

## Adsorption of Congo red Dye in Aqueous Solution unto Pumpkin Pod Pretreated with Sodium Thiosulphate

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**ABSTRACT:** The adsorption of Congo red dye from aqueous solution using Sodium thiosulphate-pretreated Pumpkin pod, was studied. The adsorbent was characterized by determining some physicochemical properties as well as the SEM and FTIR spectra. A two-level and four-factor factorial experimental design, with adsorbent dosage (0.1 to 0.3 g), pH (6.5 to 9), initial dye concentration (10 to 50 mg) and contact time (10 to 90 min) as process variables was used. Two common adsorption isotherms were fitted to the adsorption data, while the pseudo-first order and pseudo-second order kinetic models were also tested on the adsorption process. The determined properties, surface morphology and functional groups in the adsorbent, suggest that the pretreated Pumpkin pod could serve as an efficient adsorbent. Percentage dye removal increased, while adsorption capacity decreased with increase in adsorbent dosage. The pH had no effect on both the percentage removal and adsorption capacity. Increasing the initial dye concentration led to a decrease in percentage removal and an increase in adsorption capacity, while both the percentage removal and adsorption capacity increased with increase in contact time. These trends are in agreement with many reported works. The developed model could navigate the design space. A maximum percentage removal of 81.05% was obtained at optimum values of 0.3 g adsorbent dosage, initial dye concentration of 50 mg/l and a contact time of 90 minutes. The mechanism of the dye adsorption followed a heterogeneous poly layer coverage of the dye on the adsorbent surface, while the pseudo-second order kinetic model was observed to describe the kinetics of the process. Pumpkin pod pretreated with Sodium thiosulphate, can be used as a cheap and efficient adsorbent for the removal Congo red dye from aqueous solutions such as Textile industry wastewaters.

**KEYWORDS:** Adsorption, Pumpkin pod, Congo red, Wastewater, Modelling, Kinetics

### 1. INTRODUCTION

Dyes are important raw materials for the textile, leather, cosmetics, paper, printing, plastic, pharmaceuticals and food industries. Dyes exhibit great structural differences, thus they are not uniformly degraded by microbial attack. The discharge of highly colored effluents into natural water bodies is not only aesthetically displeasing, but it also impedes light penetration, thus upsetting biological processes within such water bodies (Coruh *et al.*, 2015). Dyes can be degraded under anaerobic conditions, forming aromatic amines which are colourless but may be toxic and carcinogenic (Yaneva and Georgieva, 2012). Consequently, there is huge amount of textile wastewater generation from industries as a result of textile wet processes.

The wastewater is discharged into the environment after ineffective conventional treatment techniques. Recently, approximate 12% of synthetic textile dyes used each year is lost during manufacturing and processing operations, and 20% of these dyes enter the environment through effluents that result from the treatment of residual industrial waters (Hema and Arivoli, 2008 and Hakan *et al.*, 2008). The discharge of this ineffectively treated wastewater gives rise to an alarming environmental concern related to water pollution. Congo red dye (Figure 1), 1- Naphthalenesulfonic acid, 3, 3'-(4, 4' biphenylene bis (azo) bis 4-amino, di sodium salt, is a benzenedene based dye, Known to metabolize to benzenedene, a known human carcinogen and can also cause allergic dermatitis and skin irritation.

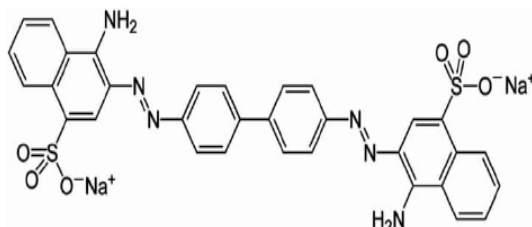


Figure 1: Chemical structure of Congo red dye (Roshan, 2015).

A possible solution can be the use of adsorbents to treat dye wastewaters. Several effective adsorbents for textile wastewater treatment have previously been investigated. Exploring the use of effective and low cost adsorbents from plant waste may contribute to environmental sustainability and offer benefits for future commercial applications (Gupta *et al.*, 2011a, 2011b). Investigations have revealed the feasibility of using inexpensive agricultural wastes as carbonaceous precursors for the removal of dyes from water and wastewater (Sekaran *et al.*, 1995).

