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An Investigation for the Removal of Indigo Caramine Dye Using Biosynthesized Zinc Oxide Nano Particles with *Lantana camara* Leaves

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Abstract: A tiny particle in the range from 1 to 1000 nanometres in size is defined as a nanoparticle. Zinc oxide nanoparticles were manufactured from fresh leaves extract and is used to decolourize Indigo Caramine dye from aqueous solution. It is a cost economical process. Various parameters like contact time, pH, dye concentration and nanoparticle dosage were studied for dyes removal. The results showed that the optimum conditions for I.C dye removal were: contact time 40 min and pH–5, Concentration of 20 mg/L and dosage 1.6 g/L. The present study also includes isotherms like Langmuir, Freundlich and Temkin. This study also incorporated Lagergren first order and Pseudo second order kinetics. The total experimental data showed that the zinc oxide nanoparticles could be used as an efficient sorbent for decolorization of dyes, particularly Indigo Caramine dye from synthetic waste waters.

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Introduction

Global challenge in the present days is the availability of pure water for both domestic and industrial applications. Wastewater treatment for numerous applications may include diverse treatment steps that are intended to evacuate contaminants before delivery. Considering the significance of wastewater treatment universally, we have to grow new innovations for financially savvy, basic, easy to use, hearty and productive frameworks. On this premise, nanotechnology specifically could be one of the likely devices in wastewater treatment to be cost effective, simple, user-friendly, robust and efficient systems. Present study is designed in such a way that, removal of Indigo caramine dye from synthetic solutions using biosynthesized zinc oxide nano particles with lantana camara leaves.

Materials and Methods

The present experimentation is carried out in batch process, for removal of IC dye from aqueous solutions by using Lantana Camara leaves with Zinc Oxide nano particles (ZNO-NPS-LC).

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Reagents and Materials

Analytical grade chemicals were used for experimentation and need no further purification. Double distilled water is used to prepare all stock and synthetic solutions. From a stock solution containing 1000 mg of IC dye in 1.0 litre, the synthetic solutions of dyes were made. By addition of 0.l M HCl and 0.l M NaOH solutions the pH of IC dye was adjusted to the desired value.

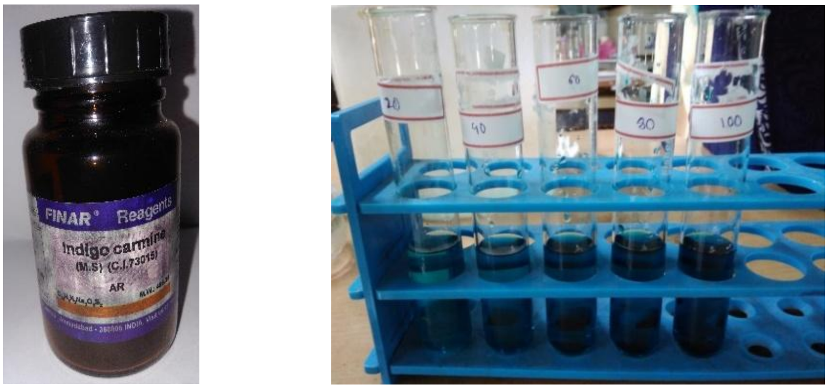

Figure 1. Indigo Caramine dye and different concentrations

Preparation of the Broth solutions and Nano particles formation

Preparation of *Lantana camara* broth

In this process 10 gm of fresh and cleaned leaves of LC are taken in a magnetic stirrer and to this 110 ml of distilled water is added and it is heated at 60^0C for 10 min. After that the solution is filtered in 250 ml conical flask using Whatmann's filter paper and it is kept aside for further process. The broth obtained is in pale yellow colour.

Preparation of Nano Particles

In this procedure 10 ml of stock arrangement is taken and to 5 gms of Zn (NO3)2.6H2O is included a 250 ml conical flask and is kept in an orbital shaker for 24 hrs so as to acquire nano particles. The nano particles arrangement is seen when the light yellow shading is changed to dull earthy colored brown shading. This solution is then centrifuged to obtain a NPS pellet which is later dried and powdered. Similar multiple experiments are conducted for obtaining huge mass of NPS powder. This final NPS powder is utilized for color removal procedures at different concentrations, pH, dosages and Temperatures.

Figure 2. *Lantana camara* leaves and Broth

Results and Discussion

Effect of Contact Time

The % removal of IC Dye was studied as a function of contact time at room temperature. 50 ml of 20 mg/L Dye solution was taken with 0.04g of Zinc oxide nano particles using *Lantana camara*(ZNO-NPS-LC) solution at different time intervals ranging from 1 to 180 min. At the start, the ions adsorbed and occupied selectively the active sites on the ZNO-NPS-LC solution. As the contact time increased the active sites on the ZNO-NPS-LC were filled.

The rate of sorption became gradually slower and reached an exhaust stage, resulting constant value. The results obtained are shown in figure 3. As a result of the experiment, the highest % removed for the IC Dye was 60% at the time of 40 min [01].

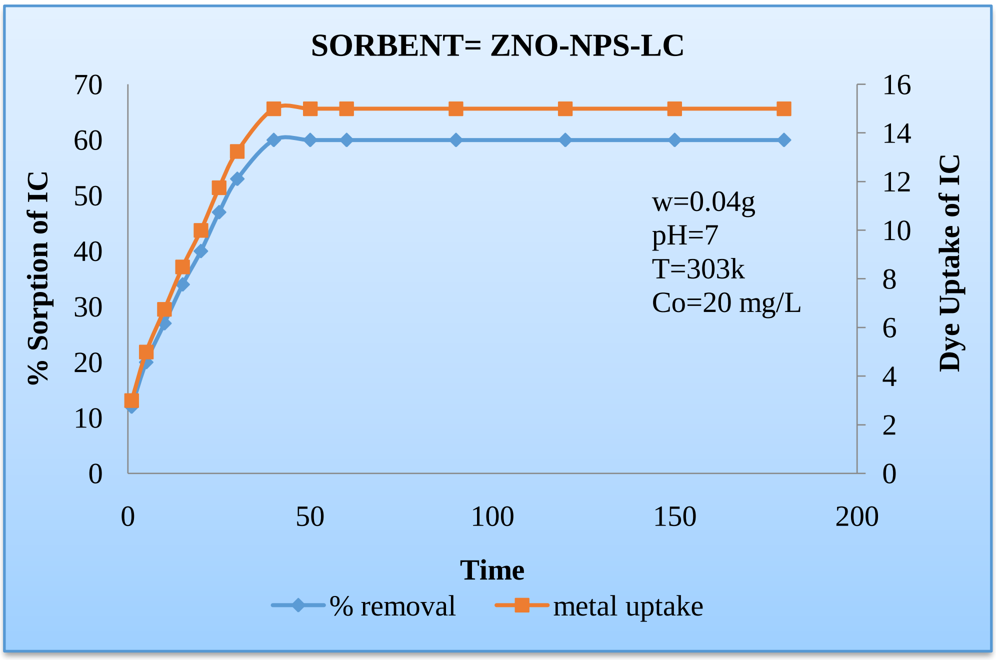

Figure 3. Effect of time

Effect of pH

The pH parameter has been identified as one of the most important parameter that is effective on Dye decolourization. In order to find the effect of pH on % removal using the ZNO-NPS with *Lantana camara* broth, experiments have been carried out at various initial pH values and results are given in figure 4. The removal was increased from 52 % to 70 % as pH was increased from 2 to 5 whereas further increase in pH had a negative effect. The maximum % removal was found to be 70 % at pH 5 for IC Dye. Therefore, the remaining all experiments were carried out at this pH value [02].

