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Removal Congo Red Dye on Surface of Nano Charcoal Activated by Used Central Composite Chemometric Method

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Abstract. In this paper; the modern method was used to remove the congo red dye (CR) from its aqueous solutions by chemometric separation. The nano charcoal activated (NCA) was used to remove the CR and the factors affecting the adsorption process were studied simultaneously to reduce the number of experiments followed and to rely on central composite design and the method of response surface by used statistica 12 program. This gives the relationship between all the variables simultaneously. It was found that the removal efficiency reached to 98.371%.

Keyword: Chemometric Method, Congo Red Dye, Nano Charcoal Activated, Adsorption, and Simultaneously.

1.Introduction:

Environmental pollution in general and water pollution in particular is a problem of significant importance in many countries [1]. Water pollution hurts human health [2] as liquid pollutants such as heavy metal ions and dyes are among most critical diseases [3]. These dyes are used in many vital industries such as paper, fabric, tanning and painting [4]. Congo red dye (CR) is one of the known industrial dyes and it is one of the oldest dyes used, Figure (1) [5]. The molecular formula for this dye is (C₃₂H₂₂N₆Na₂O₆S₂) [6]. Despite the benefits of its use in the medical field and the food field, but when discharged into water it causes pollution [7]. There are many techniques to remove the dyes from water including the adsorption method as one of the most crucial methods [8]. Adsorption is used to remove dyes from water because it is simple to operate, cheap, and highly efficient [9]. Nano charcoal activated (NCA) is widely used as an adsorbent in separation processes due to its large surface area, low price and painless preparation [10].

(NCA) had stimulated research society to solve the problems of the environment [11]. This nano is very useful for removing by adsorption of various water, air and soil contaminants [12]. Moreover, the decline in water quality has led researchers to use new adsorbents to treat water with charcoal activated [13]. NCA is an excellent adsorbent as its surface that can be adjusted with the addition of other materials and has a high ratio of the volume to the surface area. In addition, it is relatively low cost of production and it is able to reuse [14]. Using coating, stabilizing, and functionalization (NCA) can be modified surface [15]. In the (NCA) surface coating process, clusters are not formed due to the particle dispersion when water molecules containing contaminants (hydrophilicity) are attracted [16]. Therefore, the surface modification of (NCA) will improve its ability to adsorb many pollutants such as dyes and ions of heavy metals [17]. The coating of surface phenomenon helps convert the geometry of closely-packed cubic to nanoparticles in compacts and robust [18].

In this study, the nano charcoal activated (NCA) was used to remove congo red dye (CR) from its aqueous solutions by chemometric separation, making the process less expensive, more productive and environmentally friendly after determining the optimum adsorption conditions.



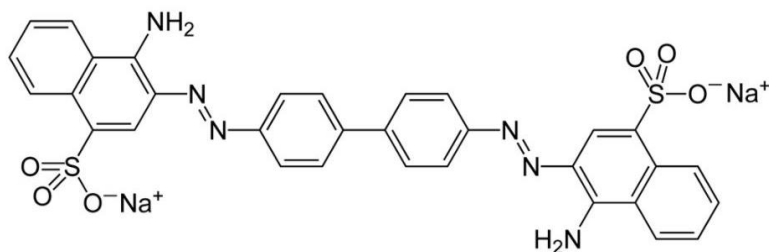


Figure 1. Chemical Structure of Congo Red Dye (CR)

2. The experimental part:

2.1. Chemicals and instruments:

Throughout this study, a high pure and analytical grade was used for all chemical substances and reagents, including nano charcoal activated (NCA) from (BDH), positive congo red dye (CR) from Sigma-Aldrich with purity 99.5%. During the experiments, ultra-pure water was used. Shimadzu UV-Vis Spectrophotometer 1650Pc was used for specifying the residual (CR) in the experiments. Adjusted pH by adding 0.01 M NaOH or 0.01 M HCl and measured by WTW pH meter InoLab 730. A shaker water bath from Amerexa was used to maintain a steady mixing temperature, and it was set to 150 rpm throughout the experiments. The stock solution of 100 mg/L of (CR) was prepared by dissolving 0.01000 g in a small volume of ultra-pure water and transferred to a 100 mL volumetric flask, diluted with water to a mark. Every day freshly prepares in the range of 30-0.05 mg/L solutions to create a calibration curve. The dye concentration in the aqueous solution was measured via the absorbance (Abs.) at $\lambda_{\max}$ equal to 522 nm by the Least-squares method.

