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Eco-Friendly Synthesized of CuO Nanoparticles Using *Anchusa strigosa L.* Flowers and Study its Adsorption Activity

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Abstract: **uui**

Environmentally friendly copper oxide nanoparticles (CuO NPs) were prepared with a green synthesis route via *Anchusa strigosa L.* Flowers extract. These nanoparticles were further characterized by FTIR, XRD and SEM techniques. Removing of Gongo red from water was applied successfully by using synthesized CuO NPs which used as an adsorbent material. It was validated that the CuO NPs eliminate Congo red by means of adsorption, and the best efficiency of adsorption was gained at pH (3). The maximum adsorption capacity of CuO NPs for Congo red was observed at (35) mg/g. The equilibrium information for adsorption have been outfitted to the Langmuir, Freundlich, Temkin and Halsey adsorption isotherm types. The temperature thermodynamic parameters like ΔG°, ΔH° and ΔS° have been calculated.

Keywords: *Anchusa strigosa L*, Congo Red dye, Freundlich Isotherm, Halsey Isotherm, Langmuir Isotherm, Temkin Isotherm.

Introduction:

Dyes are used drastically in many industries covering by dye homes, textile dyes, and paper printers[1]. A considerable proportion of synthetic dyes are misplaced yearly to waste streams for the duration of textile processing, which subsequently enters the surroundings and causes pollution the environment[2]. Textile dyes have a synthetic origins and complex fragrant molecular structures that make them tough to biodegrade when discharged within the environment. A number of dyes are carcinogenic and mutagenic, like Congo pink dye [3]. The maximum regularly used strategies for the removal of dyes infection from commercial effluents encompass ion exchange, membrane filtration, reverse osmosis, solvent extraction, and adsorption[4-9], but adsorption using numerous nanomaterial's is one of the tremendous methods amongst all the above-stated techniques for the exclusion of contamination from water because of its efficiency, simplicity, and low cost[10].

Nanomaterial along with metal nanoparticles, a few of the various metal oxides, copper oxide (CuO), and CuO-primarily based nanomaterial's, vital substances which are in large part hired in power, and clinical packages, have won sizable interest for the elimination of dyes from polluted water for environmental remediation because of their excessive performance and low cost[11]. To date, distinct physical and chemical procedures have been mentioned to manufacture copper oxides nanoparticles with distinct size, shape, and morphology. Specifically, nanoparticle synthesis. And the usage of green strategies has gained tremendous attention in the recent years[12]. Green synthesis of metallic and metal oxide NPs using environmentally friendly materials such as plant extracts has gathered much more attention due to the mounting necessity to develop such a technology[13]. Green preparation of diverse metallic oxide nanoparticles using of plant-based substances has also attracted vast attention[14]. Consequently, plant extracts were established as excellent materials for the large-scale, easy, and green synthesis of properly-dispersed metal and metal oxide nanoparticles of managed styles and sizes[15]. The aims of this project are to green synthesis of CuONPs using some flower *Anchusa strigosa L.* Flowers extract, characterization of synthesized copper oxide nanoparticles CuO NPs, study the factors affecting the green synthesis of CuO NPs and teste the obtained CuO NPs as an adsorptive nanomaterial to purify polluted water from some dyes as well as modelling the experimental results with isotherm and kinetic models.

Materials and Methods:

Congo red is an anionic dye, the sodium salt of 3,3′-([1,1′-biphenyl]-4,4′-diyl) bis(4-aminonaphthalene-1-sulfonic acid) $C_{33}H_{22}N_6NaO_6S_2$ as shown in Fig. 1. Congo red has a molecular weight of 696.7 g mol^{-1} and purity of 98.99 %. It is soluble water azo dye with a red colloidal solution. Congo red has better solubility in organic solvents. Due to their carcinogenic properties Congo red was abounded[16]. Congo red was formerly used to dye cotton but has been superseded by dyes more resistant to light and to washing. It is still used in histology to stain tissues so they can be looked at under a microscope and as an acid-base indicator because it turns red when it is near an alkali and blue when it is near an acid[17].

