsorption capacity. For activation, the carbon is treated at a temperature ranging from 800°C to 1000°C for 3 hours. The electric fan increases combustion, allowing the temperature to reach up to 1000°C. After cooling, the activated carbon is removed from the crucible and weighed. To obtain the powder, the raw carbon was ground and then sieved to separate the powders from the grains. For 500 g of Walnut shells after carbonization at 650°C for 3 hours, 212 g of carbon was obtained. After activation at 1000°C for 3 hours, 140 g of activated carbon was finally obtained.

### 2.2. Preparation of the Adsorbate Solution

The stock solution of IC 50 mg $L^{-1}$ is prepared by dissolving the indigo carmine dye in 1 liter of distilled water. The dye is from the indigoid family, with its structure shown in Fig. 1. The working solutions are prepared by successful dilutions of the stock solution to obtain the test concentrations. The colored solutions are then analyzed using a UV spectrophotometer at a wavelength of 610 nm. A calibration curve representing absorbance (A) as a function of concentration (C) according to Beer-Lambert's law is established. Fig. 2 shows the calibration curve of indigo carmine.

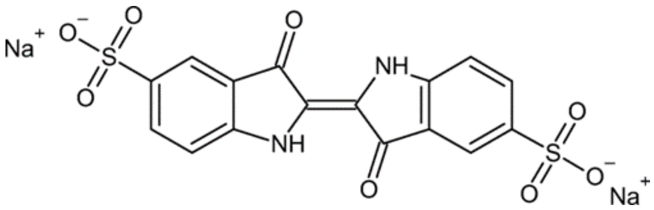


**Figure 2**. Structure of Indigo Carmine

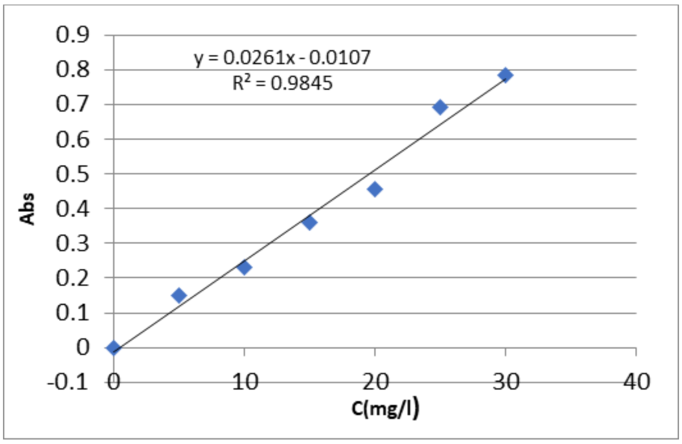


**Figure 3.** Calibration Curve of Indigo Carmine

### 2.3. Description of Adsorption Tests

Adsorption is performed by contacting aqueous solutions of organic compounds (indigo carmine) with increasing amounts of Charcoal in 250 ml beakers. The agitation time required to reach equilibrium was determined. The samples are then vacuum-filtered, centrifuged, and analyzed using a UV spectrophotometer. Concentrations are then deduced using the Beer Lambert law:

$$A = \varepsilon LC \quad (1)$$

### 2.4. Adsorption Test

#### 2.4.1. Effect of Charcoal Mass

For the adsorption tests based on Charcoal mass, five (05) samples of a 100 ml IC solution with a concentration of 50 mg/L were prepared with different masses of Charcoal (250 mg, 500 mg, 750 mg, 1000 mg, 1250 mg ), under magnetic stirring at room temperature for 60 minutes. The measured pH of the solutions was 4.70. After agitation (120 rpm), the solution was filtered and centrifuged. The filter was analyzed using a UV-visible spectrophotometer. The removal efficiency (%) of IC by adsorption is given by the following relationship:

$$R=\frac{(C_0-C_e)}{C_0} * \mathbf{100} \quad (2)$$

#### 2.4.2. Effect of Adsorption Time

To predict the time required to reach adsorption equilibrium of indigo carmine on Charcoal, we prepared five 250 ml beakers. Each beaker contains the optimal mass of activated charcoal obtained from the previous experiment and 100 ml of indigo carmine solution at a concentration with 50 mg/L, stirred magnetically at room temperature. The absorbance of the solution was measured after 20 minutes, 40 minutes, 60 minutes, 80 minutes, 90 minutes, and 100 minutes of operation. After separation by vacuum filtration, the filtrate was analyzed using a UV-visible spectrophotometer. The adsorption capacity of the Charcoal was calculated using the equation:

$$\mathbf{q_t = (C_0 - C_e) * \frac{v}{m}} \quad (3)$$

#### 2.4.3. Effect of Concentration

The initial concentration of the adsorbate was varied to determine the adsorption isotherms. Five samples of 100 ml IC solution with concentrations of (20, 40, 60, 80, and 100) mg/L were prepared under stirring at room temperature with the optimal mass of activated Charcoal and contact time from previous experiments. After vacuum filtration, the absorbance of the resulting solutions was measured.

