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Removal of Congo Red Dye from Aqueous Solution Using Different Clays Adsorbent

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ABSTRACT

The current work aims to the adsorption of Dye (Congo Red (CR)) from aqueous solutions through double adsorbing surfaces, namely Koura clay and pottery clay. Different methods are used to remove it from industrial wastewater. One of these successful methods is adsorption on solid surfaces within a wide range of dye concentrations under different experimental conditions such as (initial dye concentration, equilibrium time, adsorbing surface quantity, acidity function, and temperature). the best time for adsorption of dye (CR) on the surface of Koura clay is (1h), while its adsorption on the surface of pottery clay was (1.5 h), pH = 6, particle size =200 μ m mesh, dye concentration 15 mg/L , and 0.05 g/L adsorbent). The adsorption process balances were tested by some models such as (Langmuir, Frendelich, Elovich, Temkin, Harkin - Jura). The results showed that the adsorption of (Congo Red (CR)) dye on both surfaces of Koura clay and pottery clay followed the Frendelich model, and the adsorption isotherm was also applied to obtain thermodynamic functions within the experimental temperature range (290-320k). The values of enthalpy Δ H, entropy Δ S, and gypsum energy Δ G depend on the calculation of the equilibrium constant (Keq).

Keywords: Adsorption, water pollution, Congo Red dye, pottery clay, Koura clay

1. Introduction

Dyes are branded constructed on their chemical assembly and planned use. There are 20–30 discrete dye groups that may be eminent constructed on chromophores, with azo, anthraquinone, phthalocyanine, and triarylmethane being the most prominent. The most common dye classes are azo (about 70%) and anthraquinone (approximately 15%) [1]. Azo dyes are chemical molecules containing the functional group R- N=N -R0, with R and R0 often being aryl. The azo link, as well as several, mostly aromatic, side groups, keep azo compounds stable and allow them to create vibrant hues [2]. Azo dyes which are made up of one or more azo linkages (N=N) and sulfonic groups, are the most common artificial aromatic colors (-SO3-). These dyes typically have one to three azo bonds attached to naphthyl, with functional groups such as methyl, hydroxyl, chloro, nitro, sulphonate, and triazine amine replacing phenyl rings [3-5]. Congo red is an anionic dye commonly used in the papermaking, textiles, and plastic industries. It is a typical disazo dye with four color-assisting groups (amino and sodium sulfonate). Because of its weak degradation, this dye can be discharged with sewage, posing a significant environmental risk [6]. Because of their toxicity to aquatic life and mutagenicity to humans, the presence of such chemicals in industrial effluent might cause major environmental concerns. Despite their resistance to biodegradation under aerobic settings, dyes (particularly azo dyes) are rather easy to reductively split the azo link under anaerobic conditions, releasing aromatic amines [7-10]. 'which has an IUPAC name of : disodium 4- amino-3-[4-[4-(1-amino-4-sulfonato-naphthalen-2-yl) diazenylphenyl] phenyl] diazenyl-naphthalene-1-sulfonat ' [11,12] . chemical structure of Congo Red is shown Figure1. [13]:



Figure 1. The chemical structure of Congo Red

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Dye toxicity has been willful and exposed to have a harmful influence on the ecosystem. These investigations reveal that even at low concentrations, some dyes deteriorate and their derived compounds can be hazardous and carcinogenic [14]. Adsorption is the collecting of a material onto the surface of adsorbent solids or a removal process in which specific particles are chemically or physically attached to an adsorbent particle surface [15].

2. METHODOLOGY

2.1. Prepare the adsorbent surface

The route of washing the tops (pottery clay and Koura clay) with sanitized water was carried out several times to remove suspended substances and precipitated salts present on the surfaces. It has a granular size of $200\mu m$. as shown in (Figure (2) Koura Clay) and Figure (3) (Pottery Clay)



FIGURE 3. (Pottery Clay) Before and after washing, drying and grinding

Constituent	Pottery Clay (Wt%)	Koura Clay(Wt%)
SiO ₂	40.85	39.71
CaO	15.05	18.32
Al ₂ O ₃	10.11	9.624
Fe ₂ O ₃	8.44	6.905
MgO	6.775	5.063
K ₂ O	2.206	1.343
TiO ₂	0.779	0.929
Na ₂ O	0.509	1.357