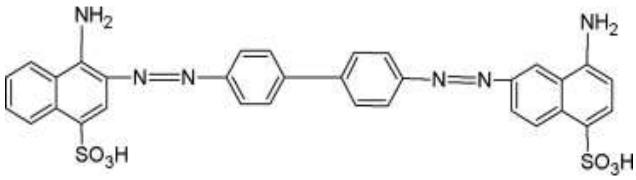


Figure 1. Structural formula Congo Red Dye[18].

Reagent and materials:

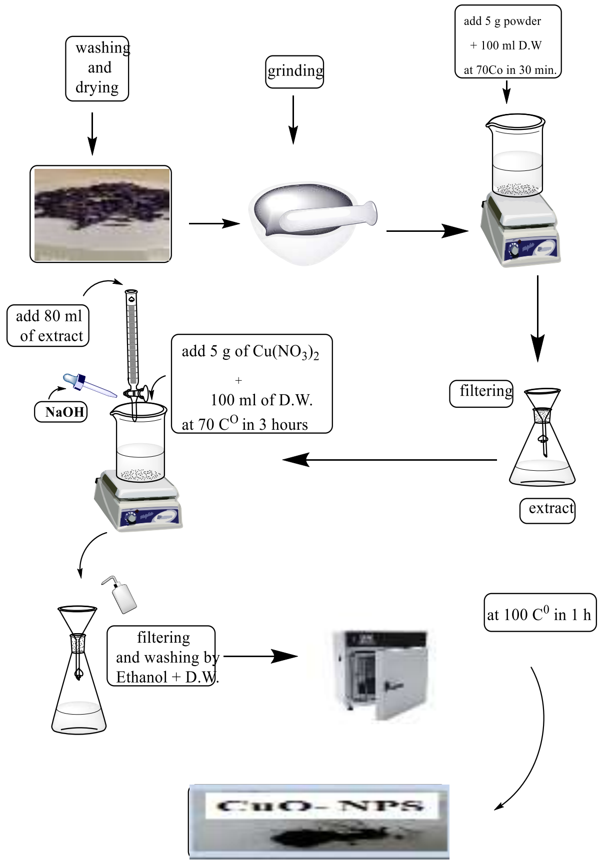
All reagent were used in this work were illustrated in Table 1.

Table 1. The material using in work

The chemical material	Purity	The manufacturer
Copper (II) Nitrate	99.9%.	Sigma-Aldrich
HCl	35-38%,	RESEARCH SINCE
NaOH	98.0%	SDFCL
Ethanol	99%	Honeywell
NaCl	+98%,	GERBU BIOTECHNIK
KCl	> 99.8%	CEPHAMLIFE SCIENCES
$MgCl_2$ and $CaCl_2$	99%.	Spectrum

Preparation of CuO NPs:

1- Bull tongue flowers were collected from areas of northern Iraq, washed, and then dried.
2- The dried paper was crushed, and it took 40 g.
3- Melt the weighted flower flour in distilled water by 200 ml .
4- Leaching solution to take out the extract.
5- Prepare a 20 g $Cu(NO_3)_2$ in 100 ml distilled water.
6- We add 160 ml of the extract to the nitrate solution with a burette.
7- We filter the product and dry the incubator producing a brown precipitate, which is copper oxide Nanoparticles, as shown in Scheme 1.



Scheme 1. Preparation of CuO NPs

Characterization

Synthesized Copper oxide nanoparticles (CuO NPs) were characterized and confirmed by FT-IR spectra (IR-Prestige 21 spectrophotometer), X-ray diffract meter (XRD-6000, Shimadzu), and SEM (FESEM FEI Nova Nano SEM 450).

Characterization of copper oxide nanoparticles

FT-IR spectrum of synthesized CuO NPs using the *Anchusa strigosa L.* flower aqueous extract is shown in Fig. 2.

