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# Kinetics and Isotherms Studies of Safranin Adsorption onto Two Surfaces Prepared from Orange Peels

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## Abstract

Removal of safranin dye from aqueous solutions using two adsorbents prepared from orange peels (Orange peels powder DP-OP and hot activated carbon HAC-OP) has been investigated under different experimental conditions. The effect of pH, contact time, adsorbent mass, and initial concentration of studied dye on the adsorption process has been studied using batch experiments procedure. The concentration of remaining dye in the solution has been estimated by means of UV-VIS molecular absorption spectrometry. The highest removal of safranin has been achieved at pH (8-9) with a maximum removal efficiency of (96 and 97%) of dye after (30 and 50 min) of agitation time for DP-OP and HAC-OP, respectively. Freundlich isotherm model represents the best fit for the experimental data; whereas the highest adsorption capacities were 55.5 $mg.g^{-1}$ and 1.24 $mg.g^{-1}$ for DP-OP and HAC-OP, respectively. The pseudo-second-order rate equation fitted well kinetic profiles for both adsorbents.

***Keywords:*** *Safranin, Dye Adsorption, Kinetics, Isotherms*

# 1. Introduction

Environmental pollution is the primary concern in several countries [1,2]. However, water pollution is the main environmental pollution in various countries [3,4]. Water pollution harms human health [5,6]. The degradation of organic particulates in wastewater by oxidative photocatalysis has gained much attention [7,8]. The textile industry considers one of the largest consumers of synthetic dyes. Therefore, it is mainly responsible for the generation and disposal of effluents, which can cause severe health and environmental problems and negative visual impact due to the coloration of the water [9,10]. The textile and leather industry and some medicines and cosmetics consume many dyes, which are discharged into sewage streams, rivers, and water bodies, which leads to the penetration of these dyes into the soil [11,12]. To protect the environment and humans from the negative impact of these dyes, which contain toxic and carcinogenic substances, scientists are looking for ways to remove the concentration of these dyes to the lowest permissible concentration, as many research and studies have been done recently to treat dyes in water and soil [13-15]. Where the removal of unwanted dyes from water and soil is considered one of the environmental problems, which led to the search for various methods of wastewater treatment, among which we can mention biodegradation, chemical oxidation, foam flotation, electrolysis, absorption, chemical coagulation, and photocatalysis [11,16-26]. The most effective way to remove these dyes from wastewater is adsorption, which is considered one of the most effective techniques because of its low cost and simplicity of design and operation, which is successfully used to remove dyes from wastewater [20,27]. Adsorption of organic pollutants at the solid/water interface is an efficient and economically cheap method to control the extent of water pollution [28,29]. The most common adsorbent is activated carbon [30-33], but it is relatively expensive and difficult to regenerate. Agricultural waste and residues have proven their effectiveness in removing dyes, which are absorbent materials in adsorption processes, which led to many researchers using this waste because of its abundance and low cost. [20]. The object of the present investigations has been to evaluate the efficiency of removal of Safranin dye using dried powder and hot activated carbon prepared from orange peels (DP-OP and HAC-OP).

# 2. Materials and methods

### *1.1. **Preparation of Adsorbent***

The orange peels, which had been used to prepare two adsorbents in this study; Dry orange Peels Powder (DP-OP) and Hot Activated Carbon (HAC-OP), were obtained from a local agricultural field (Msallata City-Libya). The dry absorbent material (DP-OP) was cleaned and thoroughly washed with double distilled water to remove unwanted particles from the surface, then dried in sunlight. It was then dried in a hot air oven at 70˚C for about 24 h. After drying, the hulls were ground in an electric grinder and then sieved to the desired particle size as <125 μm. Finally, the material was stored in separate storage bottles for further experiments. For hot activated carbon (HAC-OP) preparation, the dried material was subjected to pyrolysis at 550$^{o}$ C in a furnace for two hs. [34]. The formed biochar was removed, ground into powder, sieved, rinsed with double distilled water, and finally dried at 60$^{o}$ C. This material was designed as hot activated carbon (HAC-OP).

### *1.2. **Adsorption Procedure***

absorbent doses were optimized for maximum removal of safranin. A 50 mL of safranin solution was taken in the 150 mL of flasks with known amount adsorbent dose (0.1–1.2 g for dried material and 0.25-3.00 g for hot activated carbon), safranin concentration (3–150 mg/L for dried material and 3-25 mg/L for hot activated carbon), the pH (3−10) was set using 0.1 N HCl/NaOH and placed in a shaker for different time intervals (0–60 min) at 150 rpm. At the end of the particular time, the safranin solution was filtered, and the filtrate was analyzed at 520 nm wavelength

using a UV- spectrophotometer to determine the residual amount. The adsorption capacity ($Q_e$) and percentage removal (%R) of safranin onto the adsorbents were calculated as follows [35-37]:

$$Q_e = \frac{(C_o - C_e) x V}{M} \qquad (1)$$

$$\%R = \frac{(C_o - C_e)}{C_o} x 100 \qquad (2)$$

Where $C_o$ (mg/l), $C_e$ (mg/l), V (l), and M (g) represent the initial concentration of safranin, equilibrium concentration of safranin, volume of aqueous media, and mass of the dry adsorbent, respectively.

