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Adsorption and Statistical Analysis of Textile Effluent Contaminated with Congo red On Orange Peel

Shashikumar Prakash Mitadar, Veena-Kumara-Adi

Abstract: ***In the present study we made an effort to deploy orange peel as adsorbent to remove Congo red dye from a synthetic media. Adsorption of Congo red dye was carried out using Response Surface Methodology (RSM).The influence of various factors such as adsorbent dosage, initial concentration of adsorbate and agitation time on adsorption was analyzed. A total no of 13 runs of experiments were conducted by using the detailed conditions which is designed by RSM based Box-Behnken Design. Contour plots and surface plots were analyzed to know the effect of factors. Activated orange peel could adsorb a maximum of 96.776% of congo red dye. The values observed in the experiment well agreed with the RSM predicted values .Activated orange peel seems to be better adsorbent and RSM could be a great tool undertake adsorption.: Congo Red Dye, Adsorption, Response Surface Methodology, Orange Peel, Box-Behnken* **Design.**

Index Terms: Adsorption, Box-behnken Design ,Congo red dye, Orange peel ,Response Surface Methodology.

I.INTRODUCTION

Textile sector accounts for 10% of the manufacturing production in India, 5% of India's GDP and 13% of country's export earnings. Textile sector is the second largest source of employment in India which employs nearly 51 million people directly and 68 million people indirectly during 2015-16 (Sujit Gulhane and Ranjit Turukmane,2017).Textile products requires huge quantity of dyes. Fabrics from synthetic fibres require 52g of dye per kg of textile product and Printed fabrics from cellulose fibres require 88g per kg of textile product (Zaharia Carmen and Suteu Daniela,2012).Around 10,000 dyes are available currently in the market (I. Ameeth Basha and T. Shanthi,2018).Congo red in effluents(Fig 1) is a benzidine based anionic dye (Yusef Omidi Khaniabadi et.al,2017). Congo red is mainly released from textile, paper, printing and leather industries (Yaneva Z L et.al,2012).It is a known human carcinogen and exposure to this dye can result in the allergic problems(Sudipta Chatterjee et.al,2010). The physio-chemical properties of congo red are tabulated as Table 1**(**Nirav P. Raval et.al,2016**)**.And it is clear that it works as an acid-base indicator.

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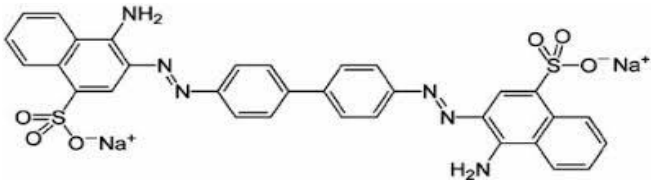

Fig 1: Molecular structure of Congo red.

Table 1: **Physio-Chemical Properties of Congo red dye**

Parameter	**Content**
Dye class	Azo dye
Molecular weight(g/mol)	696.68
Molecular formula	C32H22N6Na2O6S2
pH range	3.0-5.0

Various technologies (Vanitha Katheresan et.al, 2018) are available to abate dyes from wastewater such as Ion Exchange(Muhammad Imran Khan et.al 2015),Advanced Oxidation(PV Nidheesh et.al,2018), Electrochemical Degradation(Martin Valica and Stanislav Hostin 2016),Fenton Reaction Dye Removal (Nese Ertugay ,Filiz Nuran Acar, 2017), Membrane Filtration (Mohammad Fadhil Abid et.al 2012), Adsorption (Seema et.al 2017).Adsorption seems to be very effective and cost-effective method in removal of dyes.

Earlier adsorption of Congo Red dye using various adsorbents such as saw dust(M.Srinivas Kini et.al,2017),apple leaf powder(I. Ameeth bashaa et.al,2016),Sugarcane Bagasse(Zhanying Zhang et.al,2011),Shrimp shell powder(Youzhou Zhou et.al,2018) , Aloe vera leaves shell (Yusef Omidi Khaniabadi et.al,2017), Casurina leaves(E. Kowsalya et.al,2015), Goose Grass (Adeboye Omolara Olubunmi,2015) etc have been conducted. In present study we are exploring the possibility of adsorbing Congo Red dye from synthetic effluent using orange peel. Effectiveness of Response surface methodology in optimizing the adsorption process was studied. An effort was done to abate Congo red dye using cost effective orange peel.

II. MATERIALS AND METHODOLOGY

A. Preparation of Adsorbent:

Orange peels was used as adsorbent material. Orange peel was collected from the local juice stalls. Peels were washed with distilled water thoroughly to remove all the dirt and other impurities

(Fahim Bin Abdur Rahman et.al,2013). Later it was kept for sun drying and then it was powdered. Further it was treated with concentrated H2SO4 for 24 hrs. And it was washed several times to remove residual acid. Then it was kept for activation muffle furnace for 10mins at 350˚C.Later it was sieved and uniform size of 200µm powder material was taken and used throughout the experiment (Seema et. al , 2017)..

B. Preparation of adsorbate:

A stock solution was prepared by dissolving 1g of Congo red dye in 1000ml of distilled water(Y.C. Wong et.al,2014). And Subsequent required working concentrations (100ppm,550ppm,1000ppm) were prepared from the stock solution.