Pumpkin Pod is biodegradable, and contains phytochemical constituents such as, tannins, flavonoid, steroid, terpenoids, saponin, carbohydrates, amino acids and proteins (Akwukwaegbu, 2016, Ogbonnaya and Uwadia, 2016). These organic compounds contain polar functional groups with N, S, O atoms as well as conjugated double bonds or aromatic rings in their molecular structure, which serve as the major adsorption centres. This study investigated the performance of pumpkin pod pretreated with Sodium thiosulphate, in removing Congo red dye from aqueous solutions.

## 2. METHODOLOGY

### 2.1 Materials

Materials used include; analar grade Congo red dye, Raw Pumpkin pod, Sodium hydroxide (NaOH) and Hydrochloric Acid (HCl) to adjust the pH of the sample, De-ionized water used in preparing stock solutions and Sodium thiosulphate (Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>) used in pretreating the adsorbent.

### 2.2 Adsorbent Preparation, Activation and Characterization

The pumpkin pod was washed and dried at 65 °C for 24hours, before grinding into powder. The powdered pumpkin pod was soaked in Sodium thiosulphate (Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>) for 24 hours, using a weight ratio of 1:2w/v of the powdered pumpkin pod to the Sodium thiosulphate. It was then washed with 0.1M aqueous NaOH solution followed by distilled water until the pH became neutral. The pumpkin pod was then dried at 65 °C for 36 hours, before it was grinded to 150 µm sieve size. To characterize the adsorbent, the moisture content, volatile matter, ash content, pH, fixed carbon content and bulk density were determined. The Fourier Transform Infrared (FT-IR) spectra as well as the scanning electron micrograph of the pretreated pumpkin pod were also determined to identify the functional groups on the surface of the adsorbent as well as the nature of the surface.

### 2.3 Adsorbate Preparation

The Congo red dye used was laboratory grade. The solution was prepared with de-ionized water. 100 mg of Congo red was added to de-ionized water to prepare a standard solution of 100 mg/l. Further dilute solutions were prepared by successive dilutions with de-ionized water.

### 2.4 Experimental Design

Batch adsorption experiments were carried out using two-level four-factor factorial analysis. The experimental parameters chosen as independent variables include adsorbent dose (0.1-0.3 g), pH of the solution (6.5 - 9), initial dye concentration (10 - 50 mg/L), and Contact time (10 – 90min). The binding ratio was determined as the average of two parallel experiments. The results were analyzed using Design Expert 11 software to obtain the effects, coefficients, standard deviation of coefficients, and other statistical plots. The mathematical model utilized to find the predicted optimum response within the studied experimental conditions was given as a coded model in the form expressed in Equation 1.

$$y = \beta_0 + \sum_{i=1}^k \beta_i x_i + \sum_{1 \leq i < j}^k \beta_{ij} x_i x_j + \varepsilon$$

Equation 1

Where y is the response, x<sub>i</sub> and x<sub>j</sub> are coded variables, β's are regression coefficients, and ε is a random error, (Ponnusami, *et al.*, 2007). The uncoded variables were converted to coded variables using Equation 2.

$$x = \frac{X - [X_{max} + X_{min}]/2}{[X_{max} - X_{min}]/2}$$

Equation 2

Where x is the coded variable, X is natural variable, and X<sub>max</sub> and X<sub>min</sub> are the maximum and minimum values of the natural variable.

### 2.5 Analytical procedures

The amount of Congo red adsorbed (q<sub>t</sub>) at a time t (mg /g) were calculated using Equation 3. The adsorption capacity (q<sub>e</sub>), of the adsorbent for each concentration of Congo red ions at equilibrium were calculated using Equation.4, while the removal efficiency, i.e percentage of Congo red removed, was calculated for each run using Equation 5. Where C<sub>t</sub> (mg/L) is the liquid phase concentrations of Congo red at any time, C<sub>0</sub> (mg/L) corresponds to the initial concentration of Congo red, V (L) is the volume of the solution and W (g) is the mass of the adsorbent.

$$q_t = \frac{(C_0 - C_e)V}{W}$$

Equation 3

$$q_e = \frac{(C_0 - C_e)V}{W}$$

Equation 4

$$(\%) \text{Removal} = \frac{(C_0 - C_e)}{C_0} \times 100$$

Equation 5

### 2.6 Adsorption Isotherms

The Langmuir and the Freundlich adsorption isotherms were tested on the adsorption data. The Langmuir isotherm assumes

that all the available surface for adsorption on the adsorbent are homogeneous and that only a monolayer of the adsorbate is formed at the adsorption site, and can be represented by Equation 6.