### *1.3. **Instrumental Analyses***

The equilibrium safranin concentrations were determined by a UV-Vis spectrophotometer from JENWAY (6305). The wavelength used was 520 nm, and calibration was made using safranin concentrations of 2.0 to 16.0 mg/L with an $R^2$ of 0.999. The pH of the solution was determined by a pH meter from JEMWAY (3505). The pH was continuously controlled and adjusted using HCl/NaOH (0.1 M).

### *1.4. **Reagents***

The dye Safranin-T ($C_{16}H_{11}N_2O_4SNa$) was obtained from Merck and used without further purification. Its 1000 ppm stock solution was prepared in double-distilled water. Double distilled water was used for necessary dilutions to prepare various solutions at desired concentrations from the stock solution. Hydrochloric acid, Sodium hydroxide, and phosphoric acid were also obtained from Merck.

## 3. Results and discussion

### *3.1. Effect of pH on Adsorption Process*

Safranin molecules ($pK_a$=5.8) can be reformed to be fully protonated in highly acidic solutions and deprotonated at high pH [20]. The dye molecules show a high positive charge density at a lower pH (pH<3) [10]. The pH effect was evaluated by altering the pH between 3-10 to obtain the highest degree of safranin removal. The dried material demonstrated high adsorption at pH higher than 6, whereas the high adsorption was at pH higher than 4 in the hot activated material. As seen in Figure 1, the pH effect revealed different behavior for the dried and the hot activated materials; when the pH of the solution increased from 3 to 6, the amount of adsorbed sharply increased. Upon a further increase in pH from 5 to 10, the adsorption slightly increased and reached its maximum value at pH 9 in the dried material. At pH > 6, the surface charge is negative, favoring the safranin cations adsorption, while the presence of an excess of $H^+$ at acidic pH (pH<3) can compete with the dye cations for adsorption sites resulting in a lower percentage of dye removal. The hot activated material shows no noticeable effect on the safranin removal efficiency by increasing pH from 4 to 10. The negligible difference in adsorption was obtained when pH was changed in the mentioned range. Therefore, both materials were conducted at pH between 8-9.

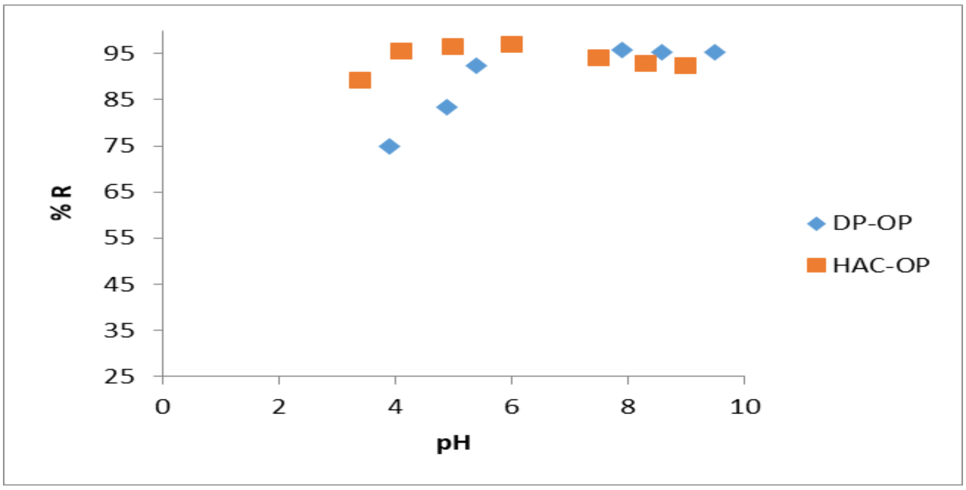


**Figure 1.** Effect of pH on safranin adsorption onto dried powder and hot activated carbon prepared from orange peels.

### *3.2. Effect of Adsorbent Dose on Adsorption Process*

The adsorbent dose is a vital parameter in adsorption studies. Optimizes the cost-effective adsorbents dosage by determining their adsorption capacity for a given initial concentration [36]. The dye adsorption was carried out by varying the adsorbent dose from 0.1 to 1.2 g for dried material and from 0.25 to 3.00 g for hot activated carbon at the optimized pH. The amounts of dye adsorbed by varying the dosage of adsorbents are presented in Figure 2. Figure 2 reveals that the adsorption capacity of safranin decreased with an increase in the amount of adsorbent and reached its maximum capacity at 0.10 and 0.25 g for dried material and hot activated carbon, respectively. The 0.10 g and 0.25 g (DP-OP and HAC-OP) of dose per 50 mL of the solution were taken as the optimized dose for further experiments.