At the initial stage, the degree of effluent ionization, which acts as weak acid in aqueous solutions, as a function of pH value seems to be independent and attains an equilibrium state with increasing the pH values from upto 5. Hence, the sorption is slightly affected over pH range amounts to the dissociation constant value of solution. At higher values (pH >5), the degree of effluent ionization increase and the adsorbent surface becomes negatively charged. Owing to the repulsive forces prevailing between adsorbent surface and solute molecules, the sorption extent decreases. This avouches that the solution pH value has adverse effect on the adsorb ability of solution at values greater than 5 pH.

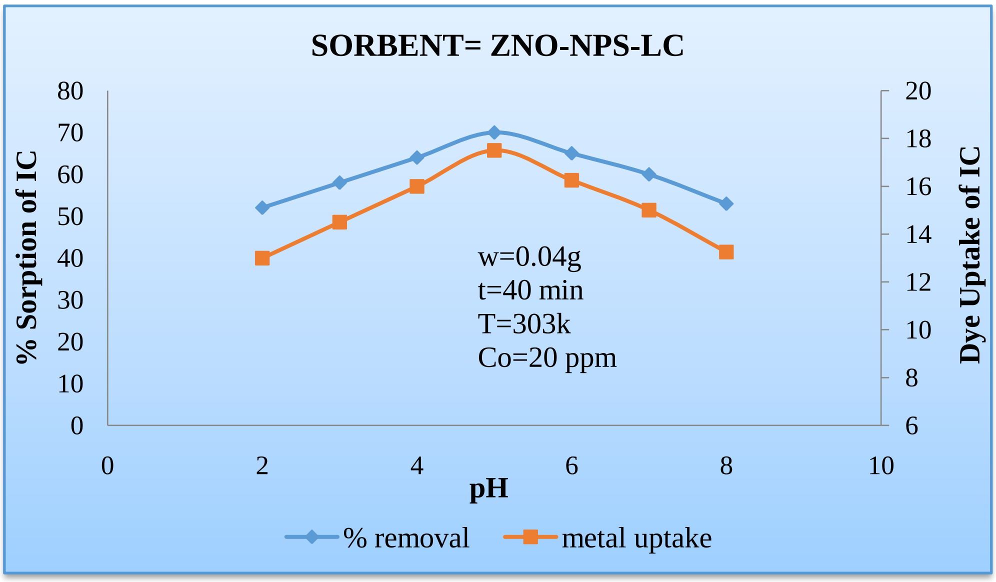

Figure 4. Effect of pH

Effect of Concentration

The percentage removal of dyes at various initial concentrations is depicted in figure 5. At concentration of Dye solution (20 mg/L), maximum % removal is obtained for IC Dye using ZNO-NPS-LC and on further increase in concentration (200 mg/L), % removal has been decreased. The capacity of % removal is maximum at a concentration of 20 mg/L. This is due to higher interaction between ZNO-NPS-LC powder and the IC Dye solution. The maximum removal of IC Dye is 70% [03].

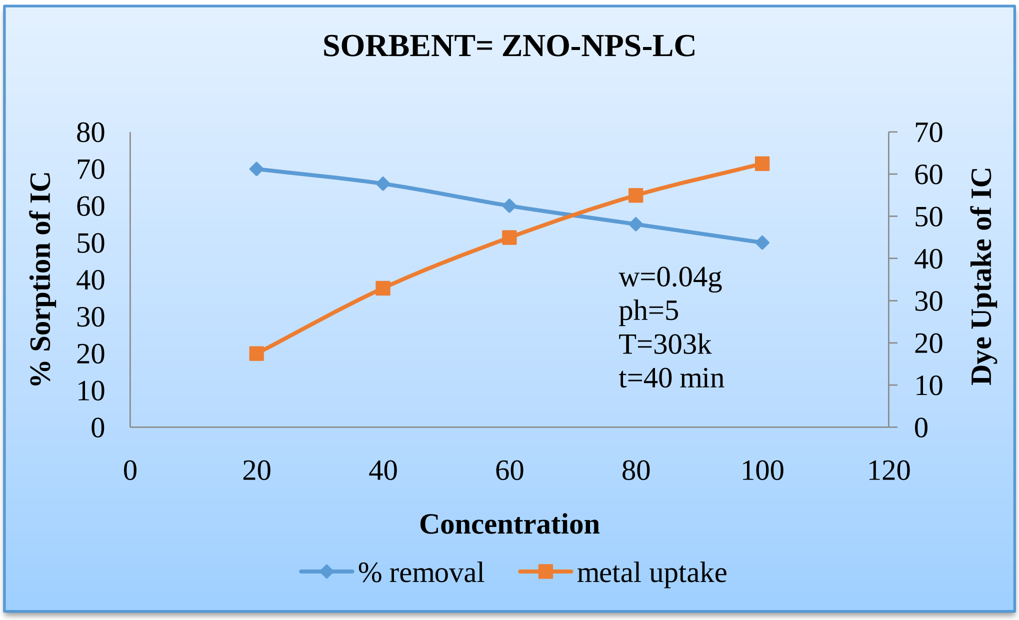

Figure 5. Effect of concentration

Effect of Dosage

The variations of percentage removal of IC Dye were studied using different dosages of ZNO-NPS-LC. Results from the figure 6 showed that % removal of IC Dye increased and uptake decreased with increase in dosage. The maximum % removal is attained at 0.08 g and was almost constant at higher dosages. This trend could be explained as a consequence of partial aggregation. Therefore, the optimum dosage was selected as 0.08 g for further experiments. The maximum % removal of IC dye is 80%. This trend can be predicated to larger surface area and availability of more sorption sites [04].