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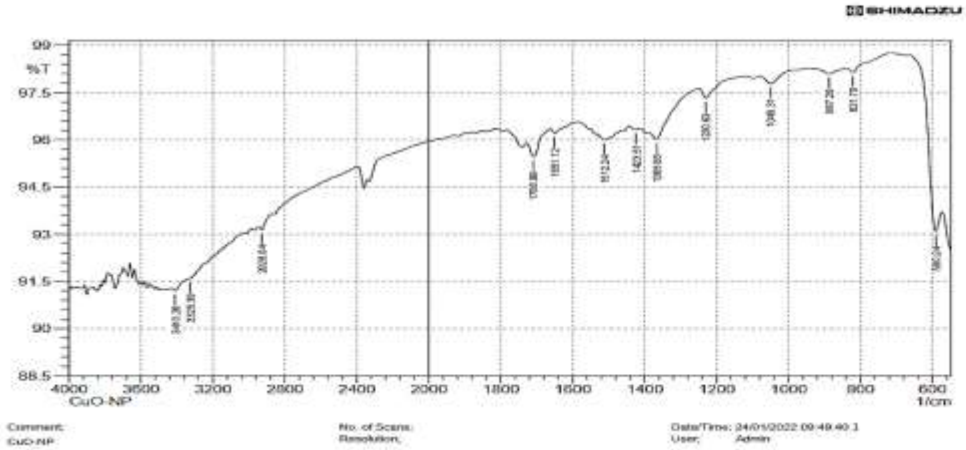

Figure 2. The FT-IR spectrum of CuO NPs.

The broad absorption band at 3410 cm^{-1} was due to the -OH group of melanin, and the broadening of the band was possibly due to the hydrogen bonding of OH groups and amine groups. The peaks at 2928 and 2830 cm^{-1} were assigned to C-H stretching, and the peak observed at 1708 cm^{-1} was due to C=C stretching. The peak at 1512 cm^{-1} was due to N-H and C-N groups. The peak observed at 1365 cm^{-1} was due to the bending of the O-H of carboxylic acid present in the CuO NP the peak at 550 and 584 cm^{-1} which are attributable to CuO stretching modes.

XRD analysis of the synthesized CuO NPs by flowers of *Anchusa strigosa L.* aqueous extract was illustrated in Fig. 3.

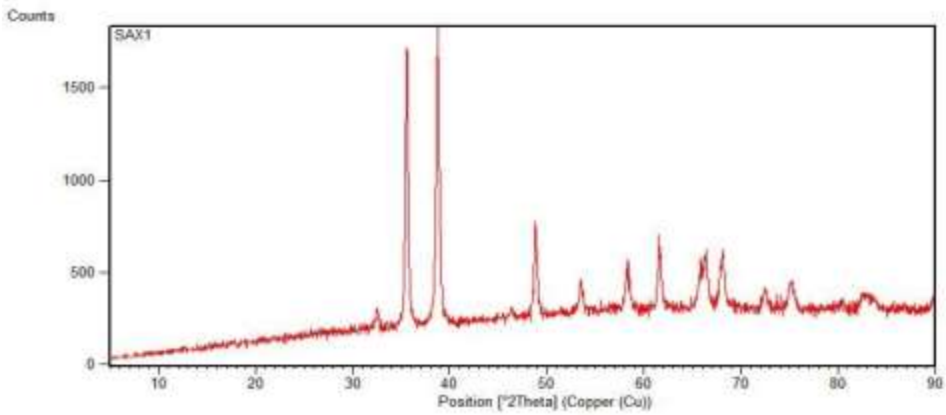

Figure 3. The X-RD spectrum CuO NPs.

XRD analysis of the synthesized CuO NPs by flowers of Anchusa strigosa L. aqueous extract was illustrated in Fig. 3. The 2θ peaks at 33.3°, 36.2°, 38.4°, 49.1°, 53.4°, 58.4°, 62.2°, 65.8°and 67.2° are attributed to the crystal planes of copper oxide at (110), (002), (111), (202), (020), (202), (113), (311) and (113) respectively. The copper oxide nanoparticles (CuO NPs) are well crystalline and the position and the relative intensity of the diffraction peaks match well with the standard phase CuO NPs diffraction pattern of the International Center of Diffraction Data card (JCPDS-80-1916). The SEM micrograph of the CuO NPs (synthesized using the extract of flowers *Anchusa strigosa L.)* is illustrated in Fig. 4.

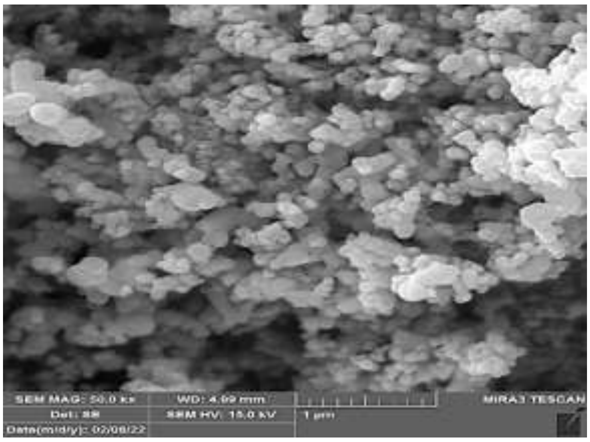

Figure 4. SEM micrograph of CuO NPs.

