



# Modelling and Optimization of the Removal of Congo-Red Dye from Waste Water Using Agricultural Waste

Adepoju Tunde Folorunsho, Uzono Romokere Isotuk\*, Akwayo Iniobong Job

Department of Chemical and Petrochemical Engineering, Akwa-Ibom State University, Ikot Akpaden, Mkpatt Enin L.G.A., Nigeria

## Email address:

uzonoisotuk@yahoo.com (U. R. Isotuk), romokereuzono@aksu.edu.ng (U. R. Isotuk)

\*Corresponding author

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**Abstract:** The continuous utilization of dye in the industries and commodity products has necessitate its development by sustainable approach. However, for the success and commercialization of these products, their cost of production should be compared to the existing products available in the market. To do these, there is a need to introduce cheap feedstock for Congo red dye removal (CDRR). Its optimization will ease the process of production and give the optimum acceptable yield. Result showed that highest CDRR yield was 104.00 (mg/L) at pH( $X_1$ ) = 1, AD( $X_2$ ) = 0 and Time ( $X_3$ ) = -1, respectively. Box-Behnken response surface methodology (BBSRM) predicted a yield of 91.233 (mg/L) for CDRR at  $X_1 = -0.423$ ,  $X_2 = -1.00$  and  $X_3 = -1.00$  variables condition, which was validated as 90.87 (mg/L). ANN genetic algorithm predicted CDRR of 92.561 (mg/L) at variables condition  $X_1 = -0.567$ ,  $X_2 = -0.89$  and  $X_3 = -1.00$ , which was validated as 91.53 (mg/L). Modelling and optimization derived equations that showed the relationship between the CDRR and variables ( $X_1$ ,  $X_2$  and  $X_3$ ) in term of coded for RSM:  $CDRR(\%) = 92.67 + 0.72X_1 - 5.67X_2 + 6.50X_3 - 7.66X_1X_2 - 4.59X_1X_3 + 6.20X_2X_3 - 0.35X_1^2 - 1.14X_2^2 - 0.12X_3^2$ ; actual factors for ANN:  $CDRR(\%) = 55.70808 + 7.18508X_1 + 9.74067X_2 + 5.77729X_3 - 3.40222X_1X_2 - 1.22467X_1X_3 + 6.61867X_2X_3 - 0.039194X_1^2 - 2.02711X_2^2 - 0.078560X_3^2$ . The study concluded that agro waste is suitable feedstock for Congo red dye removal and the statistical software proved suitable for modelling and optimization.

**Keywords:** Congo- Red Dye, Agricultural Waste, Optimisation, Modelling, Response Surface Methodology, Artificial Neural Network

## 1. Introduction

Over the years waste water management has proven to be of great concern to industries that uses dyes to colour their products. These industries include; textile, paper, plastics, cosmetics etc. the textile industry is ranked as the highest consumer of dye. There are over 10000 types of dye available commercially with about  $7 \times 10^5$  tonnes produced annually across the world [1].

Congo red is a synthetic, Azo dye with wide range of industrial application in the textile, plastic and paper industries. Azo dyes are generally non-biodegradable because of their complex structure [2]. Hence, if large quantity of this dye is released into natural water bodies by industries it causes serious ecological in balance, the dye impedes light penetration into the water bodies thus

obstructing biological processes within the water body. Additionally, this dye is toxic to some aquatic organism causing direct destruction of the aquatic environment [3].

On decomposition, Congo-red dye forms cancer causing compounds [4] prohibited in some countries [5]. However, CR is still widely use in industries in other countries. Because of this environmental and health hazard posed by the use of CR it is necessary to remove residual CR from waste water from these industries before discharge to water bodies.

Different methods have been adopted to remove CR and other dyes from waste water before discharge to the environment. These methods include; membrane separation, chemical oxidation, reverses osmosis, coagulation and adsorption. Most of these methods are quite expensive and complex due to the non-biodegradable nature of dyes [6].