 Table 1: The Chemical analysis of Pottery Clay and Koura Clay

2.2. Preparation of standard solutions and calibration curve

The standard stock solution of the Congo Red (CR) dye was prepared at a concentration of (50 ppm), by dissolving a weight of (0.05 gm) in (1000 ml) of distilled water. After those different concentrations were prepared ranging from (2 to 20 ppm) as shown in **Figure (4)**. The wavelength length λmax has measured to get the greatest absorption of the dye using distilled

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water (Reference) (Blank). Then, the absorbance was measured for each of the ten samples by UV-visible spectrophotometer (as shown in **Figure (5)** to determine the calibration curve of the dye. The Congo Red solution was measured using a UV-Vis spectrophotometer and the value of λmax (497nm) was based on this wavelength **Figure (6)**. The calibration curve was drawn for the dilute solutions, and the straightness of the calibration curve is a good indication that the calibration curve is subject to Beer's law. (Beer-Lambert) as shown in **Figure (7)**.



FIGURE 4. Preparing of Congo red (CR) dye in different concentrations



FIGURE 5. UV-visible spectrophotometer

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FIGURE 6. The maximum wavelength of Congo Red and UV -visible spectroscopy devices



FIGURE 7: calibration curve for Congo red at 290 k and PH=7

2.3. Effect of contact time

This study was conducted at a temperature of 290 K between different clays (pottery clay and Koura clay) and Congo red dye. The volume of all samples was (15 ml) concentration at a concentration (20 ppm) and the clay weight was (0.2 g). And at different time intervals (0.5, 1, 1.5, 2, 2.5, 3 hours). After that, the samples for each of the dyes are placed in a shaking water bath and then the samples were filtered with filter paper twice, and then the filtered absorbance was measured by UV-visible spectrophotometer.

2.4. Determination the weight of the adsorbent surface

The effect of surface weight on the adsorption capacity was studied to calculate the best surface weight for adsorption of Congo Red dye at a constant temperature (290 K) and pH = 7 with different weights (0.05, 0.1, 0.15, 0.2, 0.25, 0.3) g of both surfaces, and the size (15 ml) of concentration (20ppm). The prepared samples were placed in a shaking water bath for (1.5h) for pottery clay. and (1h) for Koura clay respectively, then filtered with filter paper, and absorbance was measured.

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2.5. Effect of acidic function and zero charge point

The effect of the pH was studied by preparing five samples of the solution with a volume of (15 ml) from the concentration (20 ppm) from Congo red dye, where the pH of the five samples was adjusted (2,4, 6, 8,10) by using an acidic solution of HCl and NaOH at a concentration of (0.1 M) respectively. Then, the adsorption surface with a weight of (0.05 g) from both surfaces (Koura clay and pottery clay) was placed in the five samples and placed in a vibrating water bath for (1 h) for Koura clay, and (1.5 h) for pottery clay at 290k. Then the samples were filtered and the absorbance of the filter was measured by a UV visible spectrophotometer. To determine the surface charge of the absorbent material, five samples of Congo red dye (15 ml) were prepared for each volumetric flask (20 ppm), and then (10 ml) of NaNO3 salt solution (0.1 M) was added as Adjusting the pH in the five samples (2, 4, 6, 8, 10) respectively, using an acidic solution of HNO3 and basic NaOH at a concentration of (0.1 Molar), then placing the adsorption surface by weight (0.05 g) for both surfaces (Koura clay and pottery clay) and left for (24 hours) after which the filter absorption is measured after the filtration process of the models. The pH for each ΔpH is extracted by the following equation (1) **[16]:**

 $\Delta pH = pHf - pHi$ (1)

pHi : Before adsorption with the (Congo red) dye

pHf: After adsorption with the (Congo red) dye

2.6. Effect of ionic strength

The concentration of dyes (20ppm) was placed in six volumetric bottles (100ml), then a fixed amount of surface weight (0.05g) was added and different weights of salt (NaCl) were added, ranging from (0.05, 0.1, 0.15, 0.2,0.25,0.3gm) as it was placed in the shaking device at a temperature of 290K and according to the equilibrium time of the dye with the surfaces used in this study, then filtered twice and the absorbance was measured and then the amount of adsorption was calculated.