### 2.5. Kinetic Modeling of Adsorption

The kinetic modeling of IC adsorption on Walnut shells Charcoal is performed using surface reaction kinetic models. Two important kinetic models are presented: pseudo-first-order model and pseudo-second-order model.

- **Pseudo-First-Order Model (PFO)**

The equation used is pseudo-first-order model or Lagergren model:

$$dq_t/dt = K_1\ (q_e - q_t) \quad (4)$$

After integration for initial conditions $q_t = 0$ at $t = 0$, the equation becomes:

$$q_t = q_e\,(1 - e^{-k_1 t}) \quad (5)$$

The linearized form of the equation is:

$$\ln (q_e — q_t) = \ln q_e — K_1 t \quad (6)$$

By plotting $\ln (q_e - q_t) = f(t)$, a straight line is achieved, allowing for the determination of $K_1$ and $q_e$. This model explains the events happening in the early stages of the adsorption process [8,10].

- **Pseudo-Second-Order Model (PSO):**

The pseudo-second-order model is represented by the following expression:

$$dq_t/dt = K_2( q_e – q_t )^2 \quad (7)$$

$$t/qt = 1/K_2.qe^2 + (1/qe).t \quad (8)$$

By plotting $t/q_t = f(t)$, a straight line is obtained from which $K_2$ and $q_e$ can be determined. In contrast to the first-order model, the pseudo-second-order model is applicable over a broader time interval, typically covering the entire adsorption process [10].

**2.6. Isothermal Adsorption**

The adsorption isotherm is the curve that relates the residual solute concentration (Cr) and the amount adsorbed (q). In this study, the curve was obtained by studying the adsorption of IC on WSAC. We used 100 ml of dye (IC) solutions with different concentrations: from 20 mg/L to 100 mg/L, treated with 750 mg of Charcoal, the optimal mass determined experimentally.

**2.7. Modeling Adsorption Isotherms**

There are many theoretical models to describe adsorption isotherms, but we focused on the Freundlich and Langmuir models [11].

- **Langmuir Model**

It is described by following expression:

$$1/q_e = (1/K_L\, q_m).\ 1/c_e + 1/q_m \quad (9)$$

By plotting $1/q_e$ as a function of $1/C_e$, this equation allows the calculation of the parameters $q_m$ and $K_L$ from the y-intercept and the slope, respectively.

- **Freundlich Model**

The linear equation of Freundlich is given by:

$$\ln q_e = \ln(K_F) + (1/n)\ .\ \ln(C_e) \quad (10)$$

By plotting $\ln q_e$ as a function of $\ln C_e$, this equation allows the calculation of the parameters $K_F$ and $1/n$ from the y -intercept and the slope, respectively.

## 3. Results And Discussions

### 3.1 Effect Of Walnut Shells Charcoal Mass

In the case of powdered Walnut shells Charcoal, 100 ml of indigo carmine solution (50 mg/L) was brought into contact with different masses of Charcoal for 60 minutes. Figure 4 demonstrates a decrease in dye concentration as Charcoal mass increases. As the Charcoal quantity increases, the dye removal efficiency also increases (Figure 5). This experiment shows that starting from 750 mg of Charcoal used, the elimination percentage of the dye remains nearly constant up to 1225 mg. The maximum efficiency obtained is 98 %.