$$\frac{C_e}{q_e} = \left(\frac{1}{q_m K_L}\right) + \left(\frac{C_e}{q_m}\right) \quad \text{Equation 6}$$

The Freundlich isotherm is applicable to adsorption on heterogeneous surfaces. It describes the ratio of the quantity of adsorbate adsorbed unto a given adsorbent to the concentration

of the adsorbate in solution. A linear form of it, is presented in Equation 7.

$$\ln(q_e) = \ln(K_f) + \frac{1}{n} \ln(C_e) \quad \text{Equation 7}$$

Where,  $q_e$  is amount of adsorbate per gram of adsorbent at equilibrium,  $C_e$  is concentration at equilibrium,  $K_L$  and  $q_m$  are Langmuir constants, while  $K_f$  and  $n$  are Freundlich constants. These isotherms were evaluated on the adsorption process using Table 1 (Alamrani and Al-Aoh, 2021).

**Table 1: Evaluation of constants in isotherm models**

Model	Linear form	Plot	Constants
Langmuir	$\frac{C_e}{q_e} = \left(\frac{1}{q_m K_L}\right) + \left(\frac{C_e}{q_m}\right)$	$\left(\frac{C_e}{q_e}\right) \text{ vs } C_e$	$q_m = (\text{slope})^{-1}$ $K_L = \frac{\text{slope}}{\text{intercept}}$
Freundlich	$\ln(q_e) = \ln(K_f) + \frac{1}{n} \ln(C_e)$	$\ln(q_e) \text{ vs } \ln(C_e)$	$K_f = \exp(\text{intercept})$ $n = (\text{slope})^{-1}$

**2.7 Kinetic Studies**

The pseudo-first order and pseudo-second order kinetic models were investigated. The linear form of the pseudo-first order model shown in Equation 8 was used, and the parameters ( $q_e$  and  $K_f$ ) were determined from the intercept and slope of the plot of  $\log(q_e - q_t)$  versus time, respectively. For the pseudo second order model, the linear form in Equation 9 was used, and the parameters ( $k$  and  $q_e$ ) were determined from the plot of  $t/q_t$  against time.

$$\log(q_e - q_t) = \log q_e - \frac{K_f}{2.303} t \quad \text{Equation 8}$$

$$\frac{t}{q_t} = \frac{1}{k q_e^2} + \frac{t}{q_e} \quad \text{Equation 9}$$

**3. RESULTS AND DISCUSSION**

**3.1 Physicochemical properties of Pumpkin Pod**

The properties of the adsorbent investigated are given in Table 3.1, which shows that the adsorbent has low moisture and ash contents, and that the particle density is relatively small, indicating that the pretreated Pumpkin pod should be an excellent material for use as an adsorbent for wastewater treatment.

**Table 2: Proximate analysis of pumpkin pod**

Parameters	Values
Moisture Content (%)	7.90
Volatile Matter (%)	68.50
Ash Content (%)	5.20
Carbon (%)	48.91
Mesh Size (µm)	150
pH	6.80
Bulk Density (g/mL)	1.8

**3.2 Fourier Transforms Infrared Characterization**

Figure 2 illustrates the FTIR spectra of Pumpkin pod powder which determines the types and intensities of the surface functional groups. From the spectra, a wide absorption band is observed at 3000–3600  $\text{cm}^{-1}$  with a maximum at about 3290  $\text{cm}^{-1}$ , this is assigned to O–H stretching vibrations of hydrogen

bonded hydroxyl groups. The bands at 2930 and 2860  $\text{cm}^{-1}$ , are assigned to asymmetric C-H and symmetric C-H bonds, respectively, present in alkyl groups such as methyl and methylene groups, (Yağmur *et.al.*, 2008). Stretching absorption band at 1740  $\text{cm}^{-1}$  is assigned to carbonyl C=O present in esters,