C. Estimation and spectrum analysis of Congo red:

Spectrum analysis:

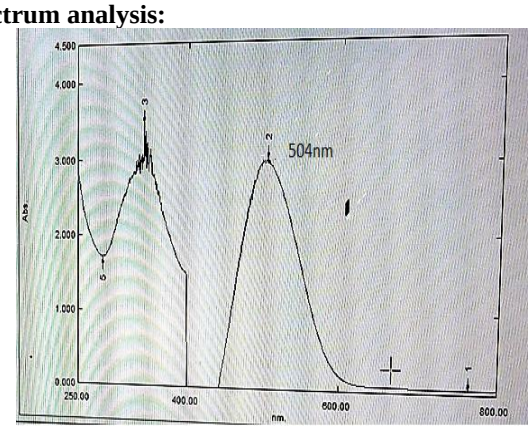

Fig 2:Spectrum Of Congo Red In UV Spectrophotometer

Spectrum analysis was carried out in UV-Spectrophotometer to know its maximum absorption wavelength. Couple of earlier studies reported that the maximum wavelength of Congo Red was at 520nm (Sayğılı G.A et.al,2015).For measuring Congo Red, spectrum analysis(Fig 2) showed that there was maximum absorption at 504nm.So 504nm was considered throughout the experiment.

Standard Calibration of Congo Red dye:

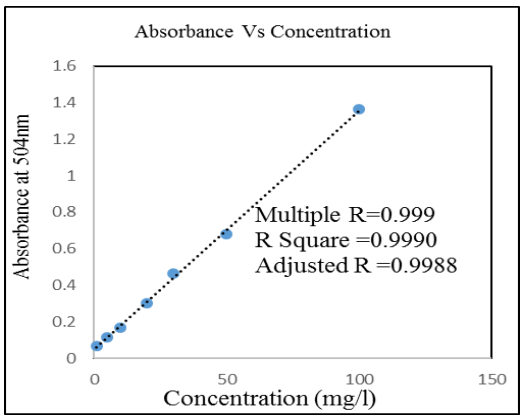

Fig 3:Standard Curve For Congo Red At 504nm

Standard curve of adsorption versus concentration was plotted(Fig 3).A Regression coefficent of 0.999 was obtained.

D. Adsorption studies:

The adsorption of Congo red dye using activated orange peel was examined in a batch operation mode. Adsorption studies were conducted on 3 parameters such as adsorbent dosage ,contact time, initial concentration of Congo red, varying one parameter at a time. This was done to fix an effective range in Box-Behnken design. The effective range (Table 2) was later used in the statistical analysis on RSM using Box-Behnken design. A total of 13 runs(Table 3) were carried out which was given by the Minitab 16 software. All the parameters were maintained in solutions and kept for agitation at a constant speed of 150rpm. Finally clear supernatant was separated and analyzed in UV-Spectrophotometer at 504nm.

Calculation:

The % Congo red removal ,R(%),was calculated after each run as follows:

Dye removal (%) at any instant of time was determined by the following equation:

% of Removal Efficiency=((Ci - Cf)/ Ci)*100 ... Equation (1)

Where, Ci and Cf represent initial and final concentration (mg/L) of Congo red dye at any instant of time, respectively.

E. Box-Behnken Design:

Box-Behnken design is a three level design in which all the design points are either:

1) At the center of the design.
2) Centered on the edges of the cube, equidistant from the center.

The factors will follow a trend of -1, 0, +1 as a result of response surface design.

Table2: Experimental Range Of Factors In RSM

Parameter	Units	Low Value	High Value
Adsorbent dosage	Grams	0.5	2.5
Concentration	PPM	100	1000
Contact time	Minutes	60	180

Table 3:Optimal Conditions for experimental plan of Box-Behnken Design

Factors: 3 Replicates: 1
Base runs: 13 Total runs: 13
Base blocks: 1 Total blocks: 1
Center points: 1

Run Order	Adsorbent dosage(g)	Initial Concentration of Congo Red in synthetic media (PPM)	Contact time(Min)
1	2.5	550	180
2	0.5	550	60
3	0.5	100	120
4	0.5	1000	120
5	1.5	100	180
6	2.5	550	60
7	1.5	1000	180
8	1.5	550	120
9	2.5	1000	120
10	1.5	1000	60
11	2.5	100	120
12	1.5	100	60

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13	0.5	550	180

III. RESULTS AND DISCUSSION

A.Effect of adsorbent dosage:

Batch adsorption study was conducted to know the effect of adsorbent dosage on removal efficiency.Adsorbent dosage was varied from 0.5g to 3.5g.Initial concentration and contact time were maintained at 100ppm and 60mins respectively for all the samples.