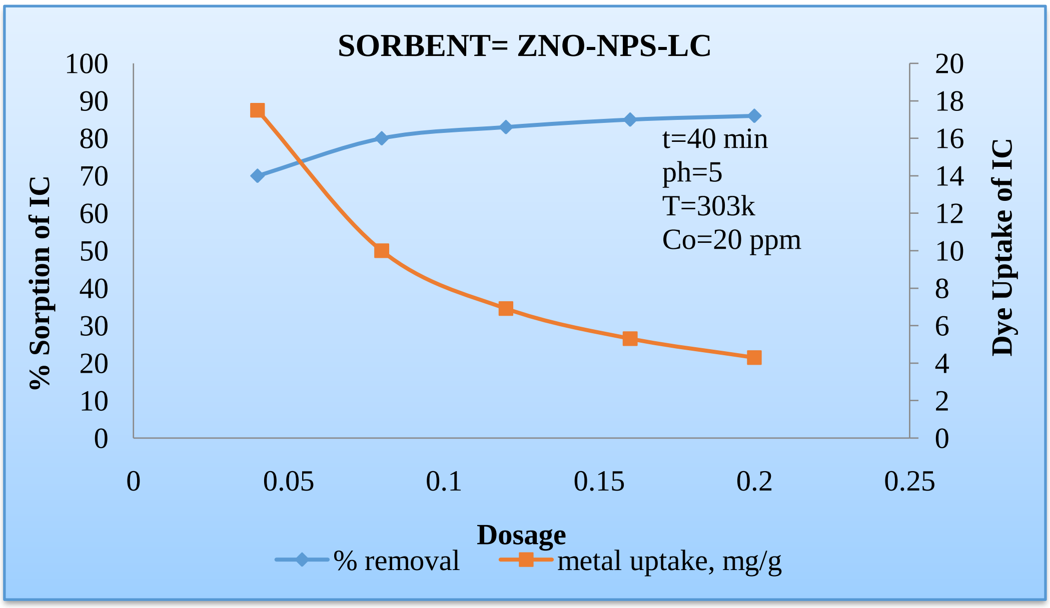

Figure 6. Effect of dosage

Effect of temperature

The dependence of temperature on the % removal of IC dye is investigated at different temperatures as given in figure 7. Results from figure 7 showed that %removal of IC dye increased from 69% to 80% with increase in temperature from 283 K to 303 K. This indicates that the % removal of Dyes using ZNO-NPS-LC was controlled by an endothermic process. The increase in removal with temperature may be attributed to either increase in the number of active surface sites available for interaction on the Dyes. The maximum % removal of IC dye is 80 % [05].

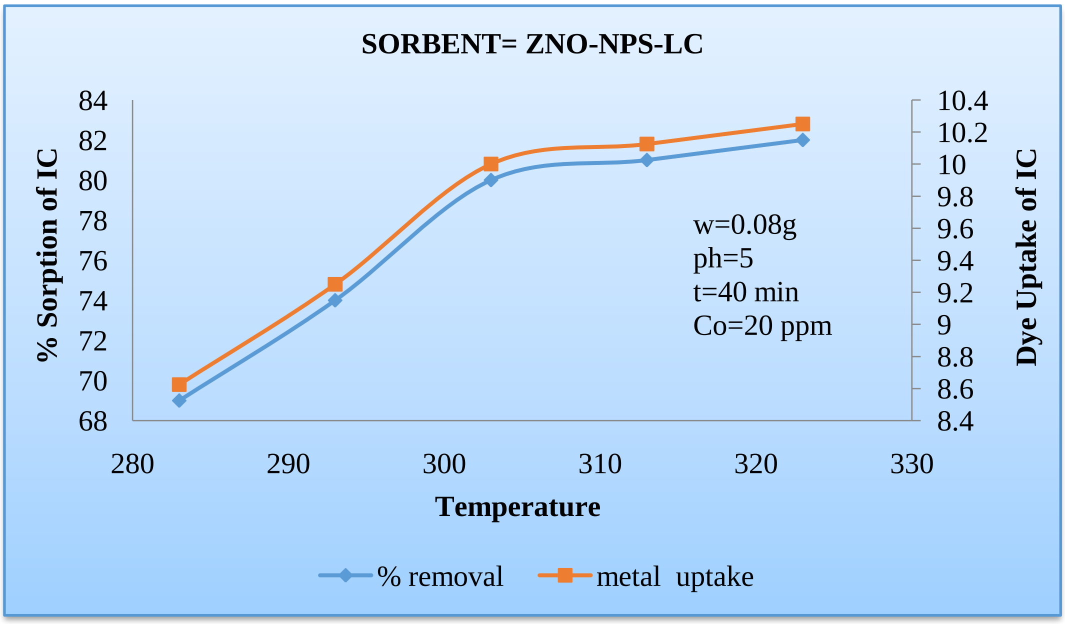

Figure 7. Effect of temperature

Isotherms

Irving Langmuir [06] developed an isotherm named Langmuir isotherm. It is the most widely used simple two-parameter equation. This simple isotherm is based on following assumptions:

- ✓ Biosorbates are chemically biosorbed at a fixed number of well-defined sites
- ✓ Each site can hold only one biosorbate species
- ✓ All sites are energetically equivalent
- ✓ There are no interaction between the biosorbate species

The Langmuir relationship is hyperbolic and the equation is:
qe/qm = bCe/(1+bCe)

Equation can be rearranged as
(Ce/qe) = 1/(bqm) + Ce/qm

From the plots between (Ce/qe) and Ce, the slope {1/ (bqm)} and the intercept (1/b) are calculated. Further analysis of Langmuir equation is made on the basis of separation factor, (RL) defined as RL = 1/ (1+bCe)

0< RL<1	indicates	favorable sorption
RL>1	indicates	unfavorable sorption
RL = 1	indicates	linear sorption
RL = 0	indicates	irrepressible sorption

Langmuir isotherm is drawn for the present data and shown in Figure 8. The equation obtained 'n' Ce/qt = 0.005 Ce + 0.201 with a good linearity (correlation coefficient, R^2~0.986) indicating strong binding of IC dyes to the surface of ZNO-NPS-LC powder.

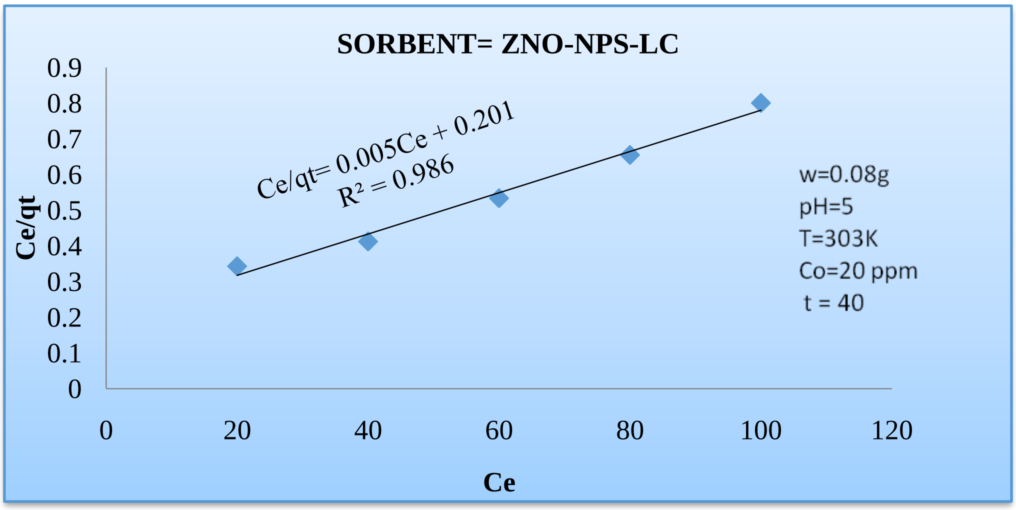

Figure 8. Langmuir isotherm for % sorption of IC dye

Freundlich isotherm

Freundlich [07] presented an empirical sorption isotherm equation, that can be applied in case of low and intermediate concentration ranges. It is easier to handle mathematically in more complex calculations.