This shows that the prepared CuO NPs were mostly spherical in shape and appear with more aggregates. This can be due to the coating of different surface functional groups from the appeared extract.

Adsorption activity evaluation

The study was performed using 0.0300g adsorbent surface weight, 25ml Congo Red dye solutions 35 mg/L,this study was performed in the thermal shaker the (180 rpm). In each solution, samples were tested by UV-Vis 1800 dual-beam digital a device with a wavelength that corresponds to λ_{max}. This study showed the impact of touch time, CuO NPs dose, acid function, ionic strength, and temperatures were in this study. Thermodynamic studies have made by stages study at the best conditions and at different temperatures were investigated.

$$\text{Removal }\% = \frac{C_0 - C_e}{C_0} \times 100 \ldots\ldots 1$$

Where: C_O, C_e Indicate the starting concentration and equilibrium concentration of residual dye mg/L straight. [19].

Results and Discussion:

Contact Time Effect

By attaing the relationship between osculate time and the efficacy of removing Congo Red using CuO NPs, the equilibrium time was definite, as seen in Fig.5 .The end resulting is leads to equilibrium time of run to after 30min of operation time.

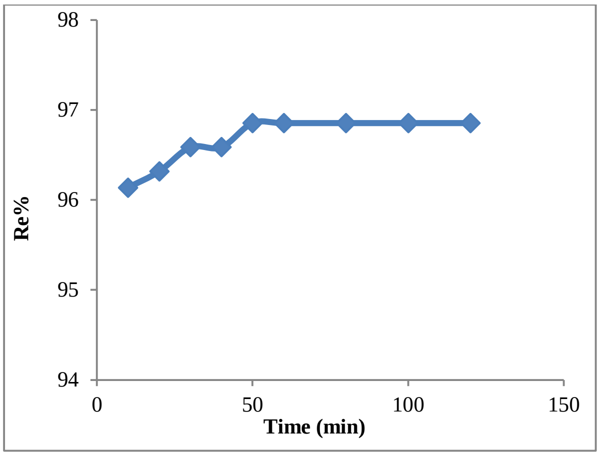

Figure 5. Impact contact time in adsorption Congo Red using CuO NPs at temp. 298 K, Conc. of dye 35 mg/L, the adsorbent's weight 0.0300 g.

Effect weight of the adsorbent

The operation experiments were conducted using a Different weights between (0.0050-0.0700) g, the dye concentration was initially 35mg/L,Was at 298 K temp. Fig.6. The results we gained indicate that the elimination rate is increases as the weight of the adsorbent surface CuO NPs increases because of the surface area of the increase absorbent material. So the removal percentage attained stable value, which is the saturation of the active sites of CuO NPs, and so 0.0300 g was selected as the best weight for the adsorbent.

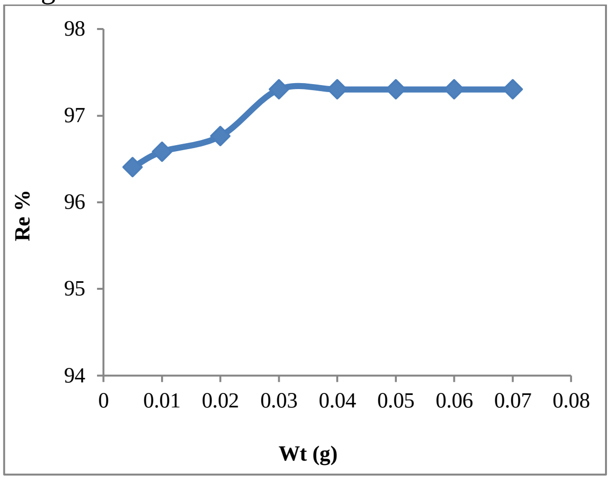

Figure 6. Impact weight of the adsorbent of Congo Red using CuO NPs, temp. 298K, Conc. of dye 35 mg/L

Effect of acid function

When examining the effect of acid function on removal rate, the range of acid function values (3, 5, 7, 9 and 1)[1] were selected Fig. 7. 35mg/L was taken, which is the dye concentration at the start, then pH laying was taken as in the above range using 0.01N of HCL and NaOH. The result shows that the lowest value of removal in pH 3, that the ratio of elimination increased with increases of the pH 5 until it reached pH 7. After pH 9, there was no marked increase, but there was a decrease in the ratio of elimination. And so acid function 3 pH was selected, conformed, and constant in other experiments.