$$[CR]_{mg/L} = \frac{Abs. - Intercept}{Slope} \quad (1)$$

2.2. The methodology of experiments:

In this study, all experiments were performed after an added amount of (CR) with nano charcoal activated in the Erlenmeyer flask, placed in the shaker water bath. In order to obtain the optimum conditions for the process of removing (CR) from its aqueous solutions, the effect of five different factors was studied simultaneously and at three levels as shown in table (1); the initial concentration of (CR), the amount of nano charcoal activated, pH, shaking time, and the temperature depended on central composite design [19] and the method of response surface [20] by used STATISTICA 12 program.

Table 1. Factors, Symbol and Coded level to Natural Level by Chemometric Separation

Factors	Symbol	-1	0	+1	Coded level to natural level
The initial concentration of (CR) (mg/L)	X1	10	20	30	10
Amount of Nano charcoal activated(g)	X2	0.2	0.4	0.6	0.2
pH	X3	2	6	10	4
Shaking time (min)	X4	15	25	35	10
Temperature (K)	X5	290	310	330	20

For the passage from coded variable level to natural variable level, the following equations were used: $X1 = ([CR]_{mg/L} - 20)/10$; $X2 = (NCA - 0.4)/0.2$; $X3 = (pH - 6)/4$; $X4 = (Time - 25)/10$ and $X5 = (Temperature - 310)/20$.

The adsorption process behaviour can be explained based on the empirical second-order polynomial model below [21]:

$$Response = \beta_0 + \sum_{i=1}^n \beta_i X_i + \sum_{i=1}^n \beta_{ii} X_i^2 + \sum_{i=1}^{n-1} \sum_{j=i+1}^n \beta_{ij} X_i X_j \quad (2)$$

Where β_0 is a constant, β_i is the linear coefficient, β_{ii} is the quadratic coefficient, and β_{ij} is the interaction effect coefficient. X_i and X_j are the symbolic values of the factors.

The amount of residual dye was measured at $\lambda_{\max}$. The quantity (CR) adsorbed onto nano charcoal activated q_e (mg/g) and the removal efficiency $R\%$, which were determined by the following equations:

$$q_e = (C_0 - C_e) \times \frac{V_L}{m_g} \quad (3)$$

$$R\% = \frac{C_0 - C_e}{C_0} \times 100 \quad (4)$$

If the initial concentration (CR) is C_0 (mg/L), the equilibrium concentration C_e (mg/L) of (CR), the Nano charcoal activated mass is m (g), and the solution volume is $V(L)$.

3.Result and Discussion:

3.1.Calibration curve for (CR):

Concentrations of (CR) ranged between (30.0-0.05) mg/L in their aqueous solutions were measured at $\lambda_{\max} = 522$ nm for creating the calibration curve, as in 'Figure 2' by the Least-squares method.