The Freundlich isotherm is given by

qe = KfCen

whereKf (mg) represents the sorption capacity when dye equilibrium concentration and n represents the degree of dependence of sorption with equilibrium concentration Taking logarithms on both sides, we get

logqe = log Kf+ n log Ce

Freundlich isotherm is drawn between lnce and log qe in Fig. 9 and the resulting equation is lnCe = 0.6 lnCe + 1.851. The resulting equation has a correlation coefficient of 0.983.

The 'n' value in the above equations satisfies the condition of 0<n<1 indicating favorable sorption.

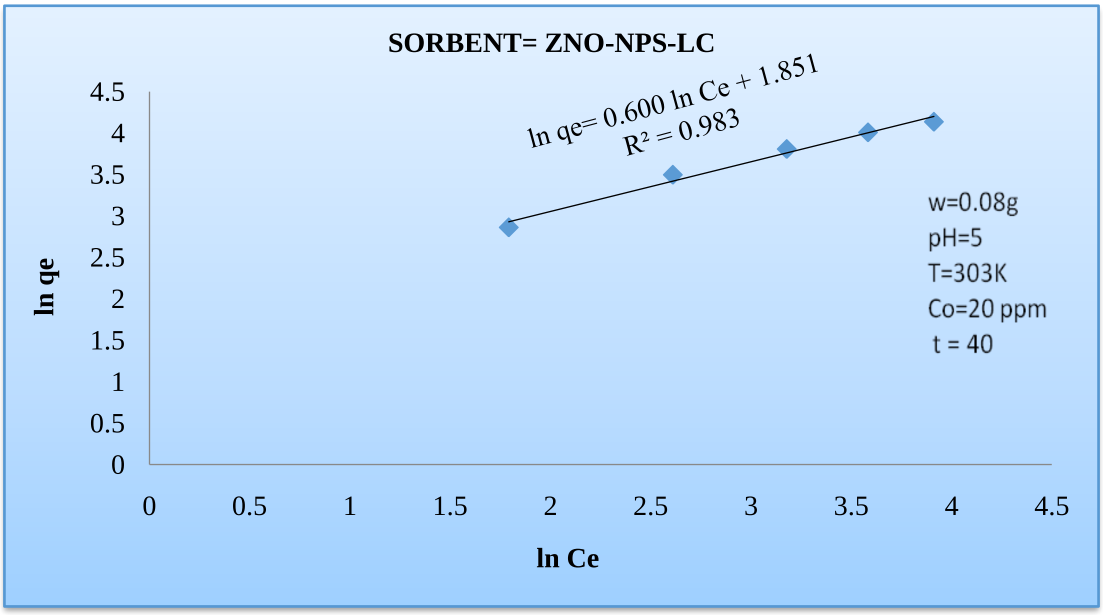

Figure 9.Freundlich isotherm for % sorption of IC dye

Temkin isotherm:

Temkin and Pyzhev [08] isotherm equation describes the behavior of many sorption systems on the heterogeneous surface and it is based on the following equation

qe = RT ln (ATCe)/bT

The linear form of Temkin isotherm can be expressed as

qe = (RT/ bT) ln(AT) + (RT/bT) ln(Ce)
where AT= exp [b(0) x b(1) / RT]

b(1) = RT/ bT is the slope
b(0) = (RT/ bT) ln (AT) is the intercept and
b= RT/b(1)

The present data are analysed according to the linear form of Temkin isotherm and the linear plot is shown in figure 10. The equation obtained for IC dye sorption is: qe = 21.27 lnCe–21.54 with a correlation coefficient 0.997.

The best fit model is determined based on the linear regression correlation coefficient (R) shown in table 1.

From the figures 8, 9 & 10, it is found that sorption data are well represented by Temkin isotherm with higher correlation coefficient of 0.997, followed by Langmuir and Freundlich isotherms with correlation coefficients of 0.986 and 0.983 respectively.

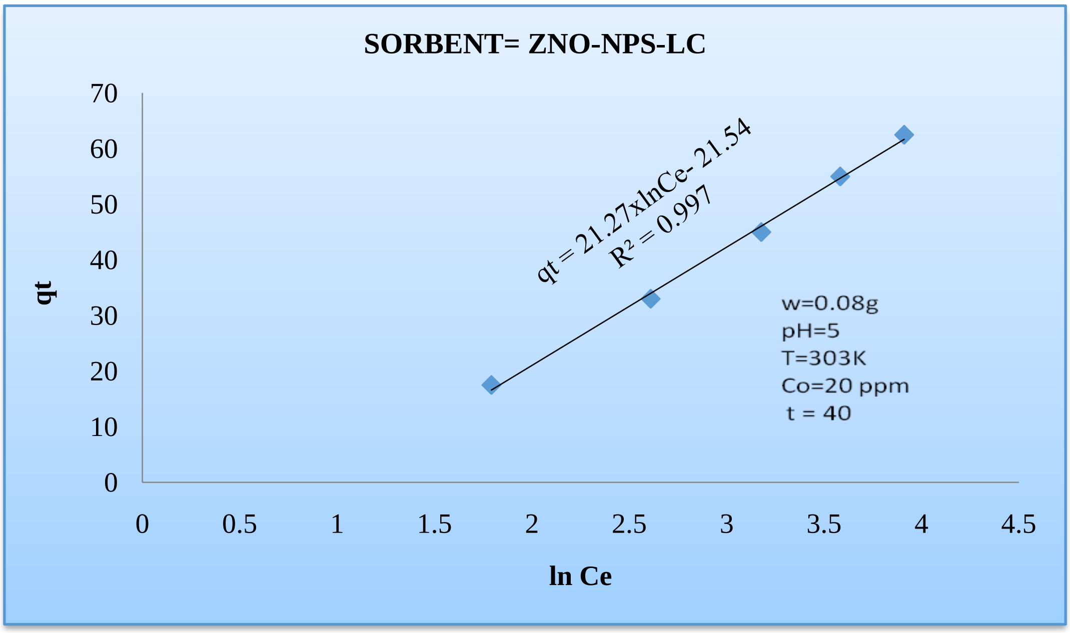

Figure 10.Temkin isotherm for % sorption of IC dye

Table 1. Isotherms constants

Langmuir	Freundlich	Temkin
q_m = 200	k_f = 6.366	A_T = 0.3632
b = 0.024876	n = 0.7496	b_T = 118.4364
R^2 =0.986	R^2 =0.983	R^2 =0.997

Kinetics of sorption

The order of biosorbate–sorbent interactions have been described using kinetic model. Traditionally, the first order model of Lagergren [09] finds wide application. In the case of sorption preceded by diffusion through a boundary, the kinetics in most cases follows the first order rate equation of Lagrangen:

$(dqt/dt) = Kad\ (qe–qt)$

whereqe and qt are the amounts adsorbed at t, min and equilibrium time and Kad is the rate constant of the pseudo first order sorption.