Researchers have resort to use of very cheap and readily available agricultural waste that requires less complex equipment to achieve efficient removal of these health threatening substance from waste water [7].

Researchers have harness the adsorptive property of agro waste to using them in adsorption studies. The use of agricultural waste as adsorbent in removal of pollutant (dye in waste water) has intensified over the years especially in the removal of dye from waste water [8]. This increase is as a result of the search for replacement of activated carbon which is very expensive and also poses high cost of regeneration, although has proved to be the most widely used adsorbent because of its micro-pore structure and wide surface area [1]. A good number of researchers have done lot of work on removal of various dyes from waste water using agricultural waste (Table 1). However, the agricultural waste utilized in this research is rice husk. Rice husk is the outer most protective covering of the rice grain sometimes called rice hull, it is separated from the rice grain by blowing air over the grains. Rice husk does not easily burn in open flame unless air is blown through the husk. Burning rice husk produces rice husk ash (RHA), but if the burning process is incomplete carbonized rice husk (CRH) is obtained.

**Table 1.** Adsorption capacity of different agricultural waste.

S/N	Adsorbent	Dye	Max. adsorption capacity[mg/g]	Reference
1	Orange peel	Aid violet	19.88	[9]
2	Rice husk	Acid yellow 36	86.9	[10]
3	Banana peel	Basic blue 9	20.8	[11]
4	Coir pith	Congo red	2.6	[12]
5	Oil palm fiber; activated carbon	Malachite green	149.35	[13]
6	Guava leaf powder	Methylene blue	185.2	[14]
7	Durian shell based activated carbon	Methylene blue	289.26	[15]
8	Almond shell	Direct red 80	90.09	[16]
9	Peanut hull	Reactive dye	55.5	[17]

Response Surface Methodology (RSM) and Artificial Neural Network (ANN) which has served as efficient mathematical techniques employed in modelling and optimization of industrial processes. RSM minimises the number of experimental runs and still provide results that are statistically accepted [18]. Artificial Neural Network (ANN) is a learning system based on a computational technique that illustrate a non-linear relationship between variable factors and responses by means of iterative training of data obtained from a designed experiment [19] ANNs show superiority as a modelling technique for data setting, data fitting and prediction abilities [20]. Several researchers have employed the use of RSM and ANN in modelling and optimization of various processes. [19] applied RSM in modelling and optimisation of *thevetia peruviana* (yellow oleander) oil biodiesel synthesis via *Musa paradisiacal* (plantain) peels as heterogeneous base catalyst. In another work, [20] used RSM in the optimization of *Sesamum indicum* oil biodiesel production. [21] Used ANN

to determined diesel engine performance and exhaust emission analysis using waste cooking biodiesel fuel. [22] Employed the use of ANN for prediction of the cetane number of biodiesel. The based prediction of performance and emission characteristics of a variable compression ratio CI engine using WCO as biodiesel at different injection timings using ANN was examined by [23].

Hence, this study employed the use of RSM and ANN in modelling and optimization of the removal of Congo-red dye from waste water using agricultural waste.

## 2. Materials and Method

### 2.1. Preparation of Rice Husk

Rice husk obtained from a local Market in Mkpato Enin L.G.A, Akwa Ibom State, Nigeria, was used as raw material for the production of the adsorbent. The rice husk was allowed to dry in an oven at a temperature of 70°C for 70 h in order to achieve better distribution; the dried rice husk was crushed using the grinder machine [retch] to obtain finer particles. The now powdered rice husk was sieved using a sieve of mesh size 1000 $\mu$ m. The sieved rice husk was acidified using 0.1 M sulphuric acid (H<sub>2</sub>SO<sub>4</sub>) and then heated on a magnetic stirrer at 100 °C till it formed a paste. The pH of the rice husk paste was adjusted to 7, this was achieved by washing the paste with deionised water. The sample was dried in an oven at a temperature of 80°C and labelled ‘modified rice husk’.