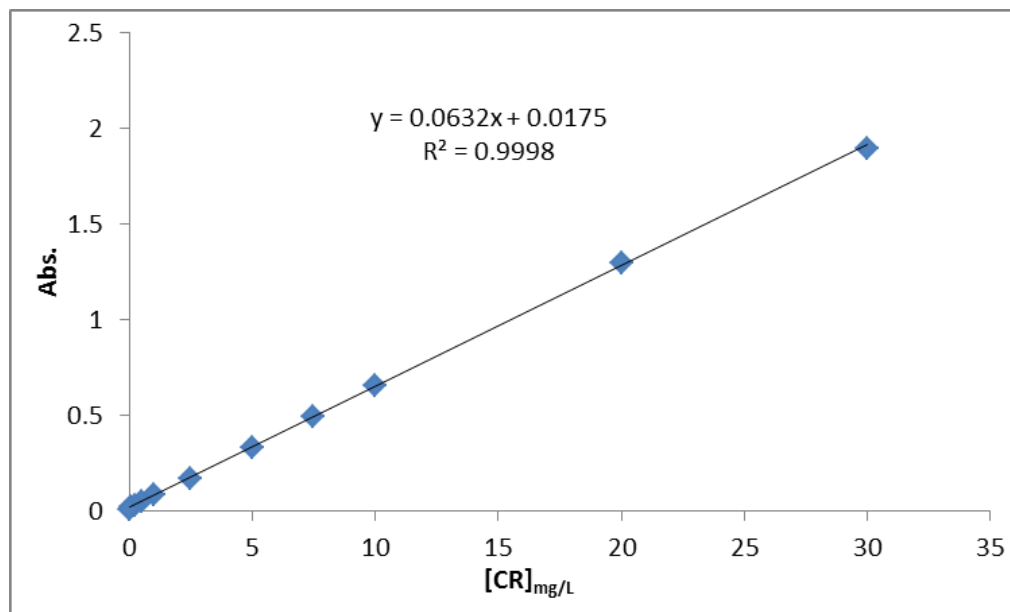


Figure 2. Calibration Curve for Congo Red Dye (CR)

3.2.The optimum conditions:

After studying simultaneously for Nano charcoal activated (NCA) to remove Congo Red dye (CR) from its aqueous solutions by chemometric separation in different conditions [22]; The following results were obtained in 'Figures 3-12'.

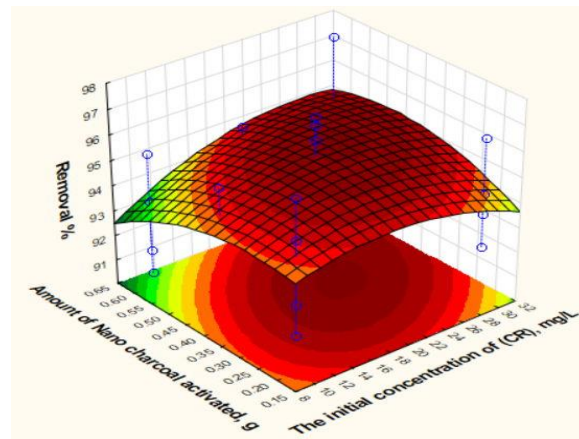


Figure 3. The relationship between the initial concentration of (CR) and amount of nano charcoal activated