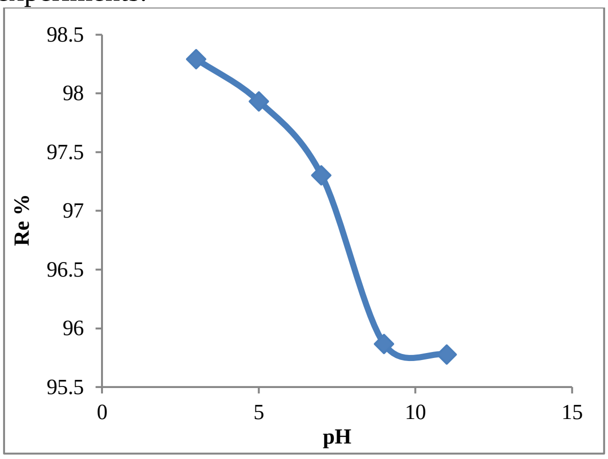

Figure 7. Impact of acid function in adsorption of Congo Red using CuO NPs at temp.298K, Conc. dye 35mg/L, the adsorbent's weight0.0300g.

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Effect of ionic strength.

Dissimilar concentrations of NaCl, KCl, $MgCl_2$ and $CaCl_2$ (0.0200-0.0800M), were chosen to research and show the effect the ionic strength on the removal efficacy ratio Fig.8. It illustrates the interdepended relationship of the percentage of salt concentrations mentioned above.

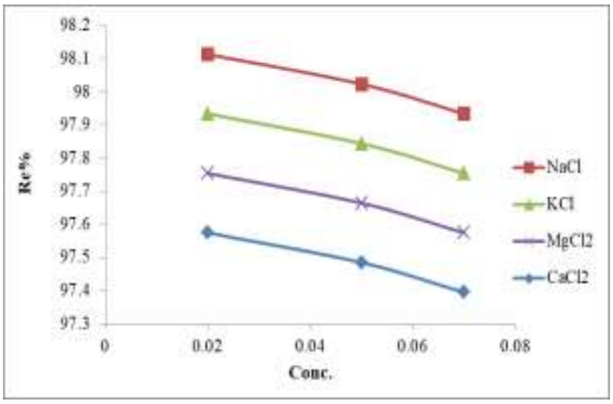

Figure 8. Effect of the ionic straight in adsorption of Congo Red using CuO NPs at temp. 298 K, conc. dye 35mg/L, the adsorbent's weight 0.030g

Form Fig. 8 it can be observed that good adsorption was obtained by using smaller ion volume and less charge.

Effect the Temperature

The study of the impact of change in temperature has been explained to show and understand the adsorption process's nature. Table contains information for adsorption, free Gibbs energy change (ΔG), the term enthalpy (ΔH) and entropy (ΔS) which are found as Equations (1,2,3and 4).[20].

$$Keq = \frac{Q_e\,M}{C_e\,V} \;\ldots\ldots\ldots\; 2$$

Where: Keq = equilibrium constant for the adsorption process at each temperature.
Qe (mg/g) = amount of dye adsorbed at equilibrium (adsorption capacity), Ce(mg/L) = the concentration of Congo Red dye at equilibrium.
V = the volume of the solution (L).
M = the adsorbent mass (g).
Fig. 9. show Vant Hoff equation for the adsorption of Congo Red dye on a CuO NP surface

$$\Delta G = -RT \ln Keq \ldots\ldots\ldots\; 3$$

ΔG = Gibbs energy change (kJ/mol).
R = the ideal gas constant (8.314 J/mol. K)
T = the absolute temperature (K)

$$\text{Ln Keq} = -\frac{\Delta H}{R\,T} + \text{Constant} \quad \ldots\ldots\ldots\ldots\ldots\ldots\; 4$$

$$\Delta S = \frac{\Delta H - \Delta G}{T} \;\ldots\ldots\ldots\ldots\ldots\ldots\; 5$$