The above equation can be presented as

$\int (dqt / (qe – qt)) = \int Kaddt$

Applying the initial condition qt = 0 at t = 0, we get

log (qe – qt) = log qe – (Kad/2.303) t
log (qe – qt) = -0.027 t + 1.162

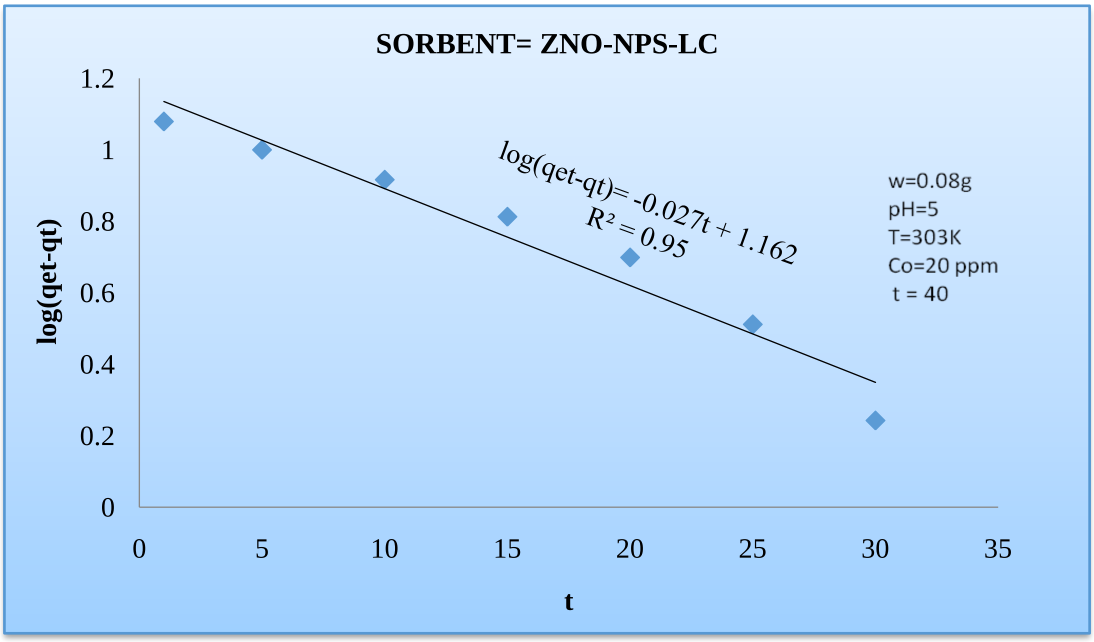

Figure 11. First order kinetics for % sorption of IC dye

Plot of log (qe–qt) versus‘t’ gives a straight line for first order kinetics, facilitating the computation of sorption rate constant (Kad). If the experimental results do not follow the above equation, they differ in two important aspects:

Kad (qe – qt) does not represent the number of available sorption sites and log qe is not equal to the intercept.

In such cases, pseudo second order kinetic equation: (dqt/dt) = K (qe – qt) 2 is applicable, where

‘K’ is the second order rate constant.

The other form of the above equation is: (dqt/(qe–qt)2) = Kdt
let qe – qt = x

dqt = dx

1/ x = K x + C
C = 1/ qe at t = 0 and x = qe

Substituting these values in above equation, we obtain:
1/(qe – qt) = Kt + (1/qe)

Rearranging the terms, we get the linear form as:

(t/qt) = (1/ Kqe2) + (1/qe) t.
(t/qt) = 0.062 t + 0.628.

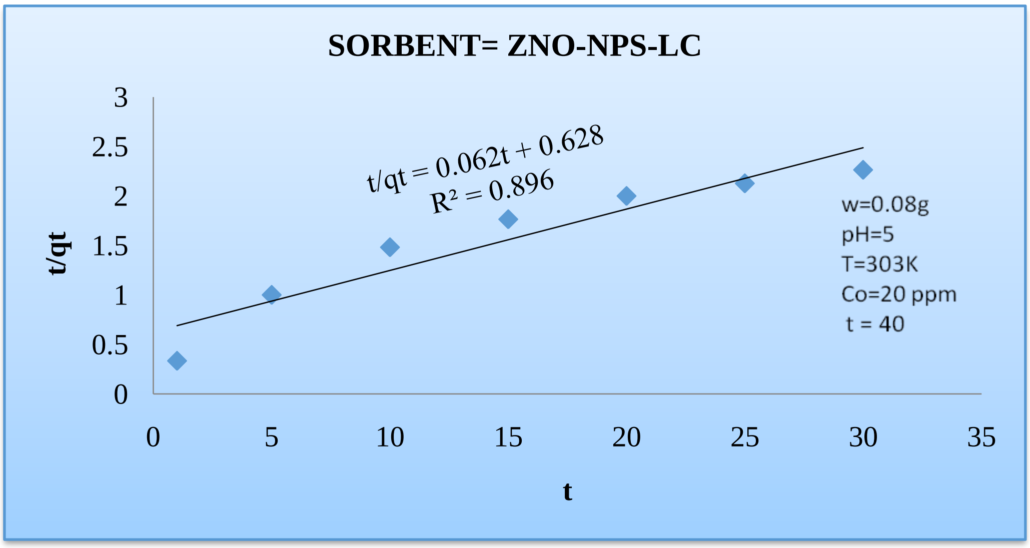

Figure 12. Second order kinetics for % sorption of IC dye

The pseudo second order model [10] based on above equation, considers the rate -limiting step as the formation of chemisorptive bond involving sharing or exchange of electrons between the biosorbate and sorbent. If the pseudo second order kinetics is applicable, the plot of (t/qt) versus ‘t’ gives a linear relationship that allows computation of qe and K.

In the present study, the kinetics are investigated with 50 mL of aqueous solution (C0= 20 mg/L) at 303 K with the interaction time intervals of 1 min to 180 min. Lagragen plots of log (qe-qt) versus agitation time (t) for sorption of IC dye the sorbent ZNO-NPS-LC powder in the interaction time intervals of 1 to 180 min are drawn in figures 11 & 12. The rate constants are presented in table 2.

Table 2. Equations and rate constants

Order	Equation	Rate constants	R^2
Lagergren first order	log (q_e-q_t)=–0.027 t + 1.162	0.062181 min^{-1}	0.95
Pseudo second order	t/q_t=0.062 t+0.628	0.006 g/(mg-min)	0.896

Thermodynamics of sorption

Sorption is temperature dependant. In general, the temperature dependence is associated with three thermodynamic parameters namely change in enthalpy of sorption (ΔH), change in entropy of sorption (ΔS) and change in Gibbs free energy (ΔG) [11].

Enthalpy is the most commonly used thermodynamic function due to its practical significance. The negative value of ΔH will indicate the exothermic/endothermic nature of sorption and the physical/chemical in nature of sorption. It can be easily reversed by supplying the heat equal to calculated ΔH.

The ΔH is related to ΔG and ΔS as

$\Delta G = \Delta H - T\,\Delta S$

ΔS < 1 indicates that sorption is impossible whereas ΔS > 1 indicates that the sorption is possible. ΔG < 1 indicates the feasibility of sorption.

The Vant Hoff’s equation is

$\log (qe / Ce) = \Delta H/(2.303\ RT) + (\Delta S/2.303\ R)$

log (qe /Ce) = – 0.736 (1 / T) + 2.771
Where (qe/Ce) is called the sorption affinity.

If the value of ΔS is less than zero, it indicates that the process is highly reversible. If ΔS is more than or equal to zero, it indicates the reversibility of process. The negative value for ΔG indicates the spontaneity of sorption. Whereas the positive value indicates is non-spontaneity of sorption.