### 2.2. Preparation of CR Dye Solution

CR was obtained from a local supplier in Uyo, Akwa Ibom State and its stock solution was prepared in double- distilled water. All the test solutions were prepared by diluting the stock with distilled water.

0.001 g of Congo-red dye was dissolved in 1000 ml of distilled water and then mixed to obtain homogeneity. The dye sample is further diluted 10 times to obtain 100 ppm of the solution. In order to compare dye removal on the same basis the pH of all the samples were maintained at 7 using 0.1 M of HCl and 0.1 M of NaOH. In order to increase the volume of the sample too much and keep the error created by pH adjustment in a reasonable. The concentration of dye solution was determined by the use of a spectrophotometer operating in the visible range on absorbance mode. Absorbance values were recorded at the corresponding maximum absorbance wavelength and dye solution was initially calibrated for concentration in terms of absorbance units.

### 2.3. Batch Experimental Study

In this experiment, a batch adsorption technique was used to study the various effects of important parameters such as amount of adsorbent, pH values, the contact time between adsorbate and adsorbent. 0.01 g of Congo-red dye sample were taken then added to 1000 ml volumetric flask. A desired amount of adsorbent then added to the sample. This

experiment was carried out at room temperature. The solutions were then subjected to magnetic stirrer for proper adsorption. Samples were withdrawn from the shaker at different time intervals. Then the adsorbents were separated from the sample by using filter paper. The absorbance was measured for supernatant solution using UV-Spectrophotometer. For determining the uptake of the dye, all inclusive sets of experiments were performed at different time intervals (30, 60, 90, 120, 150 and 180 minutes) and pH (4-10). A range from 0.5 g to 2.0 g of adsorbent was also used to perform this experiment.

#### 2.4. Calibration Curve

The effect of initial concentrations of dye and adsorbent was investigated using 0.01 g of dye (Congo red) concentration in 1000 mL volumetric flask. Different concentrations such as 10, 8, 5, 3 and 1, respectively, were made into solutions, withdrawn and calculated for their predetermined intervals of 20, 16, 10, 6 and 2 were tested using a UV visible spectrophotometer at a wavelength of 495 nm. The respective adsorbance which gives 0.149, 0.140, 0.128, 0.124 and 0.118 were then used to calculate the change in concentration of Congo-red dye in the solution. From such, the mass of Congo- red dye was calculated and the values recorded

#### 2.5. Modelling of Experimental Design

A three-level-three factor Box-Behnken Design (BBD) response surface methodology (RSM) was employed for the modelling of experiment. 17 experimental runs were produced with selected variable factors such as: pH:  $X_1$ ; adsorbent dosage-AD (g/L):  $X_2$ ; time (h):  $X_3$ , respectively. Table 2 shows the coded variable levels and their factors considered for this experiment. The response results and variables were optimized by RSM and ANN statistically. The

two combined software (RSM and ANN) produced predicted yields, residual values, coefficient of determination, adjusted coefficient of determination, the mean values, the standard deviation as well as test of significant and ANOVA table. The modelling equation in terms of the variables considered ( $X_1$ ,  $X_2$ ,  $X_3$ ), the response ( $Y_F$ ) and the error ( $\varepsilon$ ) value is expressed in Eqn. (1).

$$Y_F = \rho_0 + \sum_{i=1}^k \rho_i X_i + \sum_{i=1}^k \rho_{ii} X_i^2 + \sum_{i < j}^k \rho_{ij} X_i X_j + \varepsilon \quad (1)$$

Table 2. Coded variables levels and their factors.