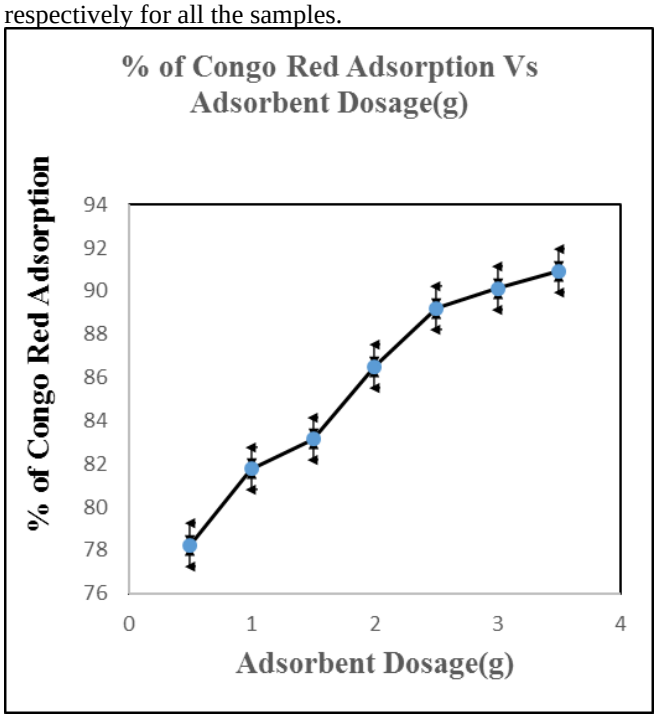

Fig 4:Effect Of Adsorbent Dosage

The removal efficiency constantly increased with increase in the adsorebnt dosage.The removal efficiency was 78.363% for 0.5g for adsorbent dosage),and it went up to 89.543% for 2.5g(Fig 4).Later the adsorption rate attained constant phase.

B.Effect of Contact Time:

Batch adsorption study was conducted to know the effect of Contact time on removal efficiency.Contact time was varied from 30mins to 300mins. Adsorbent dosage of 1g and concentration of 100ppm were maintained for all the samples.

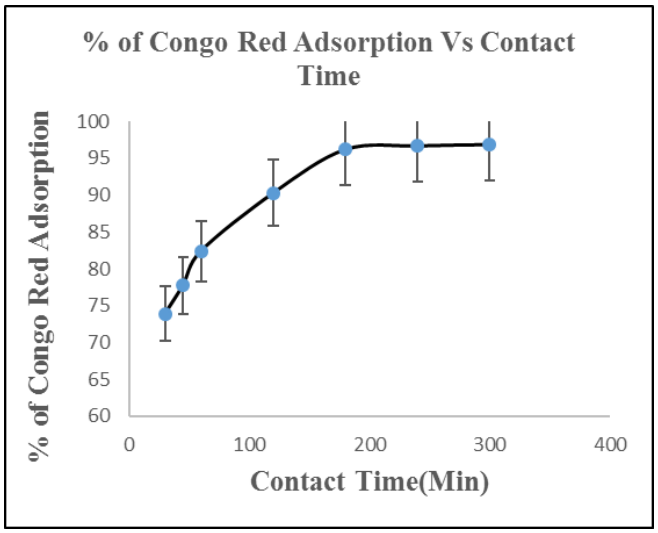

Fig 5:Effect of Contact Time

The removal efficiency was increased with increase in contact time.Removal efficiency was 73.876% for 30mins and later it went up to 96.904% for 300mins(Fig 5).

C.Effect of Concentration of Adsorbate :

Batch adsorption study was coducted to know the effect initial concentration of congo red dye on adsorption efficiency.Initial concentrations were varied from 5ppm to 5000ppm. Adsorbent dosage of 1g and contact time of 60mins were maintained for all the samples.