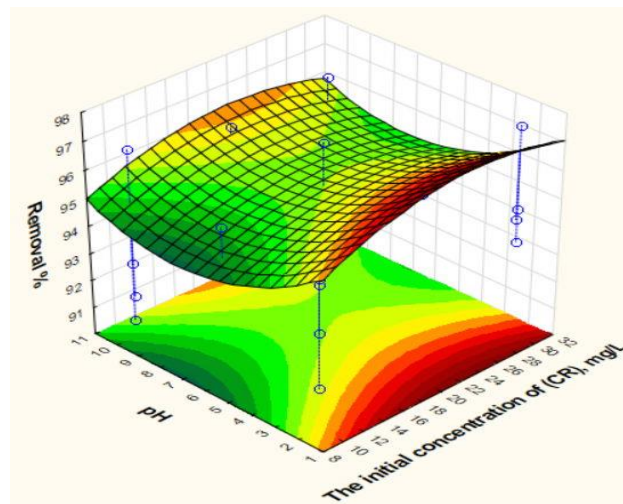


Figure 4. The relationship between the initial concentration of (CR) and pH

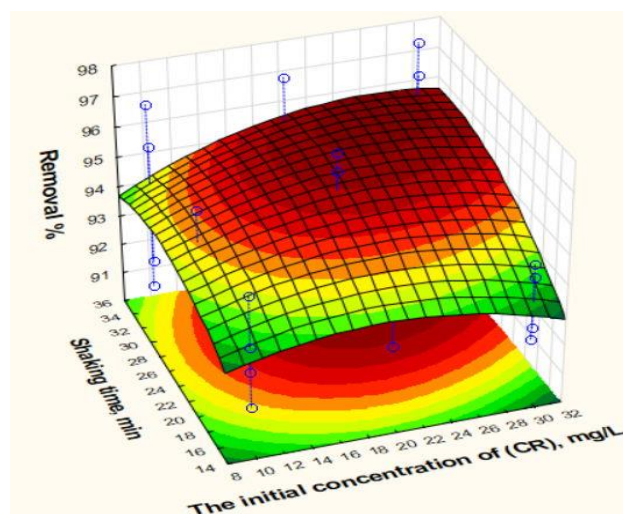


Figure 5. The relationship between the initial concentration of (CR) and Time

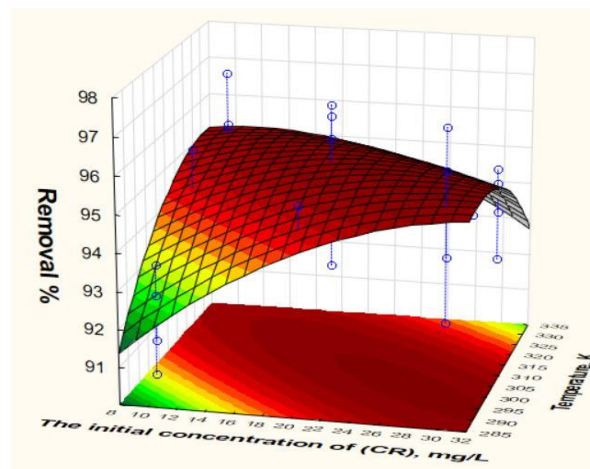


Figure 6. The relationship between the initial concentration of (CR) and temperature