Variable	Symbol	Coded factor levels		
		-1	0	+1
Ph	$X_1$	4	7	10
AD (g/L)	$X_2$	180	200	220
Time (h)	$X_3$	40	45	50

### 3. Results and Discussion

#### Optimization of CRDRR by RSM and ANN

To optimize the CRDRR, design expert trial version DX10 and Neuralpower 21365 were employed. Table 2 shows the coded variables, the CRDRR result, the predicted and the residual values by RSM and ANN. Observation from the table showed the highest CRDRR yield was 104.00 (mg/L) at  $X_1 = 1$ ,  $X_2 = 0$  and  $X_3 = -1$ , respectively. The predicted values for RSM and ANN at these variable conditions were 105.20 and 104.00 mg/L. The lowest yield of 72.40 (mg/L) was obtained at  $X_1 = 1$ ,  $X_2 = 1$  and  $X_3 = 0$ . The predicted values for RSM and ANN at these variable conditions were 73.03 and 72.263 mg/L. Table 2 shows low residual value which is the differences between the CRDRR and the predicted values. The low value of residual, account for the perfectly fitted straight plots observed in the graph of predicted against the actual (Fig. 1).

Table 3. Coded variables, CRDRR result, predicted and residue values (RSM & ANN).

Std. run	$X_1$	$X_2$	$X_3$	CRDRR (mg/L)	Predicted RSM ANN		Residual RSM ANN	
1	-1	-1	0	92.70	92.67	93.676	0.032	0.40064
2	1	-1	0	95.42	94.83	95.422	0.59	1.1245
3	-1	1	0	101.57	102.57	102.57	-1.00	0.01565
4	1	1	0	72.40	73.03	72.263	-0.63	0.063193
5	-1	0	-1	92.00	91.00	90.962	1.00	0.18212
6	1	0	-1	104.00	105.22	104.00	-1.22	0.027424
7	-1	0	1	98.00	97.37	97.85	0.63	0.02001
8	1	0	1	93.00	92.67	92.822	0.33	0.002266
9	0	-1	-1	97.00	96.78	97.394	0.22	0.39401
10	0	1	-1	93.00	92.67	92.212	0.33	0.18809
11	0	-1	1	93.66	92.44	93.62	0.22	0.69528
12	0	1	1	88.84	88.47	88.439	0.37	0.043449
13	0	0	0	98.22	98.44	98.625	-0.22	0.37461
14	0	0	0	79.78	80.37	79.781	-0.59	0.074612
15	0	0	0	92.14	92.67	92.625	-0.53	0.48539
16	0	0	0	92.50	92.67	92.625	-0.17	0.37461
17	0	0	0	78.20	78.57	78.125	-0.37	0.12539

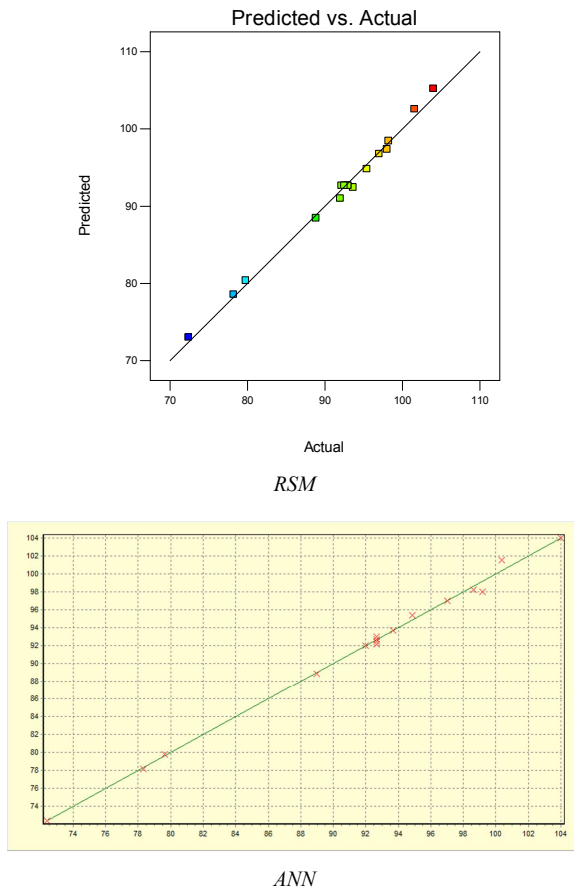


Fig. 1. Plots of predicted against the actual.