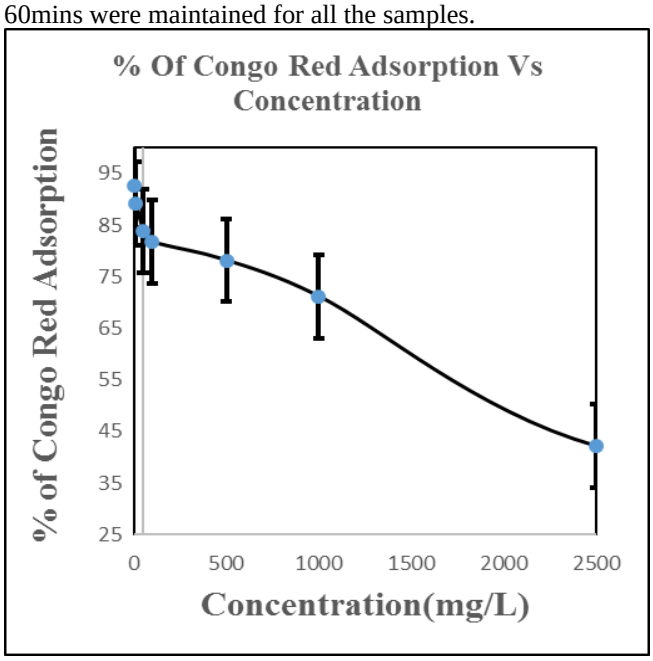

Fig 6: Effect of initial Concentration of the solution

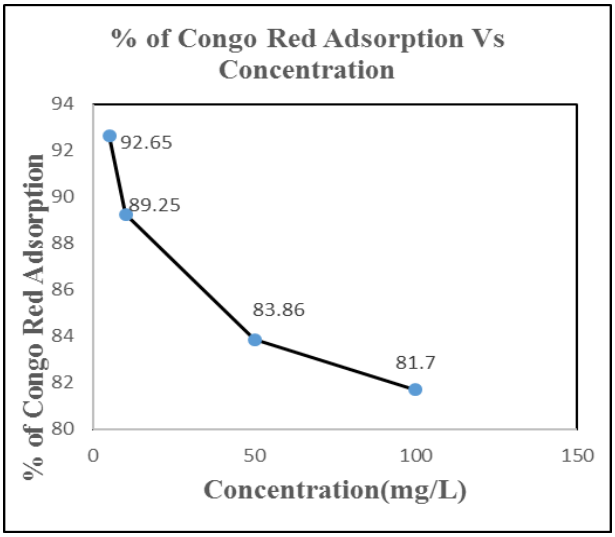

Fig 7: Effect of initial Concentration of the solution

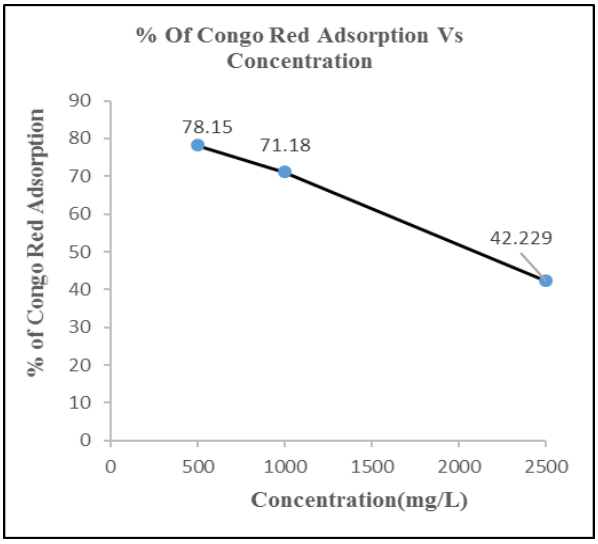

Fig 8: Effect of initial Concentration of the solution

The removal efficiency was decreased as there was increase in the initial concentration of the solution.Removal efficiency was 92.65% for 5ppm and it reduced to 29.85ppm for 5000ppm(Fig 6).

D.Isothermal Studies:

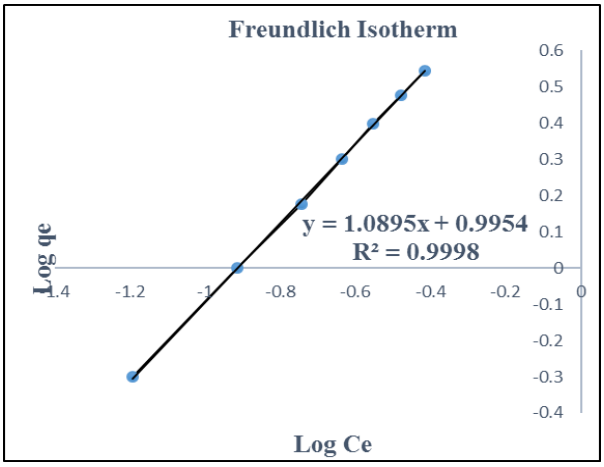

Fig 9:Freundlich Isotherm.

Adsorption Isotherms clearly relate the interaction between the adsorbate and the adsorbent which are used in the process.It shows the amount of adsorbate on the adsorbent as a function of pressure or initial concentration of the adsorbate at a constant temperature. Isothermal studies indicated that the adsorption process fits well for freundlich isotherm compared to Langmuir isotherm. The values of R-Square and n, 0.9998 and 0.9178 respectively shows that there is a good regression and slope obtained for Freundlich isotherm.

E. Box-Behnken design results

The percentage efficiency was calculated using equation (1) and the predicted values were given by the software. Predicted values and observed values are tabulated (Table 4).

Table 4: Analysis Of Predicted And Observed Values For Removal Efficiency

Run Order	% Adsorption Predicted	% Adsorption Observed
1	86.66	88.808
2	77.11	67.60
3	77.11	86.493
4	77.11	70.865
5	81.88	94.223
6	86.66	82.11
7	81.88	76.623
8	81.88	84.530
9	86.66	77.62
10	81.88	72.56
11	86.66	96.776
12	81.88	84.183
13	77.11	82.120

Table 4 displays predicted and observed percentage of Adsorption of Congo red dye.The adsorption efficiency increased from 67.60% to 96.776% when the parameters are varied.Highest percentage efficiency was obtained for an adsorbent dosage of 2.5g,contact time of 120mins and for an initial concentration of 100ppm.Analysis of variance were obtianed and it is summarised in table 5 .P value is the probability of getting a result at least as extreme as the one which was observed .F value is the residual variances in a model. P and F value will check the overall significance of the regression. Lower the value of P and Higher the value of F, more the model is significant. After the analysis if the obtained value of P is less than 0.05, then it indicates that the model is significant.