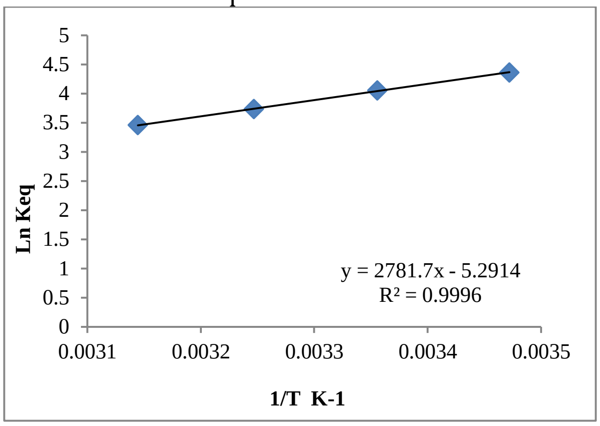

Figure 9. Draw the Vant Hoff equation for the adsorption of Congo Red dye on a CuO NP surface

Table 2. Function values ΔG, ΔH and ΔS in Congo Red using CuO NPs (288-318K)

	Tempe. K	ΔG kJ/mol	ΔH kJ/mol	ΔS J/mol.K
Congo Red	288.	10.4471-	-23.1271	-44.0275
	298	10.0421-		-43.9096
	308	9.55934-		-44.0513
	318	9.14093-		-43.9815

In Table 2. The values of ΔG, ΔS and ΔH since is both negative, then it's spontaneity, exothermic and less random.

Adsorption Isotherm:

Fig. 10. shows isothermal Congo Red dye adsorption on surface CuO NPs at acid function 8, temperature (288-318) K,0.0300g from the adsorbing Congo Red ,35mg/L of the Congo Red dye, equilibrium time 30min, this figure pointed that the adsorption stench increases with increasing the concentration of equilibrium for Congo Red. The isotherms getting from the experiments were identical to S-curve in form at Giles' discretion.

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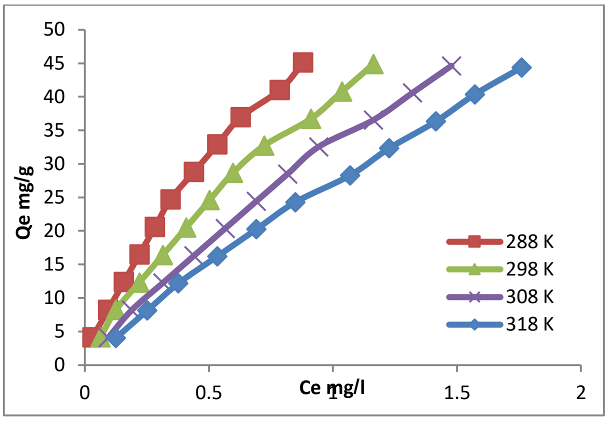

Figure 10. Isotherm adsorption for Congo Red dye from aqueous solution using surface the CuO NPs at different temp.

Isotherm Langmuir

Definition of Langmuir isotherm (Eq.5) states the adsorption process takes place across homogeneous sites of the adsorbent. [21].

$$Q_e = \frac{a\,b\,C_e}{1+b\,C_e} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\, 6$$

Where; Q_e = defined as the quantity of Congo Red adsorption at the time of equilibrium(mg/g). (a,b) are the constants of Langmuir.

$$R_L = \frac{1}{1+b\,C_0} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\, 7$$

Where :R_L = meaning refer to adsorption kinds is Irreversible(RL=0),Likely(0<R_L <1), linear(R_L=1) or (R_L >1). [21]. The (a) and (b) values are calculated from the slopes (1/a) and intercepts (1/ab) of linear plots of Ce /Qe versus Ce are shown in Fig. 11.