3. Adsorption isotherms

Ten samples of Congo red dye were prepared at concentrations (2-20 ppm) and 15 ml of each concentration of that dye. It was added to the surface with a weight of (0.05 g) from both surfaces (pottery clay and Koura clay) at different temperatures (290,300,310,320k) and at a neutral acidity function. These samples were placed in a shaking water bath and after a specified period of time (1.5h) for the dye with a pottery clay surface , (1h) was mixed with Koura clay, then the samples were filtered. filter paper twice. Then the absorbance was measured in a UV spectrophotometer at the maximum corresponding to the Congo red dye, and the concentration after adsorption was detected through the calibration curve by the following equation (2) **[17]:**

 $qe = \underline{(c^{\circ} - ce) \, Vsol} \qquad (2)$

m

qe : The amount of adsorbent (mg/g)

 c° : The initial concentration of Congo red dye (mg/l)

: Concentration at equilibrium for the Congo red dye (mg/l)

Vsol : The total volume of the adsorbent (L)

: The weight of the adsorbent (g)

The amount of CR dye adsorbed was calculated from the following equation (3):[18]

%Adsorption =
$$\frac{(Co-Ce)}{Co} \times 100$$
 (3)

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4. Results and Discussion

4.1 Contact time effect

Figure (8) shows the effect of the equilibrium time in hours on the percentage values of adsorption of Congo Red (CR) dye, at a concentration of (15 mg/l) and at pH = 7 and a temperature of 290 k on the surface of pottery clay and Koura clay with a constant weight (0.2 gm) from both surfaces, The results indicate that the equilibrium time was (1.5h) for (CR) dye on the surface of pottery clay and (1h) for (CR) dye on the surface of Koura clay, as the percentage of removal was 94.75% and 82.95% of the dye from both surfaces, respectively.



4.2. The effect of Surface Weight on adsorption

The effect of the weight of surfaces (pottery clay and Koura clay) on the amount of adsorption was studied, with the time required for the equilibrium process to be obtained for the Congo red dye with both surfaces and at a temperature of K290 and an acidic function) 7 (pH = and at a concentration of 15 mg/l)) of (20ppm) for (CR) dye, as six different weights (0.05, 0.1, 0.15, 0.2, 0.25, 0.3 g) were used. The Figures (9) show that the best weight for both surfaces for the same dye was (0.05 g). We conclude from this that with an increase in the weight of the surface of the adsorbents will decrease the adsorption amount of the dye. This may be due to the fact that most of the active sites of the adsorbent surface have been saturated at these weights so that the increase in the amount of the adsorbent surface will not affect the adsorption of the dye [19].



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4.3 Effect of temperature

Figures (10-11) show the amounts of adsorption on the adsorbing surfaces (pottery clay, Koura clay) within the experimental temperature range (290-320K) for Congo Red dye at a range of concentrations (2-20 ppm) and at an acidic function pH = 7, and those figures show the relationship Between the amount of adsorption at equilibrium and the equilibrium concentration, the Congo red (CR) dye in Figures (10 -11) indicates that the amount of adsorbent material at 290 K is the highest and at both surfaces (pottery clay and Koura clay), then it decreases slightly at The rest of the degrees, that is, the adsorption process is an exothermic process.





4.4 Thermodynamics of absorption

The following equations was used to compute the thermodynamic parameters, change in free energy (ΔG), enthalpy (ΔH), and entropy (ΔS): [20].

 $KC = Ae - \Delta H / RT$ (4)

 $Ln Xm = -\Delta H/RT + K$ (5)

where ln Xm is the natural logarithm for greatest amount adsorbed (mg/g), K is the constant of Van't Hoff equation, R is the universal gas constant (8.314.10-3 kJ/mol. K-1), and T is the temperature (Kelvin).

 $\Delta G^{\circ} = -RT \ln K \tag{6}$

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 $K = Qe \times m/Ce \times V \Delta G^{\circ} = \Delta H - T\Delta S^{\circ}$ (7)

 $\Delta S^{\circ} = \Delta H - \Delta G^{\circ} / T$

 ΔH° and ΔS° were obtained from the slope and intercept of a Van't Hoff plot of ln Kc versus 1/T (Figure 12, Table 2). The thermodynamic parameters were obtained and the negative value of ΔH° was an indication of the exothermic nature of the adsorption.