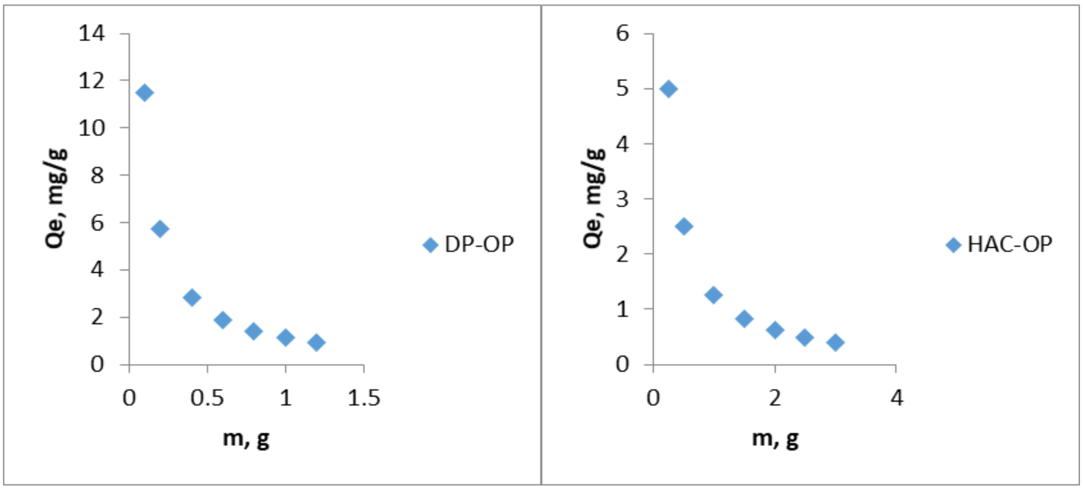


**Figure 2.** Effect of adsorbent dose on safranin adsorption onto dried powder and hot activated carbon prepared from orange peels

### *3.3. Effect of Time on Adsorption Process*

Completing the process within a short time is extremely important for industrial applications in terms of cost-effectiveness and efficiency. Contact time plays a vital role in the adsorption studies as it suggests the necessary required time of contact for deriving maximum adsorption of the dye at the adsorbent surface [Chandane and Singh, 2014; 36]. Figure 3 presents a graph for the variation in adsorption concerning time.

The adsorption efficiency increased gradually at first with the increase in contact time. The equilibrium was reached within 30 and 50 min (for DP-OP and HAC-OP, respectively). After attaining equilibrium, the adsorption efficiency mainly remained constant, which indicated that the dye molecules' rate of adsorption and desorption processes became equal after equilibrium was reached. The physical adsorption governed the adsorption process, and the dye molecules tend to revert to the solution after attaining equilibrium. The aggregation of dye molecules around the adsorbent particles with time also resisted further adsorption of dye molecules [38]

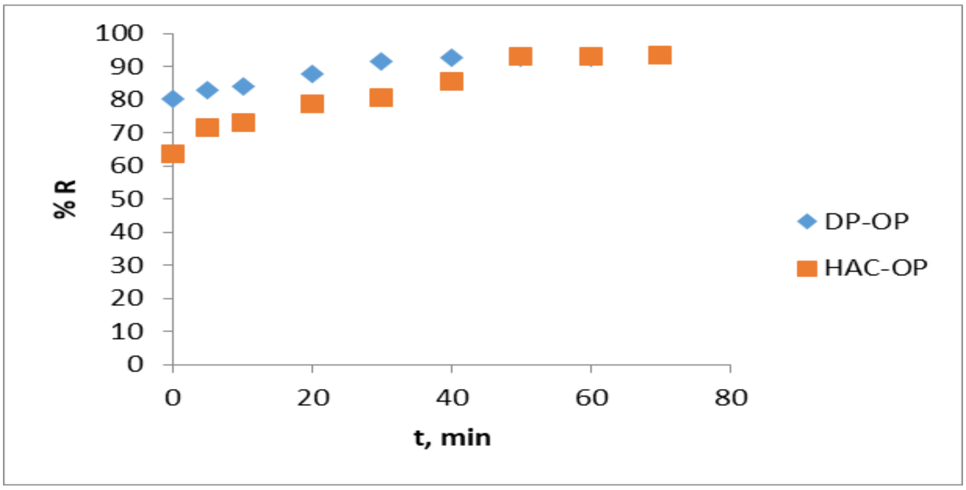


**Figure 3.** Effect of time on safranin adsorption onto dried powder and hot activated carbon prepared from orange peels

Adsorption kinetics is an important step to investigate the adsorption process. Kinetics of adsorption of safranin onto the two adsorbents (DP-OP and HAC-OP) in an aqueous medium was carried out at 25°C. The adsorption kinetics of safranin was investigated by pseudo-first order and pseudo-second-order kinetic models.