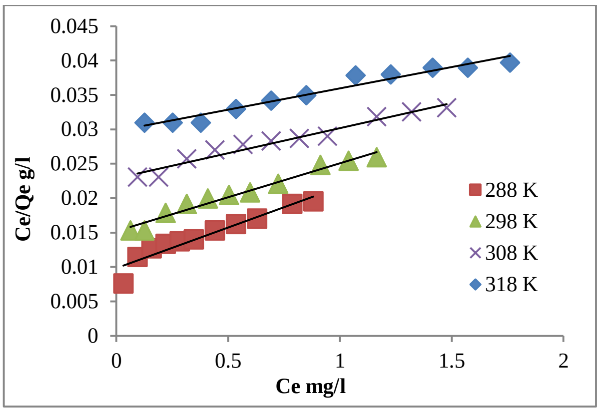

Figure 11. Langmuir isotherms for Congo Red dye using surface the CuO NPs at different temp.

Isotherm Freundlich

Freundlich isotherm pattern of adsorption states that it is multi-layered adsorption across heterogeneous active sites Freundlich isothermal. [22].

$$LogQ_e = Log\,K_F + \frac{1}{n}\,LogC_e \ldots\ldots\ldots\ldots\; 8$$

Where: k_F , n =Freundlich's constants [22]. Fig .12. show the applicability of the equation well when plotting Log Qe against the values of Log Ce

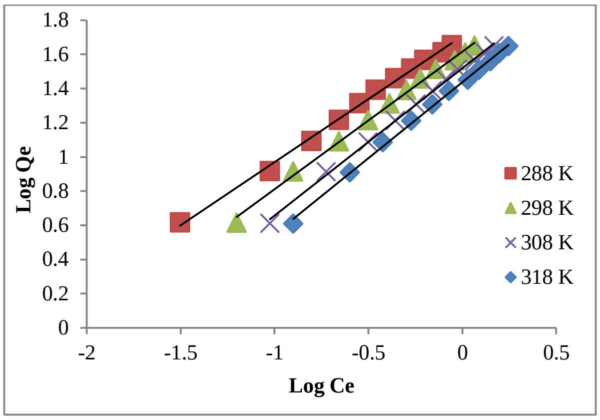

Figure 12. Freundlich isotherm Congo Red dye using surface the CuO NPs at different temp.

Temkin Isotherm

It is commonly used in the following way [23].

$$Q_e = \beta \ln A_T + \beta \ln C_e \ldots\ldots\ldots\; 9$$

Where: A_T = the binding constant for equilibrium.
β = connected to the adsorption heat [23].
where the curves of Temkin isotherm of Congo Red dye adsorption in Fig .13.

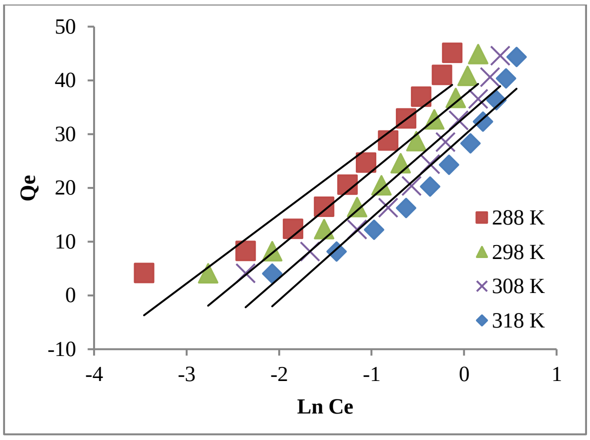

Figure 13. The Temkin isotherm for Congo Red dye using surface the CuO NPs at different temp.

Halsey isothermal

In multi-layered adsorption, the Halsey isothermal model is the very good.
The Halsey equation has the following linear form: [24].

$$\ln Q_e = \frac{1}{n_H}\ln K_H \frac{1}{n_H}\ln C_e \ldots\ldots\ldots\ldots\; 12$$

Where: KH and n_H are the Halsey isotherm constants. The graph will be between of lnQe vs. lnCe as shown in Fig .14.