## “Adsorption of Congo red Dye in Aqueous Solution unto Pumpkin Pod Pretreated with Sodium Thiosulphate”

aldehydes, ketone groups and acetyl derivatives. A strong band is seen at about  $1640\text{ cm}^{-1}$  that may be ascribed to olefinic  $\text{C}=\text{C}$  vibrations in aromatic region for the pumpkin pod (Gurten *et. al.*, 2012). Another strong band at  $1030\text{ cm}^{-1}$  represents  $\text{C}-\text{O}$  stretching vibrations. The band at approximately  $1420\text{ cm}^{-1}$  can be attributed to  $\text{C}-\text{H}$  stretching. While the band at  $1240\text{ cm}^{-1}$

confirms the presence of ester functional group (Vijayalakshmi *et.al.*, 2011). The absorption bands in the region  $450\text{--}750\text{ cm}^{-1}$  indicate the presence of alkynes and alkyl halides. (Mahapatra *et.al.*, 2012).

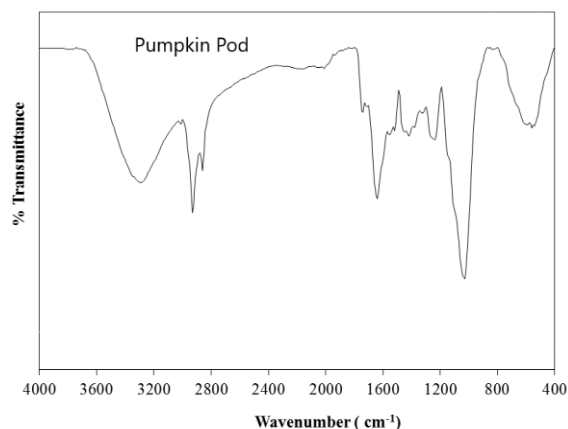


Figure 2: FTIR spectra of pretreated pumpkin pod

### 3.2 Scanning Electron Micrograph Analyses of Pumpkin Pod

The scanning electron micrograph of the Pumpkin pod is shown in Figure 3. The pore diameter and pore volume appear to be

large, while the surface is fairly smooth except for some occasional cracks. Therefore, the pretreated pumpkin powder has a high specific surface area suitable for adsorption.

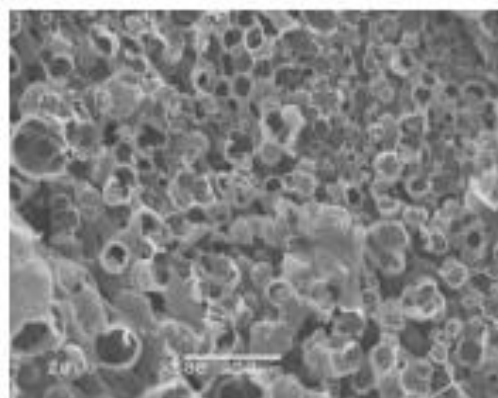


Figure 3: Scanning electron micrograph of pumpkin pod

### 3.4 Results of Adsorption Experiments

#### 3.4.1 Effect of variables

The coded values of the operating variables and the experimental results of the adsorption capacity and percentage

adsorption of Congo red (CR) using Pumpkin pod (PP) are presented in Table 3. The effects of the variables on the adsorption capacity and percentage CR removal are presented in Figures 4 to 11.

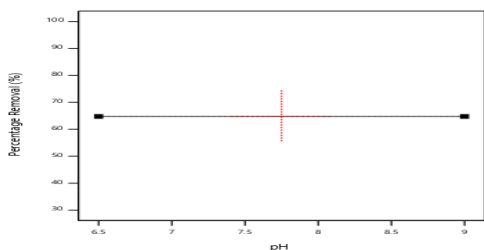
**Table 3: The two-level, four-factor experimental result of adsorption of Congo red using Pumpkin pod**

Std	Run	A:Adsorbent	B:pH	C:Initial Dye Concentration	D:Contact Time	Percentage Removal	Adsorption Capacity
		G		mg/L	Min	%	mg/g
5	1	0.1	6.5	50	10	41.3008	5.16259
13	2	0.1	6.5	50	90	46.3383	5.79229
12	3	0.3	9	10	90	94.8496	0.790414
4	4	0.3	9	10	10	60.6391	0.505326
1	5	0.1	6.5	10	10	71.1654	1.77914
16	6	0.3	9	50	90	83.7068	3.48778
3	7	0.1	9	10	10	59.1353	1.47838
14	8	0.3	6.5	50	90	81.5263	3.39693
11	9	0.1	9	10	90	66.6541	1.66635
6	10	0.3	6.5	50	10	67.8421	2.82675
8	11	0.3	9	50	10	67.2406	2.80169
2	12	0.3	6.5	10	10	64.0226	0.533521
9	13	0.1	6.5	10	90	79.812	1.9953
10	14	0.3	6.5	10	90	67.406	0.561717
7	15	0.1	9	50	10	39.4211	4.92763
15	16	0.1	9	50	90	45.2857	5.66071