The pseudo-first-order kinetic model expressed as Lagergren first-order rate equation (Eq. (3)) [31-35].

$$\text{Log}\,(Q_e - Q_t) = \text{Log}\,Q_e - K_1\,t \qquad (3)$$

Where $K_1$ is the first-order rate constant, $Q_e$ and $Q_t$ denote the amount adsorbed at equilibrium and time t, respectively. $K_1$ ($min^{-1}$) is the pseudo-first-order rate constant. It is generally used for the periods before reaching equilibrium. A plot of Log ($Q_e - Q_t$) vs. t is a line (Figure 4), and $Q_e$ and $K_1$ are obtained from the intercept and slope of the graph, respectively.

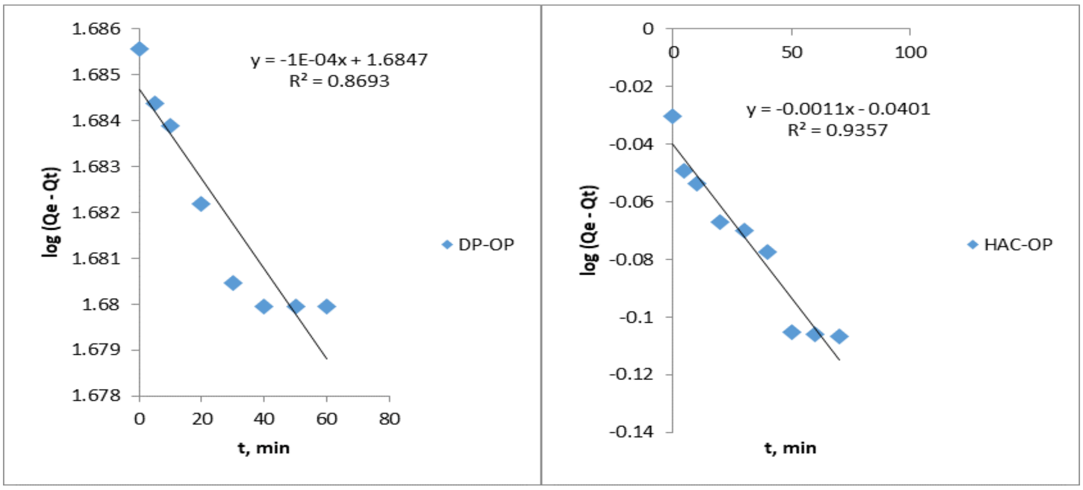


**Figure 4.** Pseudo first order kinetic plots for the removal of safranin by adsorption onto DP-OP and HAC-OP.

The pseudo-second-order kinetic model is another model used for the adsorption of kinetic data. This model is compatible with the mechanism of rate-controlling step throughout the adsorption, contrary to the pseudo-first-order kinetic model [36]. The pseudo-second-order kinetic model is given by the following equation [31-35]:

$$\frac{t}{Q_t} = \frac{1}{K_2 Q_e^2} + \frac{1}{Q_e} t \qquad (4)$$

where $K_2$ ($g.mg^{-1}.min^{-1}$) is the second-order rate constant. The plot of $t/Q_t$ vs. t is a line (Figure 5), and $Q_e$ and $K_2$ are obtained from the slope and intercept of the graph, respectively.