Experiments are conducted to understand the sorption behavior varying the temperature from 283 to 323 K. the plots indicating the effect of temperature on sorption of IC dye for different initial dye concentrations are shown in figure 13. The Vant Hoff's plots for the sorption data obtained at various initial concentrations of the IC dye are shown in figure 13. The values are ΔG = -16062.1, ΔH = 14.0923 and ΔS = 53.05673.

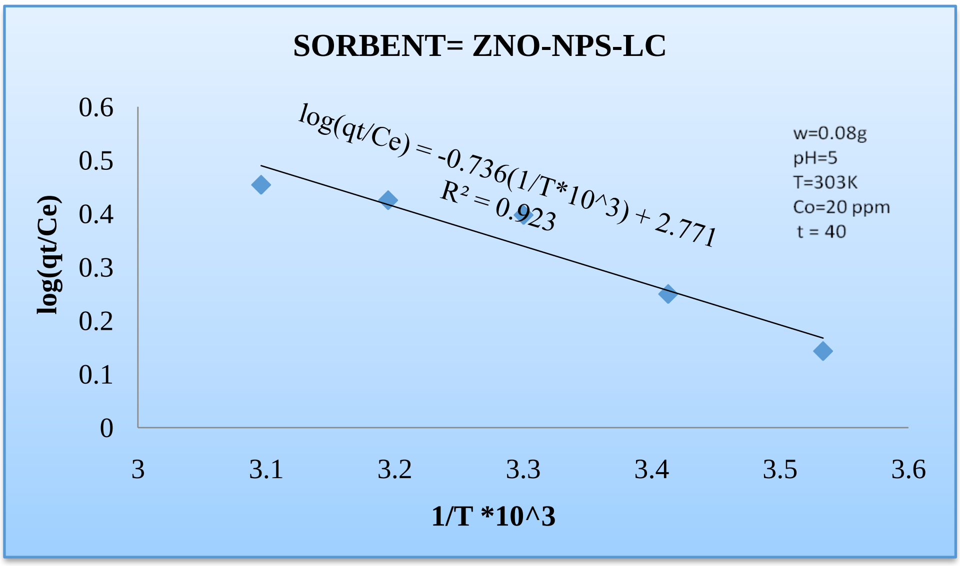

Figure 13.Vantoff's plot for % sorption of IC dye

Optimization using CCD

The parameters that have greater influence over the response are to be identified so as to find the optimum condition for the sorption of IC dyes. The quadratic model is used in the present study, to relate four independent variables and percentage sorption of IC dye. The regression equation for is % sorption of IC dye (Y) is function of pH (X1), Co (X2), w (X3) and T (X4) [12-15].

The variations in the corresponding coded values of four parameters and response are presented in table-3

Table 3. Levels of different process variables in coded and un-coded form for % sorption of IC dye using Zinc oxide nano particles powder

Variable	Name	Range and levels				
		-2	-1	0	1	2
X_1	pH of aqueous solution	3	4	5	6	7
X_2	Initial concentration, C_o, mg/L	10	15	20	25	30
X_3	Sorbent dosage, w, g	0.04	0.06	0.08	0.10	0.12
X_4	Temperature, T, K	283	293	303	313	323

The following equation represents multiple regression analysis of the experimental data for the sorption of IC dye:

$$Y = -1508.59 - 67.91\, X_1 + 4.50\, X_2 + 739.44\, X_3 + 8.81\, X_4 - 6.75\, X_1^2 - 0.11\, X_2^2 - 4377.08 X_3^2 - 0.01\, X_4^2 - 0.02\, X_1X_2 - 0.50\, X_1X_3 - 0.00\, X_1X_4 - 1.83\, X_2X_3 - 0.00\, X_2X_4 + 0.08\, X_3X_4$$

Table-4. Results from CCD for IC dye sorption by Zinc oxide nano particles *p*owder

Run no.	X1, pH	X2, Co	X3, W	X4, T	% sorption of IC dye	
					Experimental	Predicted
1	4	15	0.06	293	70.98	71.00
2	4	15	0.06	313	72.52	72.47
3	4	15	0.1	293	72.28	72.27
4	4	15	0.1	313	73.82	73.80
5	4	25	0.06	293	72.38	72.39
6	4	25	0.06	313	74.02	74.02
7	4	25	0.1	293	72.98	72.93
8	4	25	0.1	313	74.58	74.62
9	6	15	0.06	293	72.32	72.27
10	6	15	0.06	313	73.78	73.81
11	6	15	0.1	293	73.52	73.49
12	6	15	0.1	313	75.12	75.10
13	6	25	0.06	293	73.32	73.32
14	6	25	0.06	313	75.02	75.02
15	6	25	0.1	293	73.78	73.82
16	6	25	0.1	313	75.62	75.58
17	3	20	0.08	303	58.08	58.09
18	7	20	0.08	303	60.28	60.31
19	5	10	0.08	303	74.18	74.23
20	5	30	0.08	303	76.12	76.10
21	5	20	0.04	303	78.28	78.28
22	5	20	0.12	303	80.08	80.11
23	5	20	0.08	283	78.78	78.80
24	5	20	0.08	323	82.02	82.03
25	5	20	0.08	303	86.20	86.20
26	5	20	0.08	303	86.20	86.20
27	5	20	0.08	303	86.20	86.20
28	5	20	0.08	303	86.20	86.20
29	5	20	0.08	303	86.20	86.20
30	5	20	0.08	303	86.20	86.20

Table 4 represents the results obtained in CCD. Response obtained from regression in the form of ANOVA is presented in table 5. From the Fisher's F-test (Fmodel = 70621) and a very low probability value (Pmodel> F=0.000000), it is known from table–3 that the model is highly significant.

At 5% level, the computed F-value (F0.05 (14.15) = MSmodel/MSerror = 70621) is greater than that of the tabular F-value (F0.05 (14.15) tabulars = 2.42), indicating that the treatment differences are significant.

Table 5.ANOVA of IC dye sorption for entire quadratic model

Source of variation	SS	Df	Mean square (MS)	F-value	*P*> F
Model	1384.241	14	98.87	70621.4	0.00000
Error	0.021	15	0.0014		
Total	1384.262				

It is found that the X1, X2, X3, X4, X12, X22 , X32 , X42 X1X2, X1X3, X2X3 and X2X4 have high significance to explain the individual and interaction effect of independent variables on IC dye sorption. The other terms (X1X4 and X2X4) are insignificant and are not required to explain sorption. The model is reduced to the following form by removing insignificant terms.

$$\boldsymbol{Y = -1508.59 - 67.91\,X_1 + 4.50\,X_2 + 739.44\,X_3 + 8.81\,X_4 - 6.75\,X_1^2 - 0.11\,X_2^2 - 4377.08X_3^2 - 0.01\,X_4^2 - 0.02\,X_1X_2 - 0.50\,X_1X_3 - 1.83\,X_2X_3 + 0.08\,X_3X_4}$$

A synergistic effect is indicated by positive sign of the coefficient which means response increases with an increase in effect, while an antagonistic effect is indicated by a negative sign which means response decreases with an increase in effect. In the observed response values, a measure of the models variability is provided by the correlation coefficient (R^2).