(8)



Koura clay			Pottery clay			
$\Delta H(KJ/mol.K)$	$\Delta S(J/mol.K)$	$\Delta G(KJ/mol.K)$	$\Delta H(KJ/mol.K)$	$\Delta S(J/mol.K)$	$\Delta G(KJ/mol.K)$	
-7.889	-19.101	5.547	-4.124	0.374	-104.33	

4.5 Adsorption Equilibrium and Adsorption Isotherms Models

In various investigations, several models for analyzing experiment findings have been proposed, the most prominent of which being adsorption isotherms. The relationship between absorbed CR dye and the interpretation of adsorption isotherms in order to develop an equation, present the results, and design the system in these six models was demonstrated by the interpretation of adsorption isotherms in order to develop an equation, present the results, and design the system. Langmuir, Freundlich, Tekmin, Harkins-Jura, Elovitch, and Dubinin isotherms were used. The Langmuir isotherm was developed using a single layer of homogeneous absorbing material with the same energy in the surface. The Freundlich isotherm was estimated on a heterogeneous surface with a non-uniform absorption heat distribution .[21]

4.5.1 Langmuir Isotherm

The Langmuir model explains the adsorption of adsorbate onto an adsorbent surface, which results in the formation of a monolayer on the adsorbent's outer surface. It is assumed that adsorption sites are confined and homogeneous. The Langmuir isotherm can be expressed by the equation (9) [22].

$$\frac{C_e}{q_e} = \frac{1}{Q_m K_L} + \frac{C_e}{Q_m} \tag{9}$$

where Ce denotes the dye concentration in aqueous solution at equilibrium, qe the equilibrium concentration of CR dye adsorbed, and KL the Langmuir constant, which is proportional to the apparent energy of adsorption. On a plot of Ce/qe versus Ce, a straight line with a slope of (1/qmax) and an intercept of 1/ should appear (KL qmax), As shown in Figure (13).

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4.5.2 Freundlich Isotherm

The Frenudlich model yields an exponential distribution of active sites for multilayer adsorption and adsorption on heterogeneous surface energies. This model's linear form is represented by the equation (10) .[23]

 $\ln Qe = \ln KF + 1/n \ln Ce$ (10)

As illustrated in Figure (14), the Frenudlich constants KF and n, which represent the adsorption capacity and intensity, are determined from the intercept and slope of the plot ln Qe vs ln Ce, respectively. The adsorption intensity (n) was modest (n<1), indicating a very low affinity between adsorbents and adsorbate. With rising temperature, the Freundlich constant (KF) falls, indicating an exothermic process. The n values are more than 1, indicating a suitable removal situation [24].



4.5.3 Elovich Isotherm

This model's equation is based on the kinetic principle, which states that adsorption sites expand exponentially with adsorption, implying multilayer adsorption [25].

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The Elovich model's linear forms are written as follows: [26].

 $\operatorname{Ln}(q_e/C_e) = \ln K q_m - 1/q_m q_e \tag{11}$

The slope and intercept of the plot of ln(qe/Ce) vs q_e may be used to compute Elovich maximal adsorption capacity and Elovich constant As shown in Figure (15).



4.5.4 Temkin Isotherm

Plotting qe versus lnCe yielded the Temkin isotherm's linearized graph. The slope and intercept of the resulting curve were used to determine the KT value. This model assumes that the heat of absorption, which is a function of temperature in the layer, decreases linearly rather than logarithmically, as shown in the Eq (12) and Figure(16) :[27].

 $Q e = B \ln K T + B \ln C e$ (12)

Where, B = Temkin constant related to heat of adsorption KT = Temkin isotherm equilibrium binding constant qe = quantity of dyes adsorbed at equilibrium (mg/g) Ce = concentration of CR at equilibrium (mg/L)



4.5.5 Harkin-Jura Isotherm

This isotherm was applied by the two scientists Harken and Jura and named after them and describes the multi-layer adsorption on heterogeneous surfaces as shown by the following equation (13) [28].