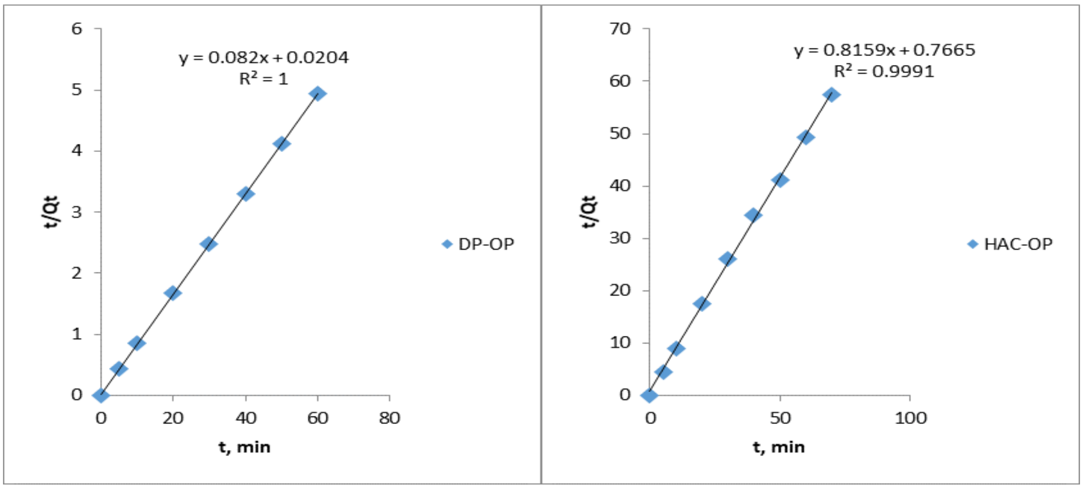


**Figure 5.** Pseudo second order kinetic plots for the removal of safranin by adsorption onto DP-OP and HAC-OP.

Graphs of Log ($Q_e - Q_t$) vs. t for the first-order kinetic model (Figure 4) and graphs of $t/Q_t$ vs. t were plotted for second-order kinetic model (Figure 5) and the constants obtained at 25° C are given in Table 1.

***Table 1. Rate constants for adsorption of safranin onto DP-OP and HAC-OP***

| **Pseudo First Order** | **$K_1$ ($min^{-1}$)** | **$Q_e$ ($mg.g^{-1}$)** | **$R^2$** |
|---|---|---|---|
| **DP-OP** | 0.0001 | 48.38 | 0.8693 |
| **HAC-OP** | 0.0011 | 0.91 | 0.9357 |
| **Pseudo Second Order** | $K_2$ ($g.mg^{-1}.min^{-1}$) | $Q_e$ ($mg.g^{-1}$) | $R^2$ |
| **DP-OP** | 0.3293 | 12.20 | 1.0000 |
| **HAC-OP** | 0.8623 | 1.23 | 0.9991 |

As can be found from Table 1, the pseudo-second-order kinetic model represented the time-dependent function of equilibrium better than the pseudo-first-order kinetic model when $R^2$ values are considered. Therefore, the kinetic adsorption accords well with the pseudo-second-order model.

### *3.4. Effect of Initial Concentration on adsorption process and Isotherms*

Initial adsorbate concentration is the main driving force during the adsorption process and significantly impacts the removal process, which was studied in 3–150 mg/L for DP-OP and 3-25 mg/L for HAC-OP. It was noted that the adsorption capacity of DP-OP and HAC-OP was increased with concentration (Figure 6). At an initial concentration of

3 mg/L, DP-OP and HAC-OP adsorption capacities were 0.79 and 0.06 (mg/g), respectively, and adsorption capacities of these adsorbents increased to 11.41 and 1.18 (mg/g), respectively at the initial concentration of 25 mg/L. It is due to the reason that at the low initial concentration of safranin, the number of empty binding sites was also higher, and percentage removal was high [3,4,39]

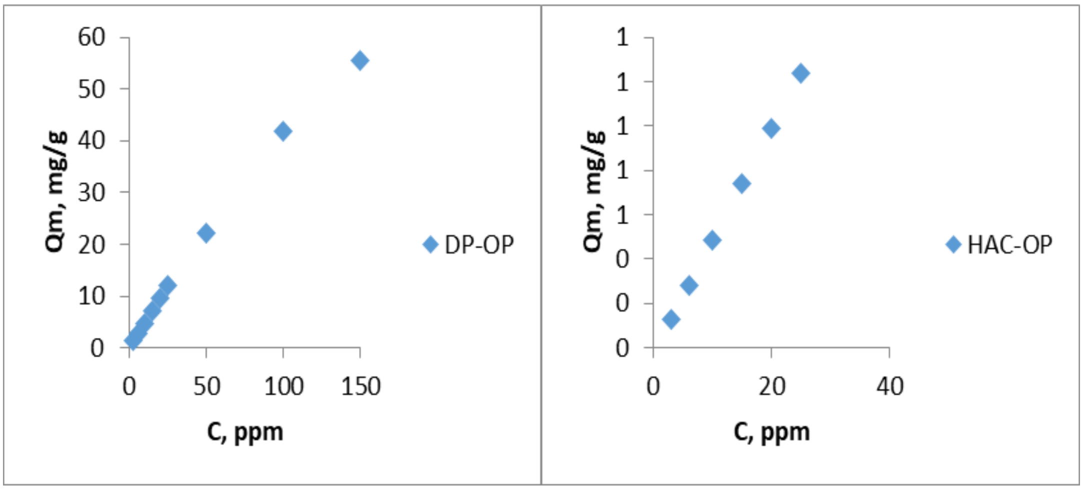


**Figure 6.** Effect of initial concentration on safranin adsorption onto dried powder and hot activated carbon prepared from orange peels

**Isotherms**

The adsorption isotherms of safranin dye onto orange peels powder (DP-OP) and hot activated carbon prepared from orange peels (HAC-OP) were investigated, and the data were analyzed by Langmuir and Freundlich, Temkin and Dubinin–Radushkevich equations.