In the present study, the value of the regression coefficient (R^2 = 0.9999) indicates that 0.001 % of the total variations are not satisfactorily explained by the model. It is proved from that table that Fstatistics value for entire model is higher. This large value means that % sorption can be adequately explained by the model equation.

Generally P values lower than 0.05 indicates that the model is considered to be statistically significant at 95% confidence level. The % sorption prediction from the model is shown in table-4. It is implied from table-4 that all the squared terms of all the variables and the linear terms are significant ($P < 0.05$). Among the interaction terms, all the terms ($P < 0.05$) are insignificant on the sorption capacity. Figure 14 and Figure 15 shows pareto chart and normal probability plot (NPP) of residual values. It could be seen that the experimental points are reasonably aligned suggesting normal distribution.

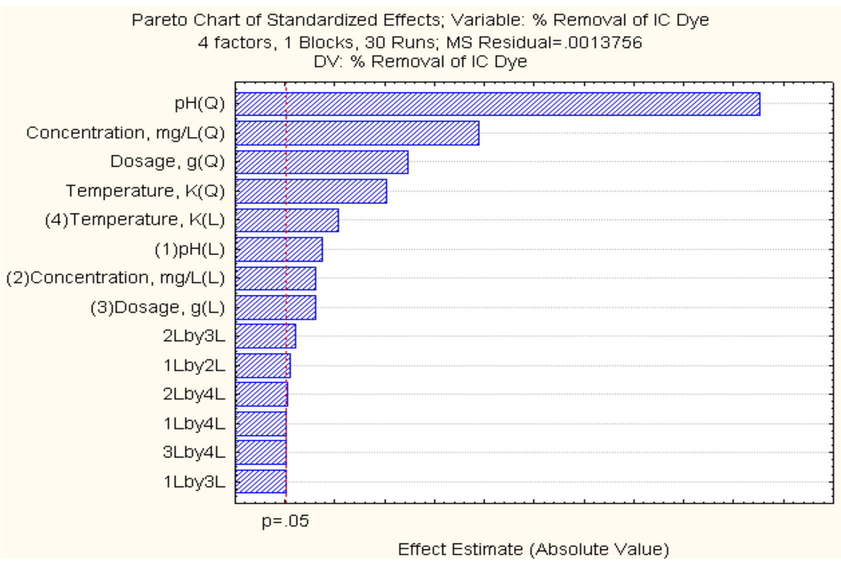

Figure 14. Pareto Chart

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Interaction effects of sorption variables

The three-dimensional view of response surface contour plots [Figures 16 (a) to (f)] show % sorption as a function of for various combinations of independent variables. The plots are represented as a function of two factors at a time keeping other factors fixed at zero level.

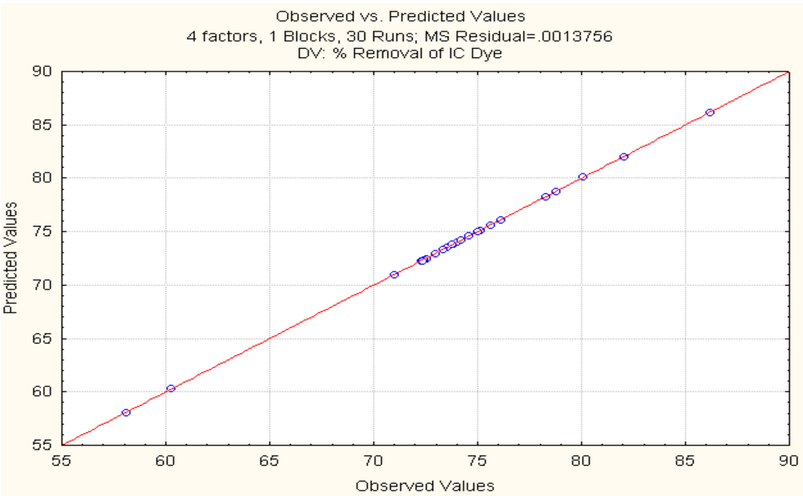

Figure 15. Normal probability plot for % sorption of IC dye

It is found from the response surface plots that the % sorption is maximal at low and high levels of the input variables. However, there exists a region where neither an increasing nor a decreasing trend in % sorption is observed. The sorption variables should be optimum to maximize the % sorption. The % sorption of IC dye is strongly influenced by the pH as evident from figures 13 (a) & (b).

The predicted optimal set of conditions for percentage sorption of IC dye is

pH of aqueous solution = 5.0409
Initial IC dye concentration = 20.4105 mg/L
Sorbent dosage = 0.0825 g
Temperature = 305.8158 K
% sorption of IC dye = 86.37342

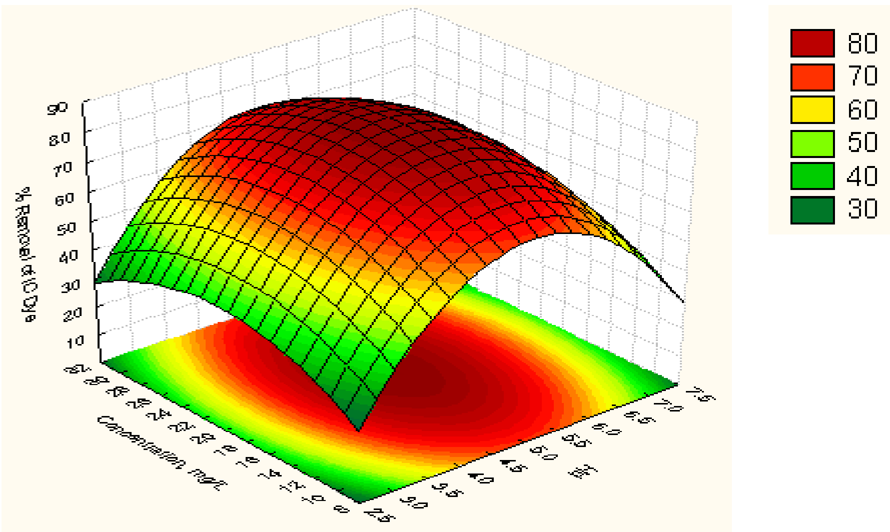

Figure 16 (a). Surface contour plot for the effects of pH and initial IC dye concentration on % sorption

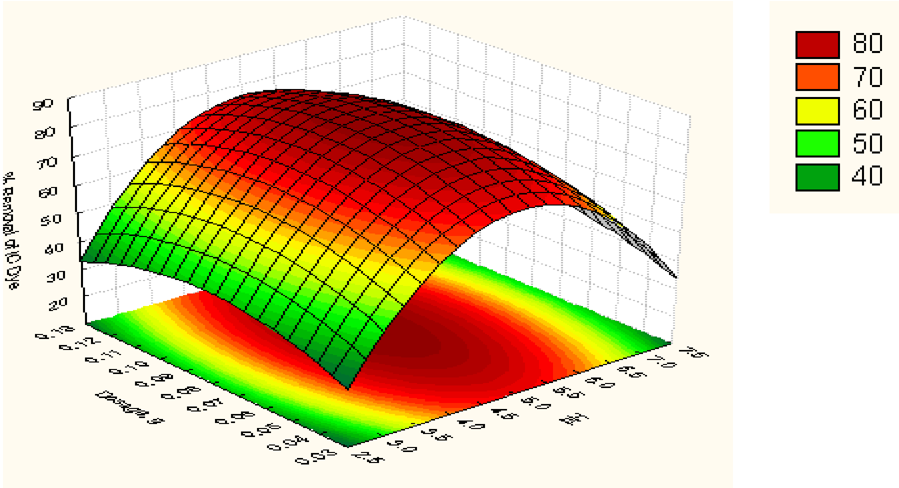

Figure 16 (b). Surface contour plot for the effects of pH and dosage on % sorption of IC dye