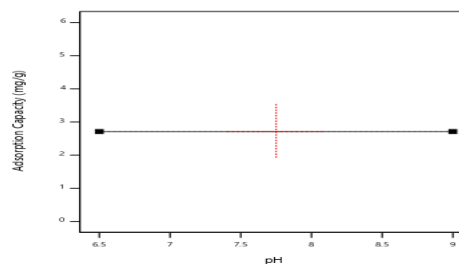
From Figures 4 and 5, both the percentage removal and the adsorption capacity of CR were not affected by change in pH within the range investigated (6.5 to 9). The percentage removal increased with adsorbent dosage, while the adsorption capacity decreased with adsorbent dosage (Figures 6 and 7 respectively). Increasing the adsorbent dosage provides larger adsorbent surface area and by extension, more active sites for adsorption (Alamrani and Al-Aoh (2021), Kumar and Bilal (2018), Shasha *et al.* (2015)).

For the initial dye concentration, Figures 8 and 9, respectively, show that the percentage removal decreased with increase in

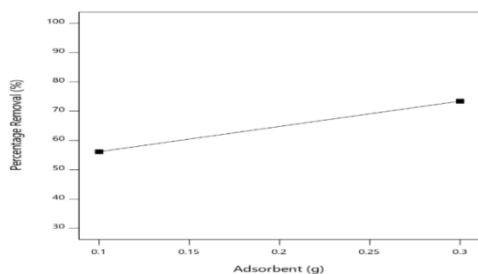
initial dye concentration, while the adsorption capacity increased with increase in the initial dye concentration. A reduction in dye removal with increase in initial dye concentration had been reported by Oboh and Antia (2016), for the adsorption of Congo red dye on Copper (II) oxide nanoparticles. Both percentage removal and adsorption capacity increased with increase in contact time (Figures 10 and 11 respectively). Several authors have reported the same trend for the adsorption of Congo red dye on other adsorbents (Alamrani and Al-Aoh (2021), Jabar *et al.* (2020), Kumar and Bilal (2018), Enenebeaku *et al.* (2016), Ibrahim *et al.* (2013)).



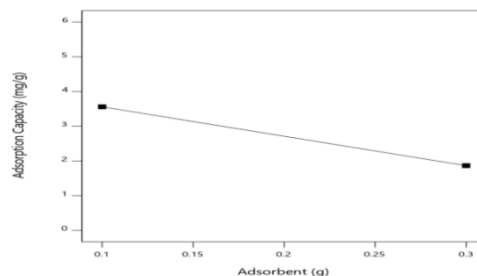
**Figure 4: Effect of pH on percentage capacity**



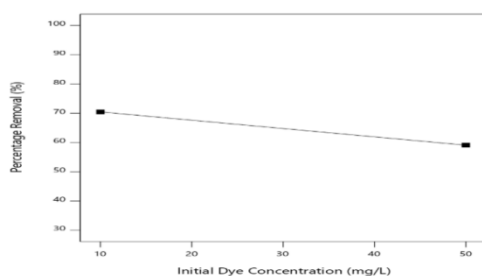
**Figure 5: Effect of pH on adsorption removal of CR**



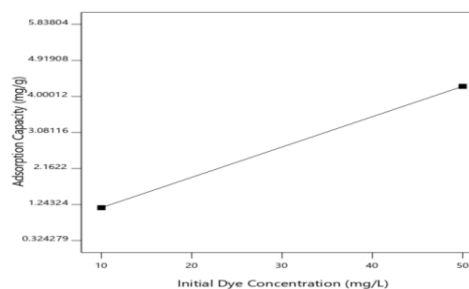
**Figure 6: Effect of adsorbent dosage on Percentage removal of CR**