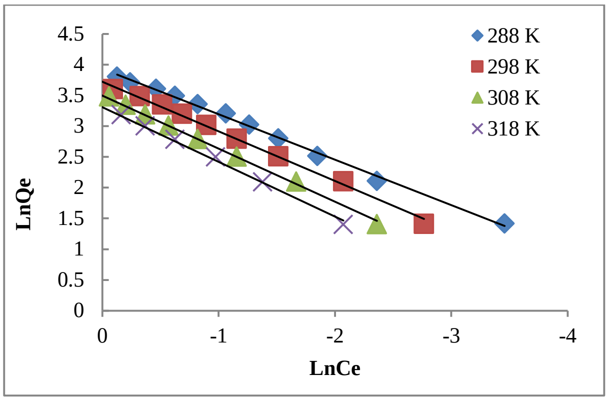

Figure 14. The Halsey isotherm for Congo Red dye using surface the CuO NPs at different temp.

The (a, b, R_L) for Langmuir constants, (n, K_F) for the Freundlich pattern, likewise the Temkin pattern constants (β, A_T) and the Halsey isotherm constants K_H and n_H with linear correlation coefficients shown in Table 3.

Table 3. Adsorption isotherm values each of Langmuir, Freundlich, Temkin and Halsey at (288 –318) K

Temp.(K)	Langmuir isotherm				Freundlich isotherm		
	-a(mg/g)	-b(mg/L	R^2	$-R_L$	K_F	n	R^2
288K	84.7457	1.2040	O.9171	0.0243	51.0387	1.3529	0.9965
298K	101.0101	0.6513	0.9692	0.0458	41.3142	1.2416	0.9965
308K	136.9863	0.3187	0.9604	0.0984	32.9382	1.1611	0.9981
318K	161.2903	0.2080	0.9556	0.1591	27.3275	1.1265	0.9978

Temp.(K)	Temkin isotherm			Halsey isotherm		
	β	A_T	R^2	n_H	K_H	R^2
288K	12.853	23.86495	0.8994	1.3529	204.5065	0.9965
298K	14.132	13.88048	0.9258	1.2416	101.5245	0.9965
308K	14.947	9.13533	0.9182	1.1611	57.85269	0.9981
318K	15.349	6.95237	0.9215	1.1265	41.52933	0.9978

From the results the (R^2) values in the table (3) for Langmuir, Freundlich, Temkin and Halsey, it turns out that the best results are in the Freundlich and Halsey values.[21,24].

Conclusions:

In this research, CuO NPs were prepared from *Anchusa strigosa* flower extract which is an adsorbent material that removes Congo red dye from water contaminants and industries that contain this dye, both medical and industrial, because this reaction was automatic and exothermic and the best results were obtained in Freundlich and Halsey at 30minutes, at a different temperature range, and the best weight was 0.030g.

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Authors' declaration:

- Conflicts of Interest: None.
- We hereby confirm that all the Figures and Tables in the manuscript are mine ours. Besides, the Figures and images, which are not mine ours, have been given the permission for re-publication attached with the manuscript.
- Ethical Clearance: The project was approved by the local ethical committee in University of Kerbala.

Authors' contributions statement:

S. A. K. wrote a part of the manuscript and collected the samples. E. T. K. Wrote another part of manuscript and interpretation the data. I. M. Sh. analyzed all Results. All Authors read the manuscript carefully and approved the final version of their MS.

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تخليق صديق للبيئة لدقائق النحاس النانوية من زهور لسان الثور ودراسة فعاليته للامتزاز

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الخلاصة:

تم تحضير دقائق أوكسيد النحاس النانوية من مستخلص ازهار لسان الثور. وقد تم تشخيص الدقائق النانوية المحضرة باستخدام تقنيات طيف الاشعة تحت الحمراء، حيود الاشعة السينية والمجهر الالكتروني الماسح. تم دراسة فعالية دقائق اوكسيد النحاس النانوية المحضرة لأستخدمها كمادة مازة لإزالة صبغة احمر الكونغو من الماء. وجد ان كفاءة الامتزاز تكون افضل عند الدالة الحامضية (3). وان اعظم سعة لامتزاز صبغة احمر الكونغو عند تركيز (35) ملغ/غرام . طبقت موديلات الامتزاز لانكماير، فريندلش ,تمكن وهلسي لدراسة التوازن لعملية الامتزاز. تم حساب الدوال الثرموديناميكية الطاقة الحرة،الانثالبي والانتروبي لعملية الامتزاز .

الكلمات المفتاحية: زهرة لسان الثور، صبغة الكونغو الحمراء، ايزوثيرم فريندلش، ايزوثيرم هلسي، ايزوثيرم لانكماير، ايزوثيرم تيمكن .