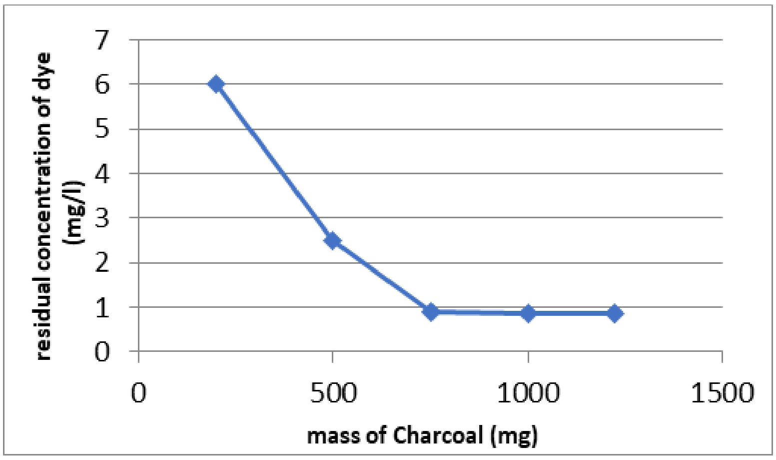


**Figure 4.** Variation in Residual Concentration of Indigo Carmine with Charcoal mass

### 3.2. Effect of Adsorption Time

Figure 7 shows that the amount of dye adsorbed by powdered Charcoal is 6.5 mg/g. The exact time of equilibrium is achieved at 40 min. Above this value, the absorbed amount remains constant (Figure 6). The adsorption kinetics curves can be divided into two zones:

- The first zone, between 5 minutes and 40 minutes, indicates rapid initial adsorption.
- The second zone, between 40 minutes and 60 minutes, the curve stays constant, clearly indicating adsorbent saturation. The curves demonstrate that the adsorption of indigo carmine on Walnut shells is faster at the beginning of the reaction until the adsorbent material reaches saturation.

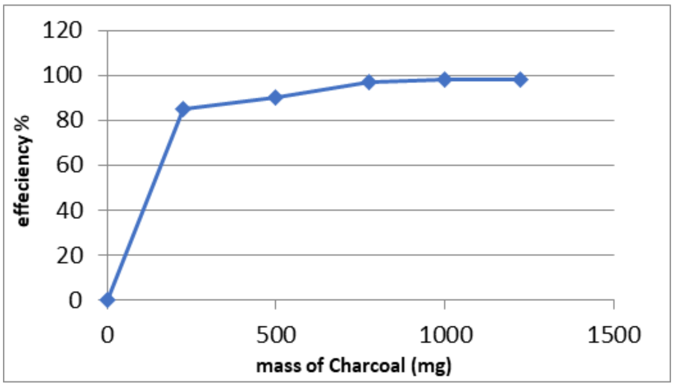


**Figure 5.** Influence of Activated Charcoal Quantity on IC Removal **Efficiency**

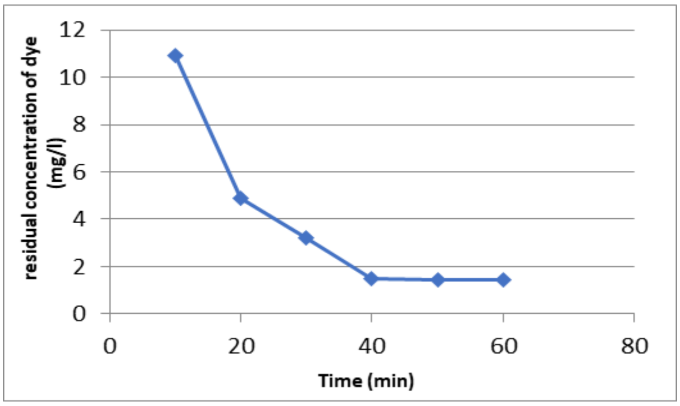


**Figure 6**. Variation in Residual Concentration of Indigo Carmine with time

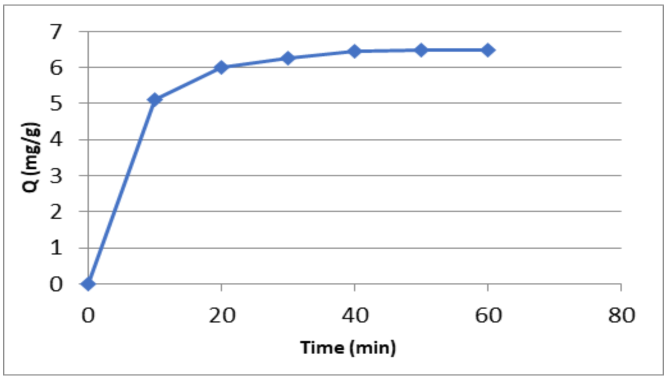


**Figure 7.** Effect of Contact Time on the Adsorption of Indigo Carmine on Powdered Walnut Shells Charcoal

### 3.3. Effect of Concentration

The study of the adsorption isotherm of IC dye (Fig. 8) shows a downward concavity, representing an L-type isotherm. At low solute concentrations, this phenomenon indicates a decrease in available sites as adsorption progresses. Theoretically, this adsorption mechanism reflects a high affinity between the adsorbate and the adsorbent. The isotherm is characteristic of a microporous material, where adsorption initially follows the Freundlich model but aligns with the Langmuir model over the entire process. Under certain conditions, there may also be competition between these two models[8,12,13,14].