Shown in Table 4 is the test of significance results for the regression coefficient. To evaluate the significance of regression, F-value and p-values using Fischer's and null-hypothesis tests were employed. Large F-value with P-value < 0.05 indicates

significant model terms, but a larger F-value with P-value  $\leq 0.0001$  implies a remarkably significant. In this study, considering all the variable terms, the P-values and larger F-values of  $X_2$ ,  $X_3$ ,  $X_1X_2$ ,  $X_1X_3$  and  $X_2X_3$  were found significant. All other terms with low F-values were not significant due to P-values  $> 0.05$ . However, the P-values  $< 0.0001$  of  $X_3$  with the largest F-value of 321.42 is the most remarkably significant model term.

Table 4. Test of Significance for Every Regression Coefficient.

Source	Sum of squares	df	Mean Square	F-value	P-value
$X_1$	4.16	1	4.16	3.95	0.0871
$X_2$	257.19	1	257.19	244.29	< 0.0001
$X_3$	338.39	1	338.39	321.42	< 0.0001
$X_1X_2$	234.40	1	234.40	222.64	< 0.0001
$X_1X_3$	84.36	1	84.36	80.13	< 0.0001
$X_2X_3$	154.01	1	154.01	146.28	< 0.0001
$X_1^2$	0.52	1	0.52	0.50	0.5033
$X_2^2$	5.47	1	5.47	5.20	0.0566
$X_3^2$	0.063	1	0.063	0.060	0.8131

Table 5 shows the results of analysis of the variance for regression equation model (ANOVA). The model with F-value = 1078.88 implies the model was significant (P-value < 0.0001), and the data obtained fitted best to the chosen quadratic model with mean -1.956 (ANN) and 91.91 (RSM) and standard deviation of 1.03 (RSM) and 2.817 (ANN), respectively. The coefficient of determination ( $R^2$ ) were 0.9932 and 0.99878, adjusted coefficient of determination ( $R^2$  Adj.) were 0.9845 and 0.99754, indicated a greater dependability between the CRDRR results and predicted values.

Table 5. Analysis of Variance (ANOVA) of Regression Equation.

Source	Sum of squares	Df	Mean Square	F-value	p-value
Model	1078.88	9	119.88	113.86	< 0.0001
Residual	7.37	7	1.05	-	-
Lack of Fit	6.84	3	2.28	17.26	0.0094
Pure Error	0.53	4	0.13	-	-
Cor Total	1086.25	16	-	-	-

RSM: Mean = 91.91, S. D = 1.03, R-Sq. = 0.9932, R-Sq. (adj.) = 0.9845

ANN: Mean = -1.956, S. D = 2.817, R-Sq. = 0.99878, R-Sq. (adj.) = 0.99754:

Regression coefficients and significance of response surface quadratic results are shown in Table 6, the variance inflation factor (VIF) 1.00-1.01 exhibited the orthogonal effects of centre points with variable factors considered. The optimization regression coefficients and significance of response surface quadratic equation derived shows the

relationship between the CRDRR and variables ( $X_1$ ,  $X_2$  and  $X_3$ ) in term of coded for RSM and actual factors for ANN are expressed in Eqns. (2) and (3).

Coded RSM

$$CRDRR(\%) = 92.67 + 0.72X_1 - 5.67X_2 + 6.50X_3 - 7.66X_1X_2 - 4.59X_1X_3 + 6.20X_2X_3 - 0.35X_1^2 - 1.14X_2^2 - 0.12X_3^2 \quad (2)$$