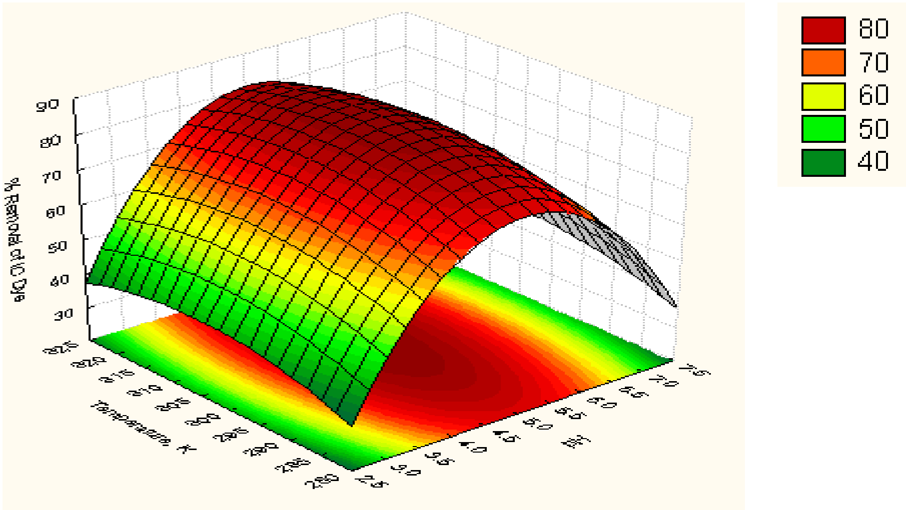

Figure 16 (c). Surface contour plot for the effects of pH and Temperature on % sorption of IC dye

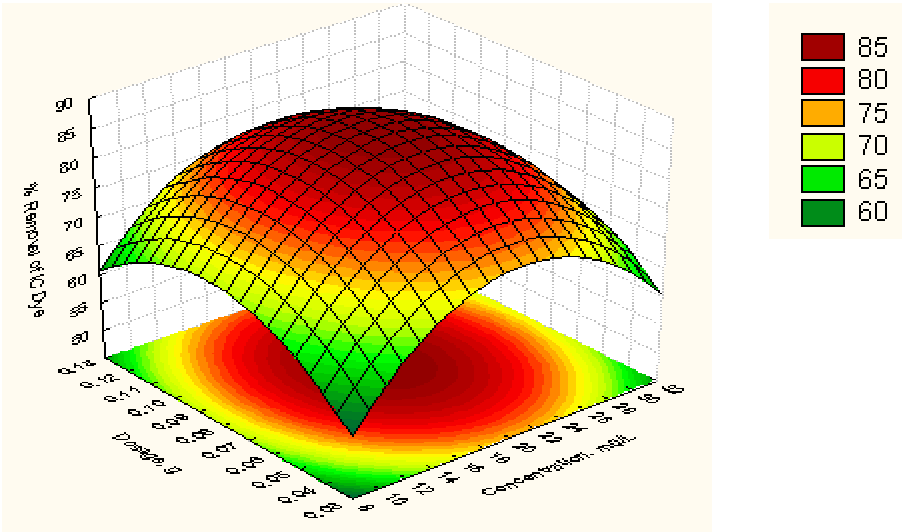

Figure 16 (d). Surface contour plot for the effects of initial concentration and dosage on % sorption of IC dye

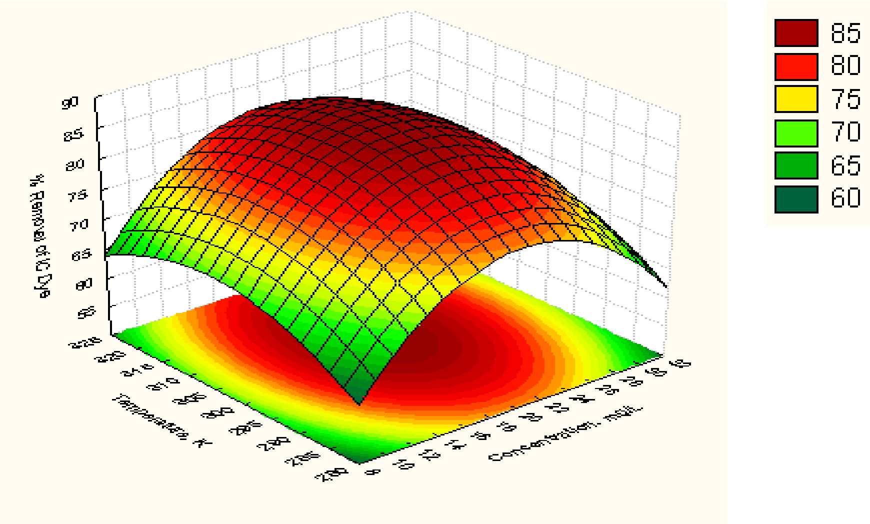

Figure 16 (e). Surface contour plot for the effects of initial concentration and Temperature on % sorption of IC dye

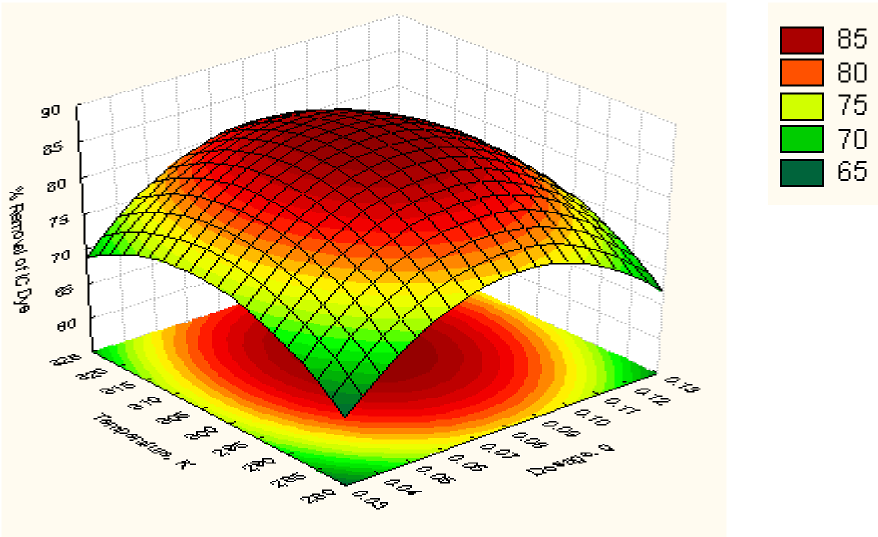

Figure 16 (f). Surface contour plot for the effects of Dosage and Temperature on % sorption of IC dye

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