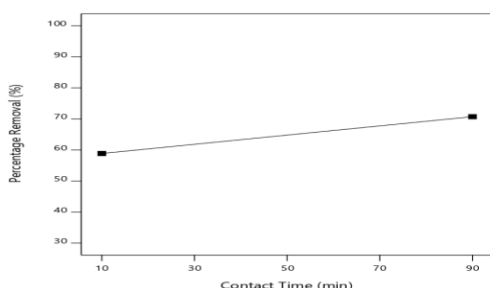
**Figure 7: Effect of adsorbent dosage on adsorption capacity**



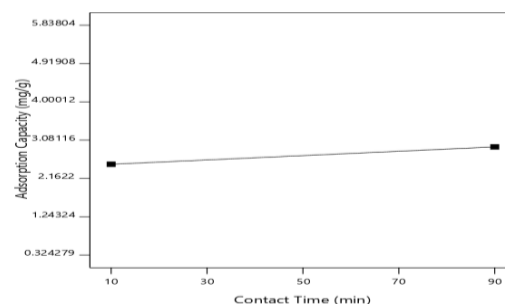
**Figure 8: Effect of initial dye conc. on Percentage removal of CR**



**Figure 9: Effect of initial dye conc. on adsorption capacity**



**Figure 10: Effect of contact time on Percentage removal of CR**



**Figure 11: Effect of contact time on adsorption capacity**

### 3.4.2 Modeling and Optimization

The derived model is presented in terms of the coded factors in Equation 10. The model can be used to make predictions about the response for given levels of each factor, while a comparison of the factor coefficients is useful for identifying the relative impact of the factors. It can be seen from the model that Adsorbent dosage had the most effect on the percentage removal of congo red, followed by contact time and initial dye concentration respectively. Adsorbent dosage and contact time had positive effects, while the initial dye concentration had a negative effect on percentage removal.

$$\% \text{Removal} = 64.77 + 8.63A - 5.69C + 5.93D + 7.36AC$$

Equation 10

**Table 4: Analysis of Variance (ANOVA) for CR removal**

<b>Std. Dev.</b>	<b>8.12</b>	<b>R<sup>2</sup></b>	<b>0.8264</b>
<b>Mean</b>	64.77	Adjusted R <sup>2</sup>	0.7396
<b>C.V. %</b>	12.54	Predicted R <sup>2</sup>	0.5556
		Adeq Precision	8.8785

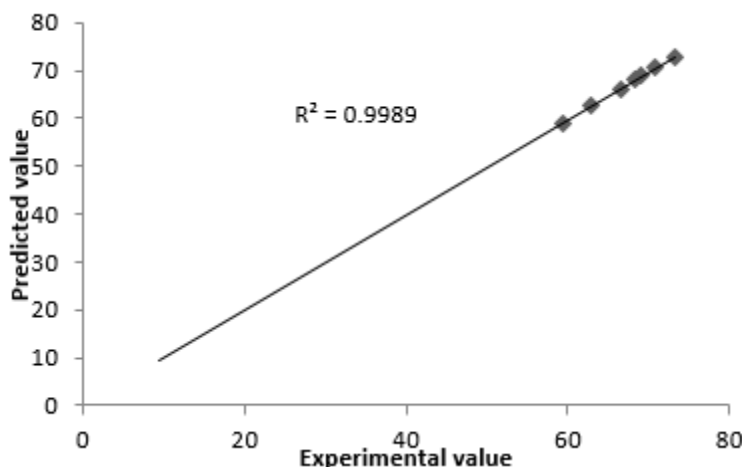
The analysis of variance (ANOVA) for this model is shown in Table 4. P-value less than 0.0500 indicate significant model terms. The P-values for A, C, D and AC are 0.0017, 0.0187, 0.0153 and 0.0046 respectively, indicating that they are significant, while factor B with a P-value of 0.9406 is not significant. The Fit statistics in Table 5, show an R<sup>2</sup> value of 0.8264, which indicates a good correlation between the model and the experimental result. The Predicted R<sup>2</sup> of 0.5556 is in reasonable agreement with the Adjusted R<sup>2</sup> of 0.7396; i.e. the difference is less than 0.2. Adeq Precision measures the signal to noise ratio. A ratio greater than 4 is desirable. The design ratio of 8.8785 indicates an adequate signal. This model can be used to navigate the design space.