Actual ANN

$$CRDRR(\%) = 55.70808 + 7.18508X_1 + 9.74067X_2 + 5.77729X_3 - 3.40222X_1X_2 - 1.22467X_1X_3 +$$

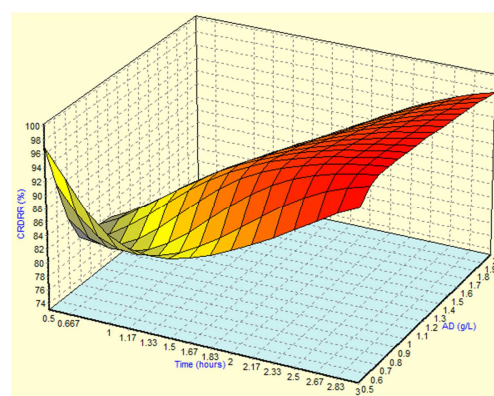
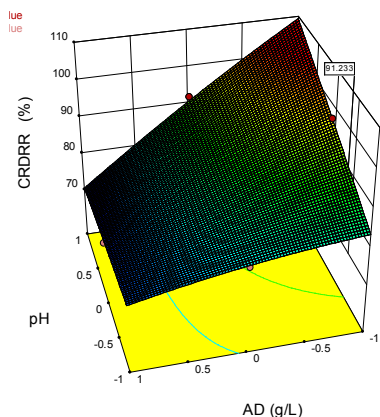
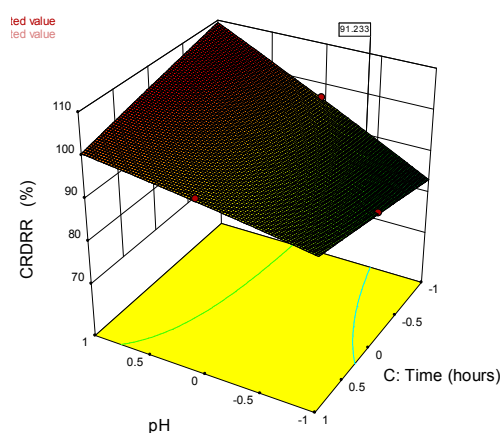
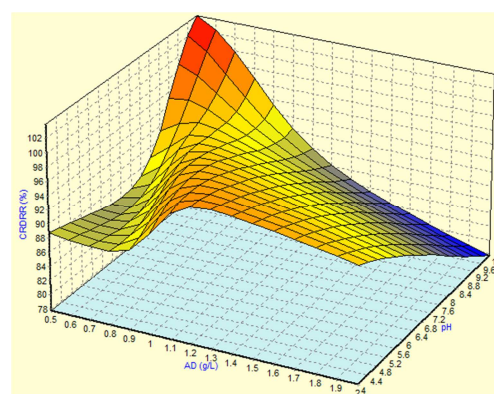
$$6.61867X_2X_3 - 0.039194X_1^2 - 2.02711X_2^2 - 0.078560X_3^2 \quad (3)$$

Table 6. Regression Coefficients and Significance of Response Surface Quadratic.

Factor	Coefficient Estimate	RSM ANN	df	Standard Error	95%CI Low	95%CI High	VIF
Intercept	92.67	55.70808	1	0.46	91.58	93.75	-
X <sub>1</sub>	0.72	7.18508	1	0.36	-0.14	1.58	1.00
X <sub>2</sub>	-5.67	9.74067	1	0.36	-6.53	-4.81	1.00
X <sub>3</sub>	6.50	5.77729	1	0.36	5.65	7.36	1.00
X <sub>1</sub> X <sub>2</sub>	-7.66	-3.40222	1	0.51	-8.87	-6.44	1.00
X <sub>1</sub> X <sub>3</sub>	-4.59	-1.22467	1	0.51	-5.81	-3.38	1.00
X <sub>2</sub> X <sub>3</sub>	6.20	6.61867	1	0.51	4.99	7.42	1.00
X <sub>1</sub> <sup>2</sup>	-0.35	-.039194	1	0.50	-1.54	0.83	1.01
X <sub>2</sub> <sup>2</sup>	-1.14	-2.02711	1	0.50	-2.32	0.042	1.01
X <sub>3</sub> <sup>2</sup>	-0.12	-0.078560	1	0.50	-1.31	1.06	1.01