Table 5:Analysis of Variance

Source	DF	Seq SS	Adj SS	Ad MS	F	P	R
Regression	9	900.9	900.9	100.	4.34	0.01	S
Linear	3	850.3	850.3	283.	8.91	0.00	S
A	1	182.5	182.5	182.	44.38	0.00	S
B	1	511.8	511.8	511.	124.4	0.00	S

C	1	155.9	155.9	155.	37.90	0.00	S
Square	3	23.32	23.32	7.77	1.89	0.30	-
A*A	1	2.368	6.353	6.35	1.54	0.30	
B*B	1	4.241	0.012	0.01	0.00	0.96	-
C*C	1	16.71	16.71	16.7	4.06	0.13	-
Interaction	3	27.32	27.32	9.10	2.21	0.26	-
A*B	1	3.084	3.084	3.08	0.75	0.45	-
A*C	1	15.31	15.31	15.3	3.72	0.14	-
B*C	1	8.931	8.931	8.93	2.17	0.23	-
Residual error	3	12.34	12.34	4.11	-		
Total	12	913.3					

Where,
A:Adsorbent Dosage
B:Initial Concentration
C:Contact Time
R:Remark
S:Significant

Table 5: Summary Of The Model

S	R-Sq (Predicted)	R-Sq	R-Square(adj)
2.02812	84.53%	98.65%	94.60%

R-Sq of the fit was obtained as 84.53% and the R-sq after analysis was obtained as 98.65% and 94.60% of adjusted R-sq can be seen which shows the variation.(Table 5).P value indicates the statistical significance of difference. T value is an intermediate step and it is typically decided by the value of standard error. Coefficient value helps to find the T value when it is divided by the standard error. If the value of P is a smaller than the chosen level of significance i.e. 0.05, then the connection between the predictor and the response obtained after the experiment is statistically vital.

Table 6: Multiple Linear Regression Coefficients

Term	**Coefficient**	**SE Coefficient**	**T**	**P**
Constant	84.5300	2.0281	41.679	0.000
A	4.7770	0.7171	6.662	0.007
B	-7.9989	0.7171	-11.155	0.002
C	4.4146	0.7171	6.157	0.009
A*A	-1.6671	1.3415	-1.243	0.302
B*B	0.0716	1.3415	0.053	0.961
C*C	-2.7044	1.3415	-2.016	0.137
A*B	-0.8780	1.0141	-0.866	0.450
A*C	-1.9565	1.0141	-1.929	0.149
B*C	-1.4942	1.0141	-1.474	0.237

F.Surface Plots

Surface plot gives 3-dimensional view which provides a clear idea of the responses based on the factors.Here we are taking 2 factors at a time and checking the effect on percentage removal.A series of surface plots are shown below(Fig 10(a-f)).

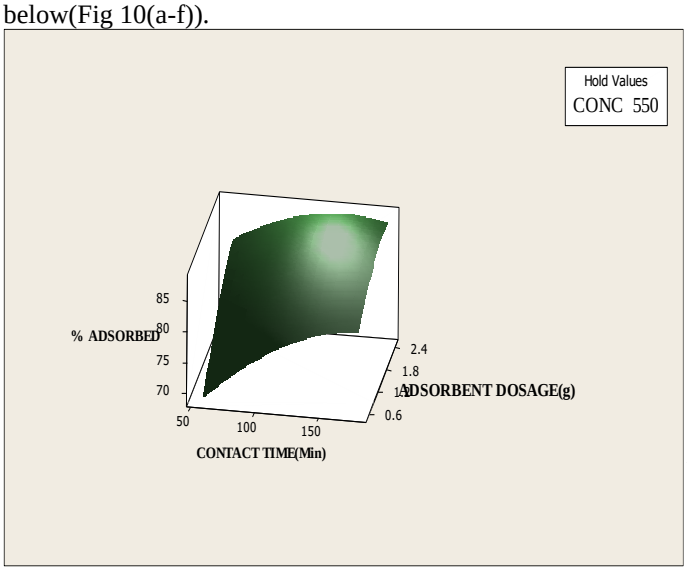

Fig 10(a): **Surface plot of % adsorbed Vs. Adsorbent Dosage, Contact time**

The surface plot indicates that the higher percentage of adsorption occurs at the higher dosages. As adsorbent dosage increased, the removal efficiency was also increased. Contact time also have its role. It is proved that as contact time increases, the removal efficiency also increases. Combined effect of higher adsorbent dosage and longer contact time will fetch higher percentage removal of Congo red dye (Fig 7(a)).

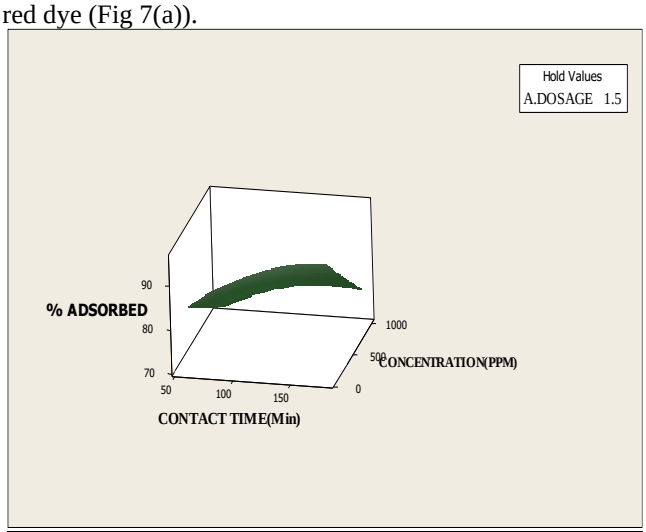

Fig 10(b): **Surface plot of % adsorbed Vs. Concentration, Contact time**

Surface plot which is plotted above indicate that there is higher adsorption efficiency at lower concentration of Congo red dye. As concentration increases the removal efficiency constantly decreased. With increase in contact time removal. Efficiency increases but it decreases with increase in the initial concentration of the solution