**Table 5: Lack of fit test**

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	3139.95	5	627.99	9.52	0.0015
A-Adsorbent	1192.33	1	1192.33	18.08	0.0017
B-pH	0.3848	1	0.3848	0.0058	0.9406
C-Initial Dye Concentration	517.82	1	517.82	7.85	0.0187
D-Contact Time	561.83	1	561.83	8.52	0.0153
AC	867.59	1	867.59	13.15	0.0046
Residual	659.52	10	65.95		
Cor Total	3799.47	15			

The model was also experimentally validated by conducting seven adsorption experiments at various values of the process variables. The plot of the CR percentage removal predicted by the model against the percentage removal obtained from the experiments is shown in Figure 12. Again an R-square value of 0.9993, indicates that the model is able to predict the

adsorption process. The maximum percentage removal of CR for the adsorption process was found to be 81.01 % at optimum values of 0.3 g of adsorbent dosage, pH of 9., initial dye concentration of 50 mg/l and a contact time of 90 minutes. The maximum adsorption capacity of the adsorbent was found to be 16.03 mg/g.



**Figure 12: Experimental validation of model**

**3.5 Adsorption Isotherms**

The result of fitting the Langmuir and Freundlich models to the adsorption data is presented in Table 6. The Freundlich model which gave an R-square value of 0.947, fitted better than the

Langmuir model which gave an R-square value of 0.348. This suggests that the adsorption of Congo red on treated pumpkin pod, follows heterogeneous poly layer coverage of the dye on the adsorbent surface.

**Table 6: Evaluated adsorption parameters for Langmuir and Freundlich isotherms**

Pseudo-first order					Pseudo-second order				
$q_e^{exp}$ (mg/g)	$q_e^{cal}$ (L/mg)	$K_f$ (h <sup>-1</sup> )	$R^2$ (COD)	$R^2$ (Adj.)	$q_e^{exp}$ (mg/g)	$q_e^{cal}$ (mg/g)	$K_f$ (h <sup>-1</sup> )	$R^2$ (COD)	$R^2$ (Adj.)
3.188	0.556	0.550	0.009	0.131	3.188	2.982	-0.740	0.996	0.994

**3.6 Adsorption Kinetics**

The pseudo-first order and the pseudo-second order kinetic models were tested on the adsorption data, the results are presented in Table 7. The pseudo-second order model fitted the adsorption data (R-square = 0.996), while the pseudo-first order model showed a lack of fit (R-squared = 0.009). This indicates

that the kinetic behavior of CR adsorption unto the treated pumpkin pod can be satisfactorily explained with the pseudo-second order adsorption equation. Jabar et al (2020), also reported that the pseudo second-order kinetic model best described the adsorption of Congo red dye unto Moringa oleifera

**Table 7: Evaluated parameters for pseudo-first order and pseudo-second order kinetics for the adsorption process of CR.**

Langmuir Parameters				Freundlich Parameters					
q <sub>m</sub> (mg/g)	b <sub>L</sub> (L/mg)	R <sub>L</sub>	R <sup>2</sup> (COD)	R <sup>2</sup> (Adj.)	q <sub>m</sub> (mg/g)	K <sub>f</sub> (mg/g)	n (L/g)	R <sup>2</sup> (COD)	R <sup>2</sup> (Adj.)
16.025	0.026	0.550	0.348	0.131	16.025	0.628	1.121	0.947	0.929

#### 4. CONCLUSION

From the values of the properties determined, the surface morphology and functional groups established from the SEM and the FTIR spectra respectively, the characterization of the adsorbent indicates that Sodium thiosulphate pretreated Pumpkin pod can serve as a good adsorbent for wastewater treatment. The adsorption of Congo red dye on the adsorbent was found to be dependent on adsorbent dosage, contact time and initial dye concentration, while pH had no effect on the adsorption process within the conditions investigated. These trends align with reports of related works in literature. An optimum percentage dye removal of 81.01 % can be achieved. The mechanism of adsorption follows heterogeneous poly layer coverage of the dye on the adsorbent surface, which is consistent with the Freundlich model, while the kinetics of the adsorption process can be described by the pseudo-second order kinetic model. Pumpkin pod pretreated with Sodium thiosulphate, can be used as a cheap and efficient adsorbent for the removal Congo red dye from aqueous solutions such as Textile industry wastewaters.

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