Theoretical Langmuir isotherm describes monolayer adsorption onto a homogeneous adsorbent surface [35,40]. The linear equation of Langmuir is defined in Eq. (5):

$$\frac{C_e}{Q_e} = \frac{1}{b\,Q_m} + \frac{C_e}{Q_m} \qquad (5)$$

where Ce is the equilibrium concentration of adsorbate (mg/L), Qe is the amount adsorbed in equilibrium (mg/g), b is the Langmuir constant ($L.mg^{-1}$), and Qm is the maximum amount adsorbed of dye in mg/g.

The Freundlich isotherm model is based on a heterogeneous surface with unequal energies [35,41,42]. Its empirical equation is defined in Eq. (6):

$$\text{Log } Q_e = \text{Log } K_f + \frac{1}{n}\text{Log } C_e \qquad (6)$$

where n is the marker of the desirability extent of the adsorption process and $K_f$ is the amount of capacity of adsorbent adsorption in (mg/g $(L/mg)^{1/n}$) [43].

In order to estimate the porosity and free energy used for adsorption, the Dubinin-Radushkevich model was investigated [44]. The linear equation of this model is defined in Eq. (7):

$$\text{Log } Q_e = \text{Log } Q_m - \beta\, \varepsilon^2 \qquad (7)$$

where $Q_m$ is the maximum amount adsorbed (mg/g), β (constant) porosity factor and ε Polanyi potential that is defined by Eq. (8):

$$\varepsilon = RT\,\text{Log}\,(1 + \frac{1}{C_e}) \qquad (8)$$

where R is the global constant of gas (R=8.3144 $J.mol^{-1}.K^{-1}$) and T is the temperature (in Kelvin).

The Temkin isotherm model shows between adsorbate/adsorbate interactions, and the linear form of Temkin isotherm is given by Eq. (9) [35]:

$$Q_e = B \text{ Log } A + B \text{ Log } C_e \qquad (9)$$

where B corresponds to the heat of sorption ($KJ.mol^{-1}$) and A is the equilibrium binding constant ($L.g^{-1}$) [35,45].