Shown in Fig. 1 are the 3D plots representing the effect of variable factors on the CRDRR while keeping one factor constant at zero level per plot. Observation from the plots shows that there was a high mutual interaction between pH and AD (X<sub>1</sub>X<sub>2</sub>), between the time and AD (X<sub>2</sub>X<sub>3</sub>), and low-interaction between the pH and time (X<sub>1</sub>X<sub>3</sub>). Meanwhile, variables factors considered during experiment contribute differently to the response. Some may contribute greatly; other may have little impact, while some showed no significant contribution. In this study, the contribution of each variable to the CRDRR is depicted in Fig. (3). Adsorbent dosage (AD) contributed greatly with 45.87%, PH with 28.37% while time contributed a percentage of 25.76%.

Result of optimization analysis further showed that the BBD RSM predicted a yield of 91.233 (mg/L) for CRDRR at X<sub>1</sub> = -0.423, X<sub>2</sub> = -1.00 and X<sub>3</sub> = -1.00 variables condition, which was validated by carrying out three experimental runs, and an average CRDRR of 90.87 (mg/L) was obtained. Similarly, ANN generic algorithm predicted CRDRR of 92.561 (mg/L) at variables condition X<sub>1</sub> = -0.567, X<sub>2</sub> = -0.89 and X<sub>3</sub> = -1.00, which was validated by carrying out another three experimental runs, and an average contents of 91.53 (mg/L) CRDRR was observed. The results revealed that both softwares are good for modelling and optimization techniques with their average validation CRDRR yield well within the range predicted.



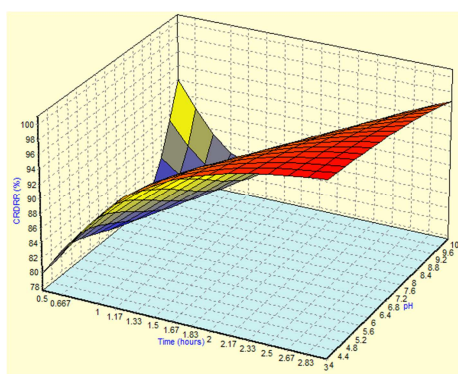
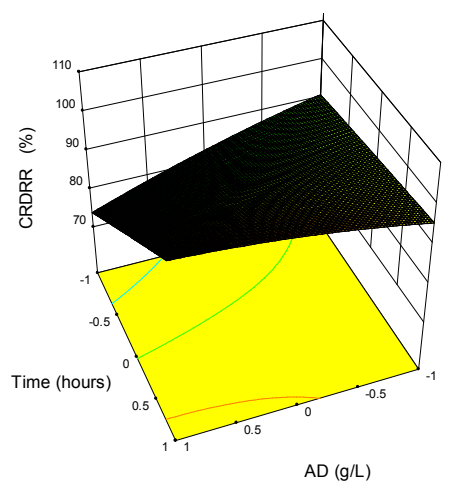


Fig. 2. RSM and ANN 3-D's plots.

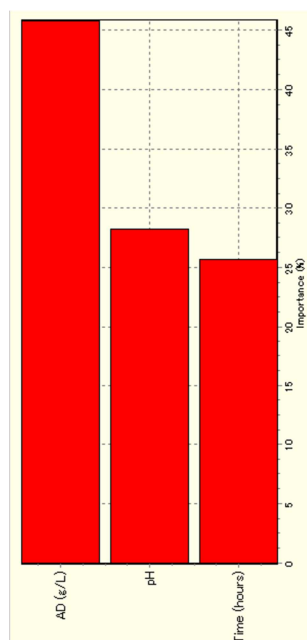


Fig. 3. Level of importance contribution.

## 4. Conclusion

This study focused on modelling and optimization of the removal of Congo-red dye from waste water using agricultural waste using statistical software. The results of this study showed that agro waste is suitable feedstock for

Congo red dye removal and the statistical software proved suitable for modelling and optimization. But, ANN show superiority over RSM in terms of optimum prediction and coefficient of determination.

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