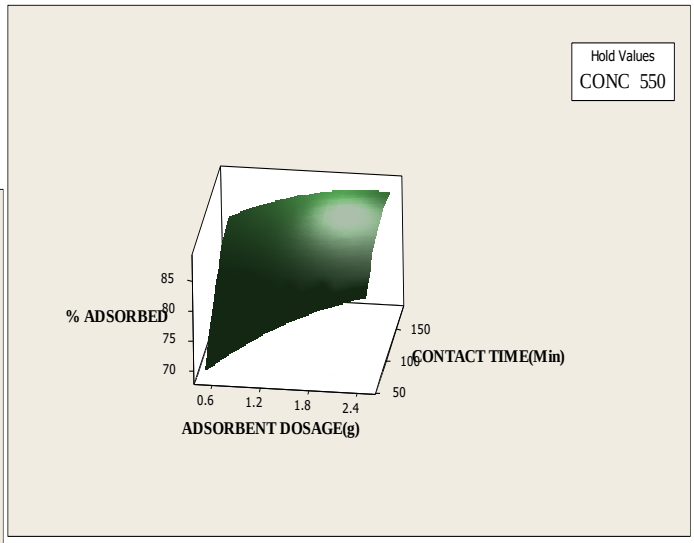

Fig 10(c): **Surface plot of % adsorbed Vs. Contact time, Adsorbent Dosage**

As the surface plot is indicating that the combined effect of dosage and contact time increases the removal efficiency, lower removal efficiency was found at lower dosages and at less contact time(Fig 7(c))

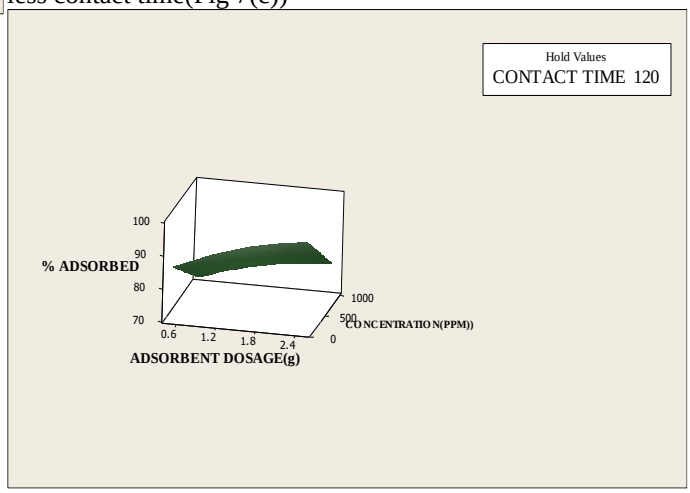

Fig 10(d):**Surface Plot Of % Adsorbed Vs. Concentration, Adsorbent Dosage**

The surface plot indicates that maximum removal of dyes can be obtained at the higher dosage of adsorbent and at the lower concentration of the solution(Fig 7(d)).

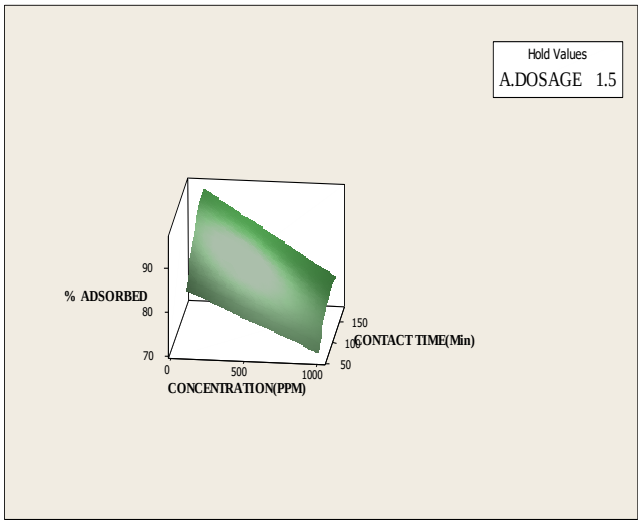

Fig 10(e): Surface Plot Of % Adsorbed Vs. Contact Time, Concentration

The above surface plot determines that there is a potential to get the higher adsorption efficiency of Congo red dye at the lower concentration and at the higher contact time. the removal efficiency was good at 180mins compared to the 60mins(Fig 7(e)). The surface plot indicates that the maximum efficiency of removal can be obtained with lower concentration dye and higher dosage of adsorbent. Removal efficiency is good at 100PPM compared to 1000ppm(Fig 7(f).