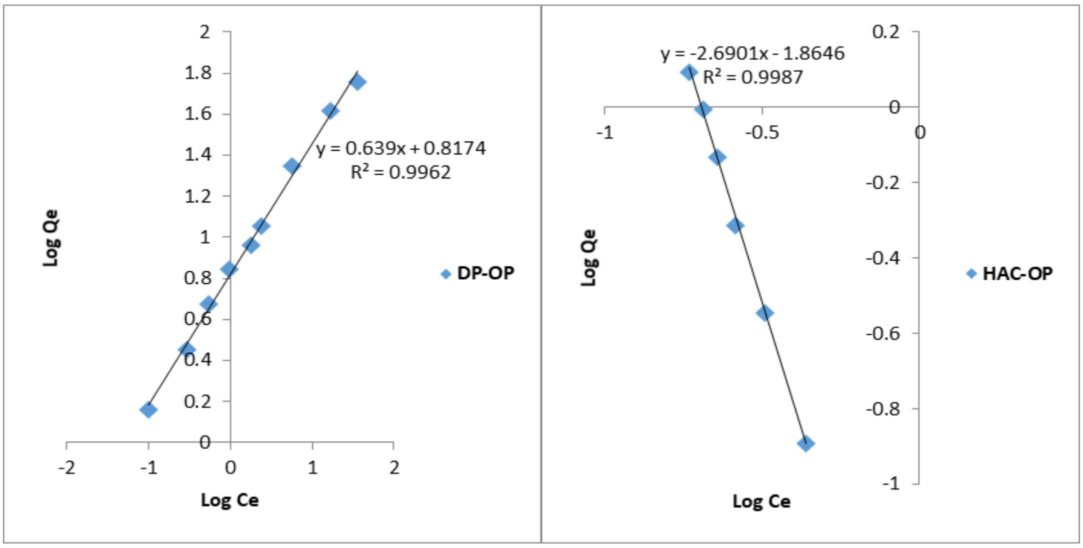


**Figure 7.** Freundlich Isotherm for adsorption of safranin onto DP-OP and HAC-OP

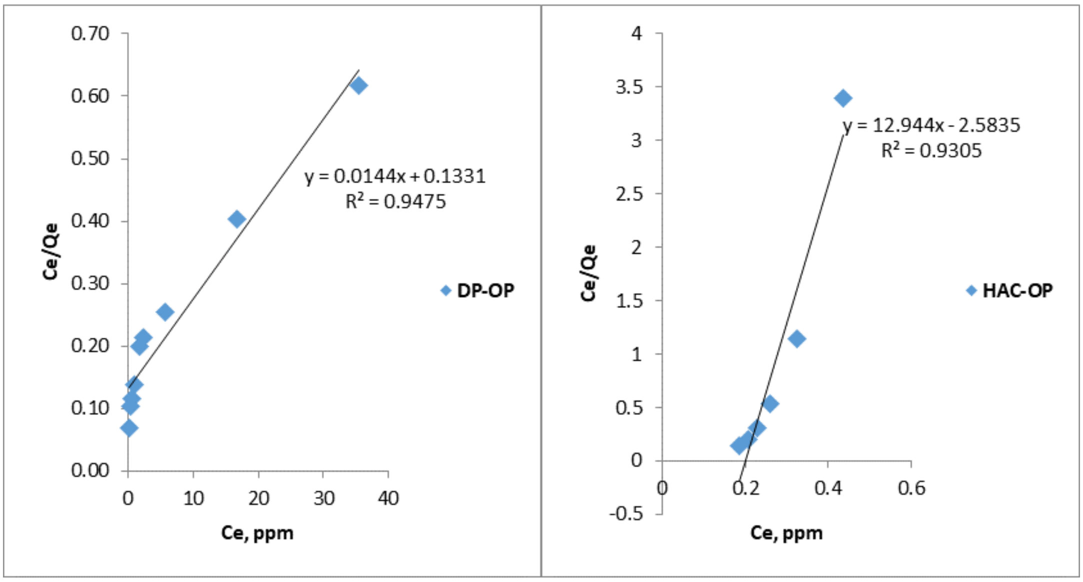


**Figure 8.** Langmuir Isotherm for adsorption of safranin onto DP-OP and HAC-OP

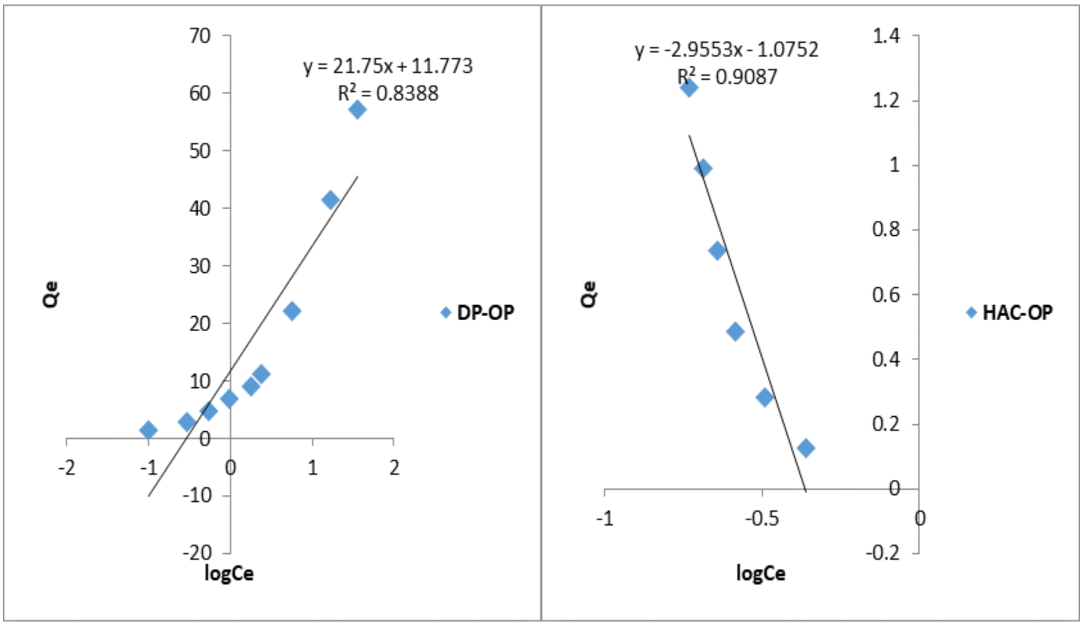


**Figure 9.** Temkin Isotherm for adsorption of safranin onto DP-OP and HAC-OP

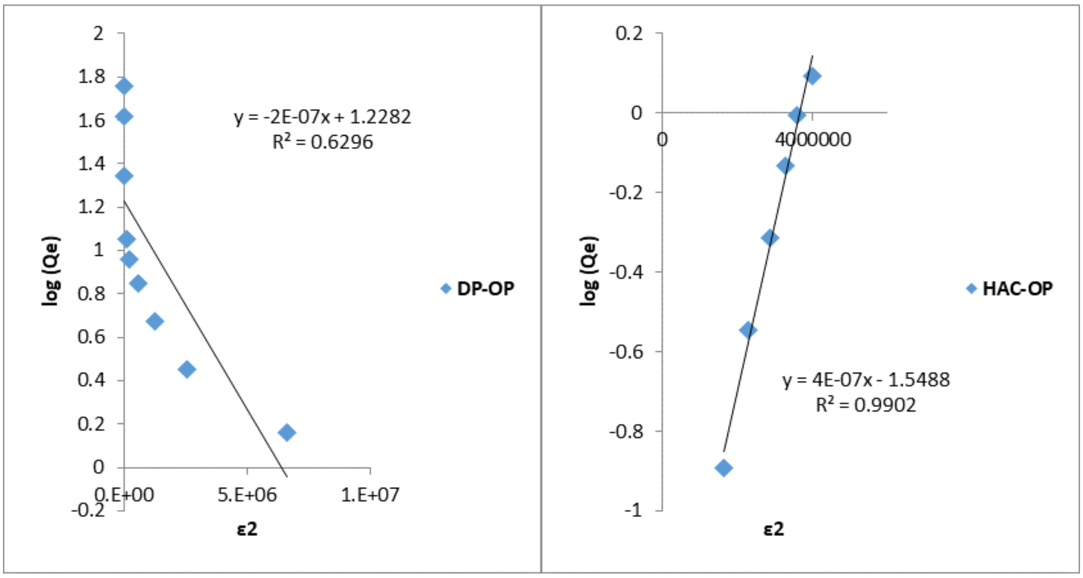


**Figure 10.** Dubinin–Radushkevich Isotherm for adsorption of safranin onto DP-OP and HAC-OP

According to the isothermal graphs (Figures: 7,8,9,10) and the results obtained from Table 2, Freundlich isotherm shows the best interpretation of the safranin adsorption using orange peels powder and hot activated carbon prepared from orange peels. Therefore, the results indicate that the adsorbent level is non-homogeneous or heterogeneous and has unequal and non-uniform energies. The parameters 1/n and n in the Freundlich equation can determine the desirability of the adsorption process. If n=1 adsorption is linear; if $n < 1$, adsorption is a chemical process; finally, if $n > 1$, then adsorption is a physical process [35]. According to Table 2, the value of $n > 1$ is between 1 and 10 in the case of safranin adsorption onto orange peels powder, which indicates the physical adsorption process. However, the value of $n<1$ in the case of safranin adsorption onto hot activated carbon prepared from orange peels indicates a chemical adsorption process. Table 2. Adsorption isotherm constants for adsorption of safranin onto DP-OP and HAC-OP