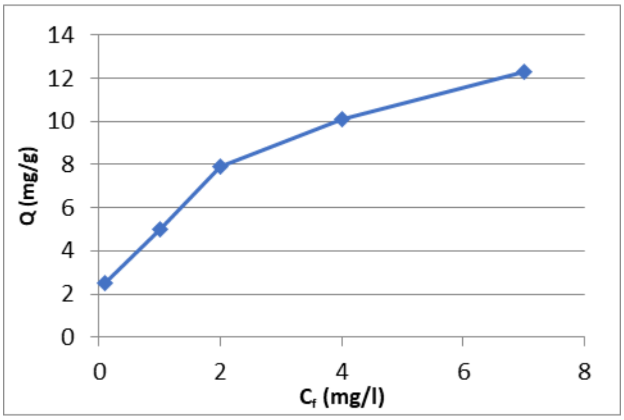


**Figure 8.** Adsorption Isotherm of Indigo Carmine on Walnut shells

### 3.4. Modeling of Results:

#### 3.4.1. Kinetic Modeling:

The pseudo-first-order and pseudo-second-order kinetic models were determined to elucidate the adsorption mechanisms of IC on a Walnut shell adsorbent. From the equations (Fig. 9 and 10), the corresponding linear plots for the two models studied were obtained. From these lines, determine the kinetic constants K, the correlation coefficients $R^2$, as well as the theoretical amounts adsorbed at equilibrium $q_e$ [14] .

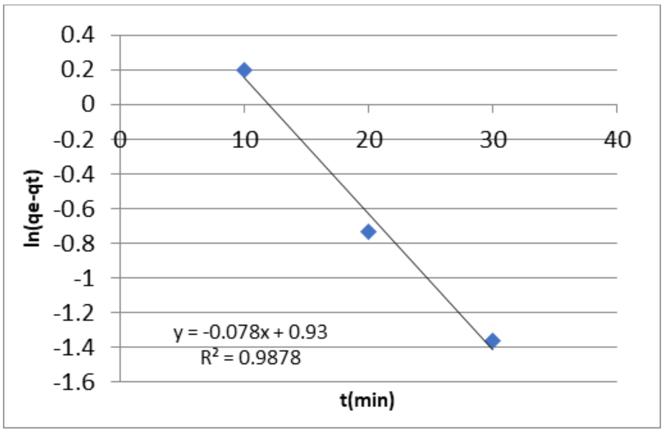


**Figure 9.** Pseudo-First-Order Model of IC Adsorption on Walnut shells

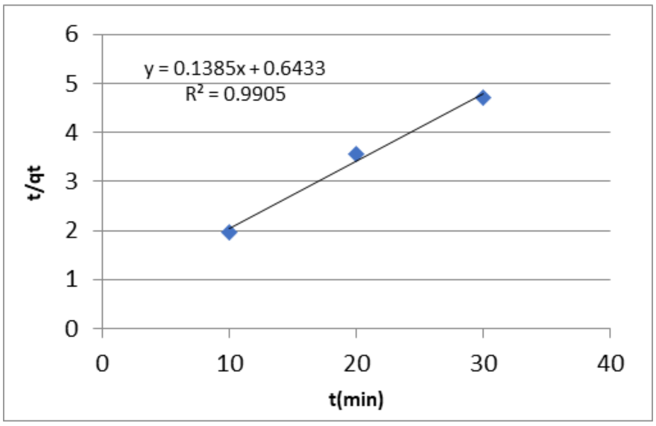


**Figure 10.** Pseudo-Second-Order Model of IC Adsorption on Walnut shells

The kinetic data obtained from the figures are summarized in the table 1:

**Table1.** Kinetic data of IC adsorption for the pseudo-first-order and pseudo-second-order models

| | $K_{1,2}$ | $q_e$, calc.(mg/g) | $R^2$ |
|---|---|---|---|
| First-Order Model | 0.078 | 2.54 | 0.9878 |
| Second-Order Model | 0.0475 | 6.83 | 0.9905 |

In these results, it is observed that the adsorption process of IC dye on Walnut shells follows the pseudo-second-order kinetic model, with a very high correlation coefficient ($R^2$= 0.9905). Comparing the calculated amounts of dye adsorbed at equilibrium, the ($q_e$ = 6.83 mg/g) from the pseudo-second-order model presents a satisfactory result with a high adsorption capacity.