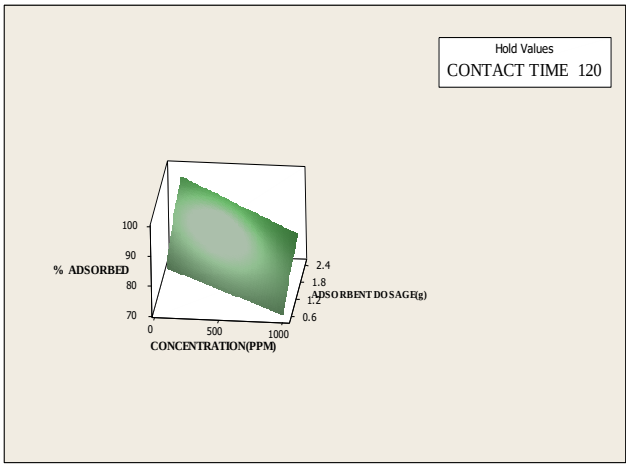

Fig 10(f):Surface Plot Of % Adsorbed Vs. Adsorbent Dosage, Concentration

G. Contour Plots:

Contour plots are the 2-dimensional view representations where there will be clear indication of the effect of parameters on the response. A series of contour plots are shown below(Fig 11(a-f)).

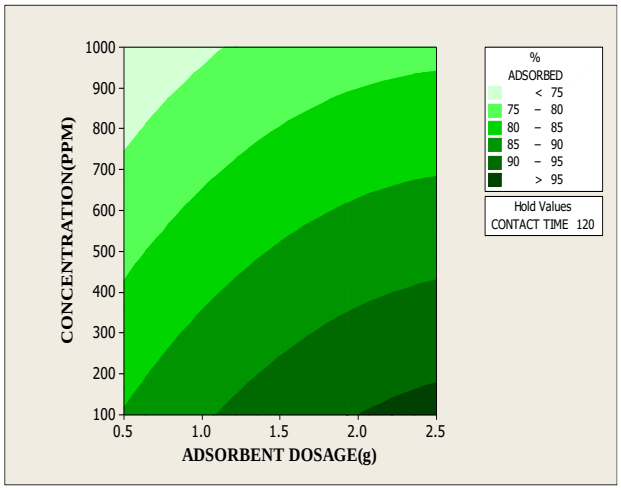

Fig 11(a): Contour Plot of % Adsorbed Vs. Concentration, Adsorbent Dosage.

Dark shaded portion at the bottom right region of Contour plot indicates that the higher removal efficiency is located at the higher adsorbent dosage i.e. at 2.5g compared to 0.5g.And there will be good adsorption of dye molecules at lower concentrations (Fig 8(a)). In the shaded portion at top right region of the contour plot indicates that the maximum removal efficiency can be obtained at higher contact time compared to lower contact time. And higher dosage will also contribute to the higher adsorption (Fig 8(b)).

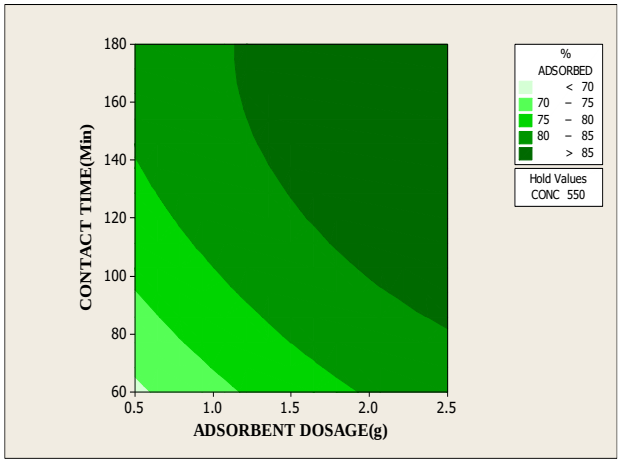

Fig 11(b): Contour Plot of % Adsorbed Vs. Contact time, Adsorbent dosage

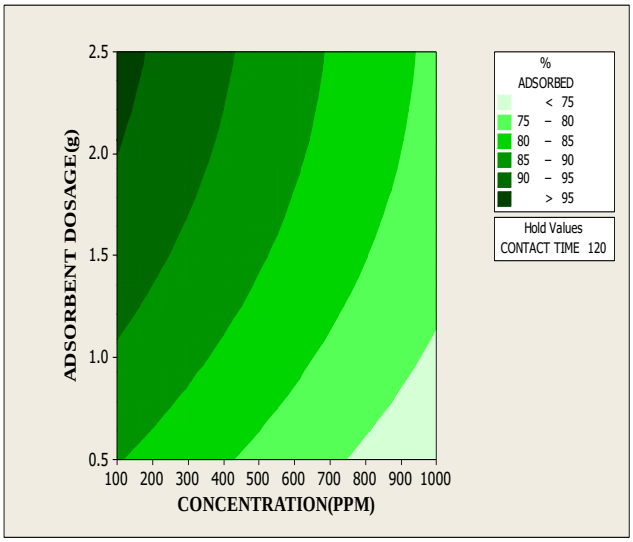

Fig 11(c):Contour Plot of % Adsorbed Vs. Adsorbent dosage, Concentration.