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4.5.6 Dubinin - Radushkevich Isotherm

The Dubinin–Radushkevich isotherm is commonly used to explain the adsorption mechanism onto a heterogeneous surface with a Gaussian energy distribution. The model has had a lot of success fitting data with high solute activity and a wide range of concentrations **[29]**. The Dubinin-Kaganer-Radushkevich (DKR) model has the linear form Eq (14) **[7]**:

$$\ln q_e = \ln Xm - \beta E^2 \tag{14}$$

When 1 mole of sorbate is introduced onto solid surfaces from an absolute solution, the constant expressed regarding the mean free energy E(KJ mol-1) of sorption may be found using the following equation Eq. (15):

 $\mathbf{E} = 1/\sqrt{2B} \tag{15}$

The mechanism of adsorption was deduced by solving the aforementioned equation. When E is between 8 and 16 KJ mol-1, the sorption process is called chemisorption, and when E is less than 8 KJ mol-1, it is called physical sorption [**30**].



Table 3: Parameters of the isothermal models of adsorption of Congo red dye on the surface of Koura clay and pottery clay at 290 K and acidity function pH = 7

adsorption isotherm Koura ciay pottery ciay

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	Constants			Constants		
langmuir	KL	Qm	R2	KL	Qm	R2
	0.31	0.224	0.7823	0.8686	1.086	0.8544
Freundlich	Kf	Ν	R2	Kf	Ν	R2
	0.028	0.233	0.9201	5.105	0.391	0.9431
Temkin	KT	В	R2	KT	В	R2
	0.529	5.7198	0.7072	0.876	5.192	0.774
Elovich	Qm	Κ	R2	Qm	Κ	R2
	-2.079	0.785	0.8242	-3.679	-0.096	0.9193
Harkins-Jura	А	В	R2	А	В	R2
	0.2442	0.5074	0.4135	0.323	-0.021	0.7614
Dupin	LnQm	В	R2	lnQm	В	R2
	5.0667	0.0057	0.9102	4.4807	0.0017	0.9395

4.6 Effect of pH and (pHzpc)

For two s surfaces (koura clay and pottery clay), the influence of solution pH on CR dye removal from solution was tested under identical conditions. The results are shown in Figure (19), which show that the dye's adsorption behavior was identical from(pH 2 to pH 10). [31] Where it was found from Figure (19) that the highest amount of adsorption of the dye used in this study was at the value of the acidity function = 2 at both surfaces. Zero Point Charge (pHzpc): is a concept relating to adsorption phenomenon and defined as the pH at which the surface of an oxide is uncharged. If positive and negative charges are both present in equal amounts, then this is the isoelectric point (iep). However, the ZPC is the same as iep when there is noadsorption of other ions than the potential determining H+/OH–at the surface [32]. The results are shown in Figure (20)



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4.7 Effect of ionic strength

By varying the quantity of NaCl from 0.05 to 0.3 mol/L, the effect of ionic strength on dye adsorption was investigated. A decrease in CR removal capacity was caused by an increase in ionic strength As shown in the Figure (21). The electrostatic interaction between opposing charges at the solid sorbent surface and on dye molecules may have been veiled by the presence of salt (NaCl) in the solution, resulting in the adsorbed quantity reducing as the NaCl concentration increased [33].



Conclusion:

1- At a temperature of 290K and a weight of 0.05 g, the surface of pottery clay was more efficient in adsorption of CR dye, with a removal rate of 94.75 percent, while the removal rate of the same dye on the surface of Koura clay was 82.95 percent.

2- The study showed that the adsorption isotherms of CR dye on both surfaces (pottery clay and Koura clay) were following the Freundlich model depending on the values of the correlation coefficient (R^2).

3- The study indicates that the effect of the acidic function of CR dye on two surfaces of Koura clay and pottery clay had the highest amount of adsorption at PH = 2. While the zero charge point for both surfaces = 6.5.

4- The study showed the effect of the temperature of the CR dye on the surface of the ball clay, where the adsorption was (exothermic) and the value of ΔG is positive indicating that the adsorption is not spontaneous and that ΔS was negative and indicates randomness during adsorption, while the adsorption of the same dye on the surface of the pottery clay was also (exothermic). and ΔG is negative and indicates spontaneous adsorption, and the value of ΔS is positive.

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