### 3.4.2. Isotherm Models

The plots of the Langmuir and Freundlich isotherms at the adsorption equilibrium of IC dye were developed (Fig. 11 and 12) with the different characteristic parameters for each isotherm.

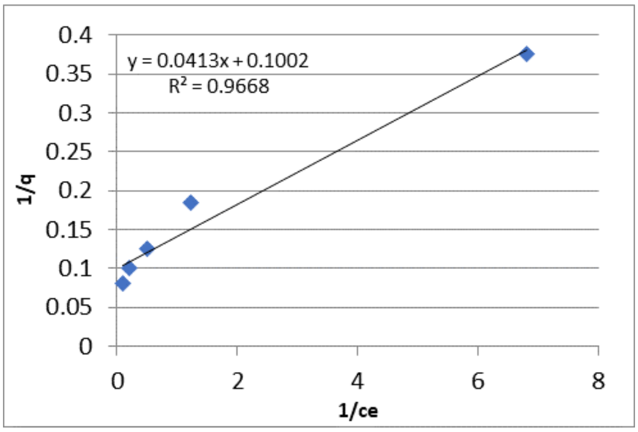


**Figure 11.** Langmuir Model of IC Adsorption on Walnut shells

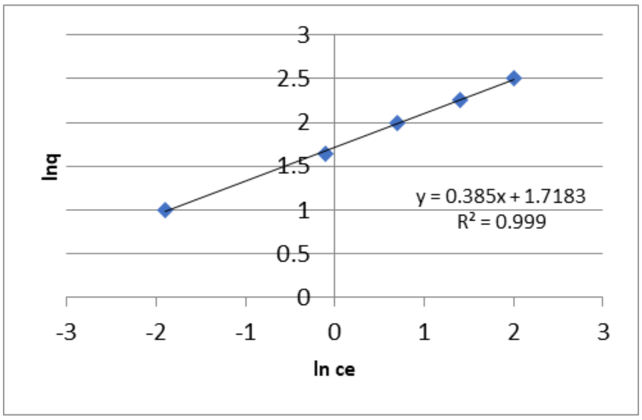


**Figure 12.** Freundlich Model of IC Adsorption on Walnut shells

**Table 2.** Langmuir- Freundlich constants and correlations coefficients of isotherm models

| Langmuir model | | | Freundlich Model | | |
|---|---|---|---|---|---|
| $Q_m$ (mg/g) | $K_L$ | $R^2$ | $K_F$ | 1/n | $R^2$ |
| 9.808 | 2.4 | 0.9668 | 5.63 | 0.385 | 0.999 |

By comparing the parameters of the two models, we can determine the suitable adsorption mechanism model for IC dye. It's noted that the correlation coefficient $R^2$ for the Langmuir model (0.9668) is lower than that for the Freundlich model (0.999). This confirms that the adsorption of IC dye follows the Freundlich model. Moreover, the numerical value of 1/n being between 0 and 1 indicates to dye adsorption is adequate according to this model [12].

## 4. CONCLUSION

In this study, the objective was to use biological materials (Walnut shells-based activated carbon) to remove indigo dye (Indigo Carmine or IC) from colored wastewater resulting from the washing of jeans. By varying different adsorption parameters and mathematical models, described the adsorption mechanism of Indigo Carmine dye and obtained optimal values. After varying the amount of Charcoal used, it was observed that the optimal amount of Walnut shells to treat an IC solution (50 mg/L) in a 100 mL solution is 750 mg. After reaching equilibrium in 40 minutes, the removal efficiency was approximately 98. %. This adsorption capacity is favored by the contact time between the adsorbent and adsorbate until saturation. The maximum adsorption capacity obtained experimentally is 6.5 mg/g. Kinetic studies show that the adsorption process of IC dye follows the pseudo-second-order model with a high correlation coefficient ($R^2 = 0.9905$) and the theoretical equilibrium adsorbed quantity of 6.83 mg/g. Thus, the adsorption mechanism follows the Freundlich model with a numerical value of $1/n << 1$, indicating that the dye adsorption is multilayered. With this high efficiency and without the use of chemicals, we can say that the use of Walnut shells-based Charcoal in the treatment of colored wastewater is an ecological and economical method. In the vision of sustainable development and ecological transition, the use of biological Charcoals aims to reduce intentional and unintentional pollutant emissions in wastewater and to limit the impact of industrial activities on various natural resources: water, soil, and ecosystems.

### Authors' contribution

All authors contributed equally to the preparation of this article.

### Declaration of competing interest

The authors declare no conflicts of interest.

### Funding source

This study didn't receive any specific funds.

### Data availability

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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