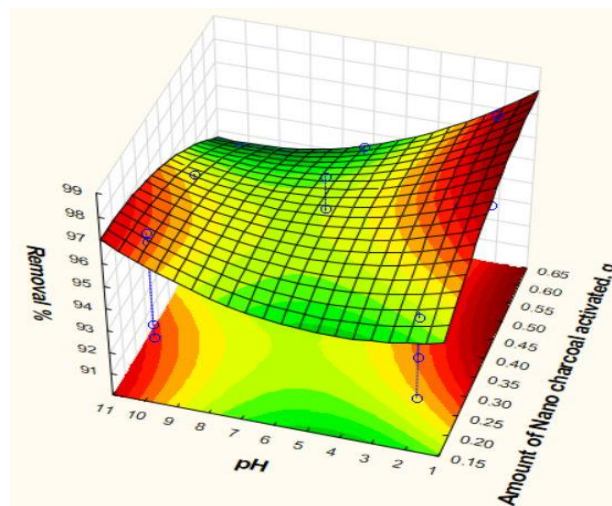


Figure 7. The relationship between amount of nano charcoal activated and pH

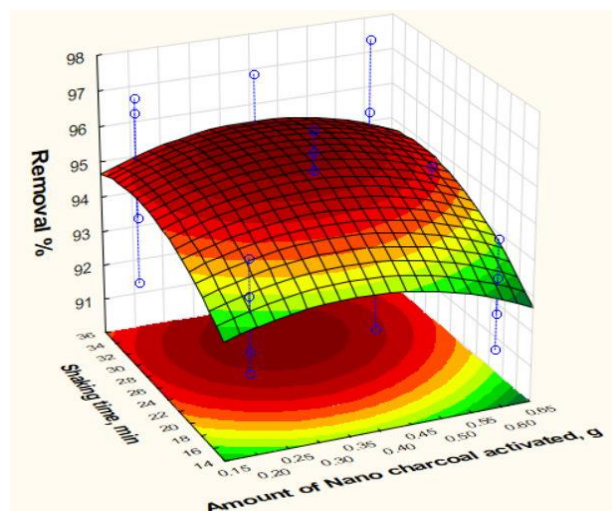


Figure 8. The relationship between amount of nano charcoal activated and Time

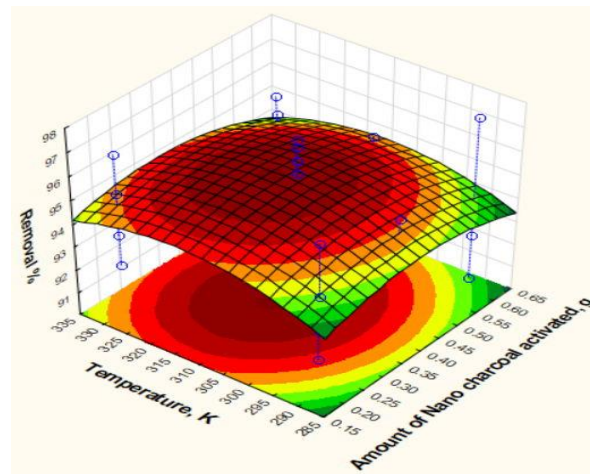


Figure 9. The relationship between amount of nano charcoal activated and temperature