*Table 2. Adsorption isotherm constants for adsorption of safranin onto DP-OP and HAC-OP*

| Adsorbent | DP-Op | HAC-OP |
|---|---|---|
| **Freundlich Isotherm** | | |
| **$K_f$ (mg/g (L/mg)$^{1/n}$)** | 6.57 | 0.014 |
| **N** | 1.56 | 0.372 |
| **$R^2$** | 0.9962 | 0.9987 |
| **Langmuir Isotherm** | | |
| **b ($L.mg^{-1}$)** | 0.108 | 5.027 |
| **$Q_m$ ($mg.g^{-1}$)** | 69.44 | 0.077 |
| **$R^2$** | 0.9475 | 0.9305 |
| **Temkin Isotherm** | | |
| **A ($L.g^{-1}$)** | 3.48 | 2.31 |
| **B ($KJ.mol^{-1}$)** | 21.75 | 2.955 |
| **$R^2$** | 0.8388 | 0.9087 |
| **Dubinin–Radushkevich Isotherm** | | |
| **$Q_m$ ($mg.g^{-1}$)** | 18.91 | 0.028 |
| **B** | $2x10^{-7}$ | $4x10^{-7}$ |
| **$R^2$** | 0.6296 | 0.9902 |

## Conclusion

The adsorptive potential of two adsorbents prepared from orange peels for the removal of safranin from aqueous solutions has been examined. Highest removal was achieved at contact time of 30 and 50 min for DP-OP and HAC-OP, respectively; it was found to increase as the initial concentration was increased for both adsorbents. The removal of safranin was maximum at pH of (8-9) and adsorbent dosage of 0.1 g/50 ml. The process of safranin adsorption on the two adsorbents obeyed best to the Freundlich isotherms with regards to their correlation coefficients, $R^2$. The kinetic data of safranin adsorption on the two adsorbents followed the pseudo-second-order model. The experimental study shows that the dry orange peels can give better efficiency for the removal of safranin than the hot activated carbon prepared from orange peels.

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