In Contour plot, darker shaded portion at top left region indicates that there will be higher adsorption at lower concentrations. And lighter shaded portion indicates that the removal efficiency decreased at higher concentration (Fig 8(c)).

In contour plot, darker shaded portion at the top left region indicates that the removal is at its high when the contact time is increased. But when the concentration increased the removal rate decreased (Fig 8(d)).

The dark shaded portion which is located at the bottom right portion indicate that there is a potential for the adsorbent to adsorb the dye at higher contact time and at the lower concentration(Fig 8(f)).

The results show that the maximum percentage removal of Congo red dye using orange peel is upto 96.776% at 100ppm of initial concentration, 120mins of contact time and at a dosage of 2.5g.So it can be said that orange peel is efficient as it is having more surface area to adsorb Congo red dye

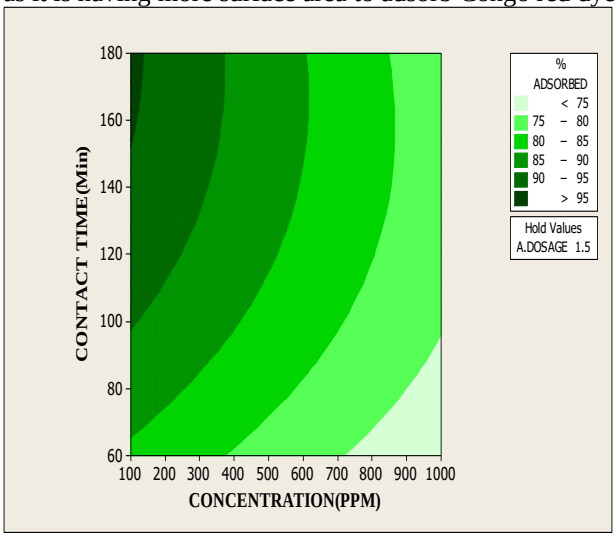

Fig 11(d):Contour Plot of % Adsorbed Vs. Contact time, Concentration

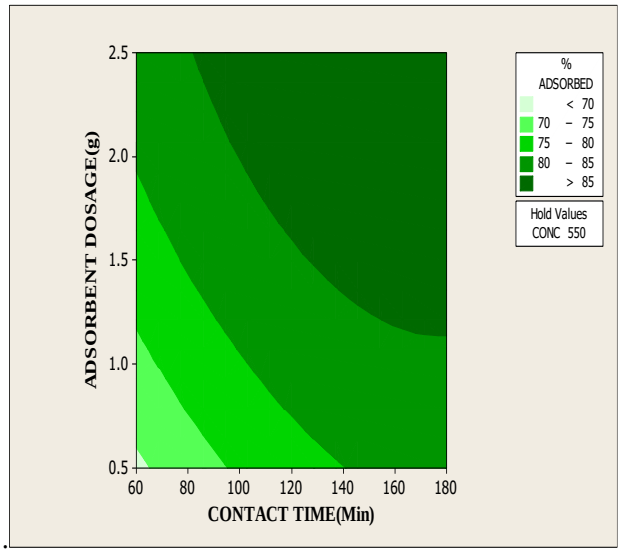

Fig 11(e): Contour Plot Of % Adsorbed Vs. Adsorbent Dosage, Contact Time

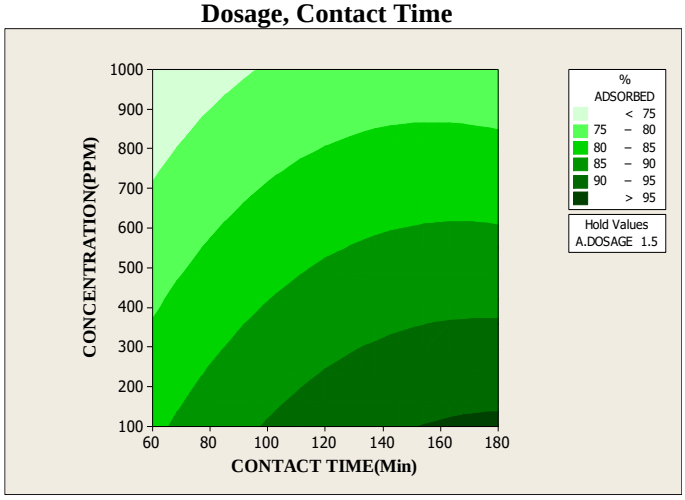

Fig 11(f): Contour Plot of % Adsorbed Vs. Concentration, Contact Time

Dark shaded portion at top right portion indicates that there will be good adsorption at the higher contact time and adsorbent dosage. Combined effect will increase the adsorption rate (Fig 8(e)).

IV.CONCLUSION

Orange peel can be a used as an adsorbent of Congo red dye.Behnken design is a good tool as it optimizes the design.Experimental results showed that 96.776% of Congo red dye was adsorbed using Orange peels.Box-Behnken design can be adopted as there was proper coordination between predicted and observed values.P and F values indicates that the effect of 3 parameters is significant on adsorption is significant.Surface plots and contour plots clearly shows the effect of all the parameters on adsorption of Congo red dye.So It can be said that Orange peel is a good adsorbent of Congo red dye.

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