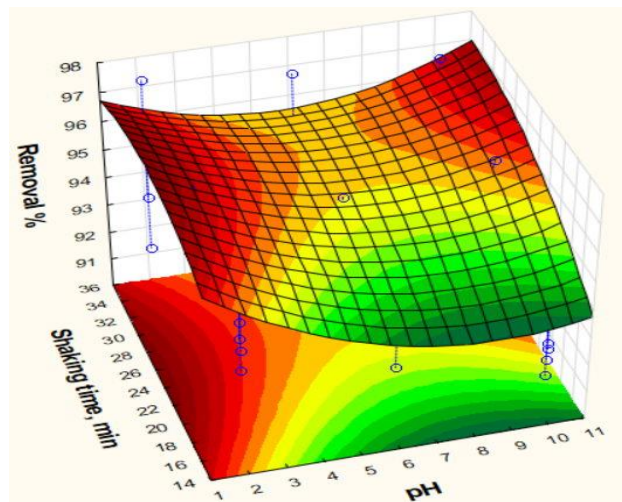


Figure 10. The relationship between pH and time

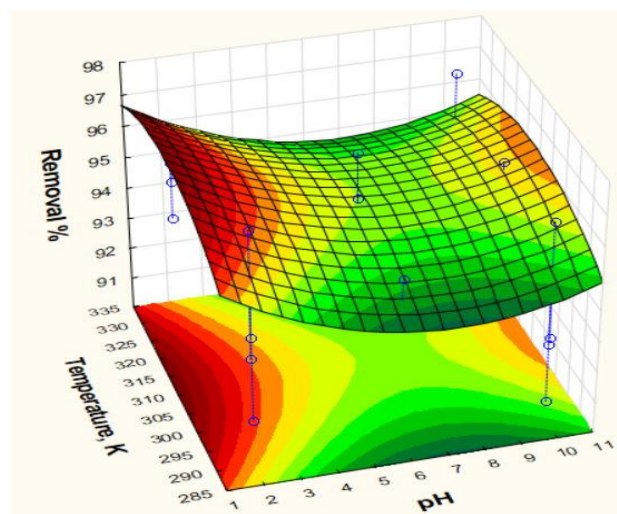


Figure 11. The relationship between pH and temperature

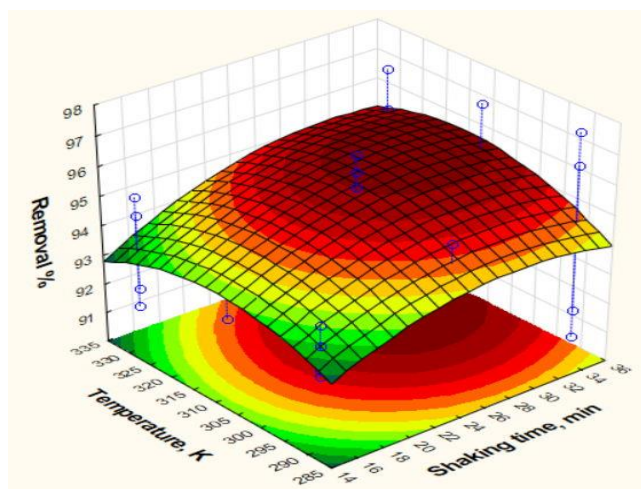


Figure 12. The relationship between time and temperature

By studying the relationship between the five variables with each other and using the statistica 12 program, optimal conditions were found for each factor [23] as shown in Table 2.

Table 2. Optimal conditions by chemometric separation for (CR)

Factors	Symbol	Optimal Conditions
The initial concentration of (CR) (mg/L)	X1	15.341
Amount of Nano charcoal activated (g)	X2	0.258
pH	X3	4.671
Shaking time (min)	X4	20.470
Temperature (K)	X5	309.455

When applying this model to the (CR) dye, it was found that the removal efficiency R% reached 98.371%, as shown in ' Figure 13 '.

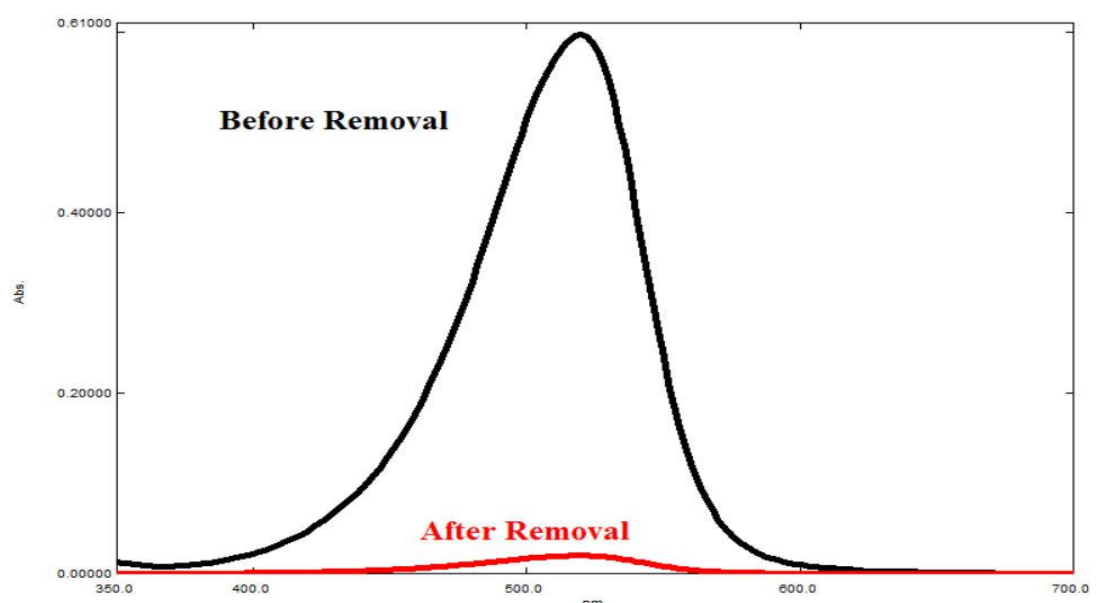


Figure 13. The removal efficiency of congo red dye (CR)**4. Conclusions:**

The chemometric separation method is one of the modern, easy and inexpensive methods as it depends on reducing the number of experiments and thus reducing pollution inside the laboratory. The method relied on the use of nano charcoal activated to remove the congo red dye (CR) from its aqueous solutions based on five factors which are the initial concentration of (CR), the amount of nano charcoal activated, pH, shaking time and the temperature depended on Box-Behnken design and the method of response surface by used statistica 12 program and to study the effect of these factors on each other at the same time. The obtained results are satisfactory and compared to the classic methods used in the process of removing dyes.

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