Full Factorial Experimental Design Analysis of Acid Blue 147 Dye Removal from Aqueous Solution by Carbon Adsorption.

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ABSTRACT

A full factorial experimental design technique at two levels and four factors (2^4) was used to optimize the adsorption at 615nm of Acid Blue 147 in aqueous solutions onto activated carbon. The effect of pH (3 and 5), initial dye concentration (20 and 60mg/l), adsorbent dosage (0.01and 0.05g), and contact time (30 and 60min) on removal efficiency of the adsorbent for the dye were investigated at 298K. From the analysis of variance and cube plot, adsorbent dosage was observed to be the most significant factor affecting the adsorption process. However, at 95% confidence level, removal efficiency of 96.80% was achieved when conditions of adsorbent dosage, contact time, pH and initial dye concentration were set at 0.05g, 45 minutes, 3 and 60mg/l respectively.

(Keywords: factorial experimental design, adsorption, activated carbon, optimization, Acid Blu3 147)

INTRODUCTION

Rapidly changing technologies, industrial products, and applications are causing worldwide waste problems and contaminating the environment. If waste products are not properly managed, public health and the environment could be threatened.

Dyes are widely used in industries such as textile, rubber, paper, plastic, cosmetic, leather dyeing, and color photography, and as additives in petroleum products (Demirbas and Nas, 2009: Dawood, 2010). Among all industrial sectors, textile industries are rated as high polluters, taking into consideration the volume of discharge and effluent composition (Ozbay *et al.*, 2013).

Globally, thousands of the dye stuffs are being synthesized daily and also being released in the environment in the form of effluents during synthesis and dyeing processes (Jadhay *et al.*, 2011). The discharge of colored wastes into the receiving water bodies not only affects their aesthetic nature but also interferes with the transmission of sunlight and therefore reduces the photosynthetic activity (Femandes *et al.,* 2007: Lin *et al***.**, 2008: Rastogi *et al.*, 2008). Some of the dyes are known carcinogenic and mutagenic substances with serious health implications (Coruh and Elevli, 2015).

Acid Blue 147 (AB147) is an anionic dye used in the textile industry for dyeing of all natural fibers, for example, wool, cotton, silk and synthetics; polyesters, acrylic and rayon. The dye is used in other application fields such as in paints, inks, plastics, and leather. It is also used as a tracking dye to monitor the progress of electrophoretic separation. This tracking dye usually migrates with DNA molecules. Therefore, in the monitoring of DNA involving longer experimental runs, AB147 is the tracking dye of choice. The dye is irritating to eyes and skin on contact. Inhalation causes irritation of the lungs and respiratory system (TCI America, 2015). The very wide application of this dye therefore, means that the removal of the dye from solution is an important investigation.

Several physicochemical processes are generally adopted to treat dye wastewater. This includes adsorption (Al-Qodah, 2000), coagulation and flocculation (Halliday and Beszedits, 1986), membrane filtration (Gupta *et al.,* 1990), electrochemical process (Faouzi *et al.,* 2007), irradiation, and ozonation (Daneshvar *et al.,* 2007). However, all these processes except adsorption are not economically viable because of high cost and the formation of other toxic substances during chemical treatment. Being a conventional process adsorption is practiced by most wastewater treatment plants due to its easy handling, low cost, and better efficiency (Sudamalla *et al.,* 2012).

The classical approach of studying adsorption of dye in solutions, in which one factor is varied at a time have several limitations. The major disadvantage of this strategy is that it fails to consider possible interactions between the factors (Niehues *et al.*, 2010). This limitation of the classical method can be addressed by optimizing all the influencing parameters through statistical experimental design. This is a collection of mathematical and statistical techniques which are useful for developing, improving and optimizing the process and can be used to evaluate the relative significance of several influencing factors, even in the presence of complex interactions.

Factorial design (Niehues *et al.*, 2010) is an experimental strategy that allows the simultaneous manipulation of many factors and possible synergistic and antagonistic interactions between them can be determined. In addition, the development of mathematical models permits an assessment of both the relevance and statistical significance of the factor effects. System optimization can be attained performing a smaller number of experiments than that needed for univariate techniques resulting in lower reagent consumption and considerably less laboratory work.

In this study, the carbon adsorption at 615nm of AB147 dye ($C_{25}H_{27}N_2NaO_6S_2$) (Figure 1) was investigated by statistical experimental design. A full factorial experimental design was applied using 2^4 (two levels and four factors) approach in order to examine the main factors affecting the carbon adsorption and their interactions. Factors such as initial AB147 concentration, contact time, pH and carbon dosage were investigated.

METHODOLOGY

Preparation of Activated Carbon

The activated carbon used in this work was prepared from *Brachystegia eurycoma* seed hulls by chemical activation with H_2PO_4. Detail procedure for obtaining highly porous carbon from the precursor was earlier reported (Okibe *et al.,* 2013).

Preparation of Acid Blue 147 Solution

Stock solution of the dye was prepared in a 1000ml capacity volumetric flask by dissolving 1.0g AB147 in small amount of de-ionized water in the flask and made up to the mark. The concentration of the solution was 1000mg/l.

Full Factorial Design of Experiment for the Adsorption of AB147

The variables investigated were coded as; pH (A), Initial AB147 concentration (B) mg/l, Carbon dosage (C) g, and Contact time (D) min (Table 1). The adsorption process optimization was studied by varying these factors at two levels (2^4), high (+1) and low (-1) with two central points (0) to give a total of 18 experimental runs (Table 2). Statistical analyses were performed using Design-Expert 6.0.6 software.

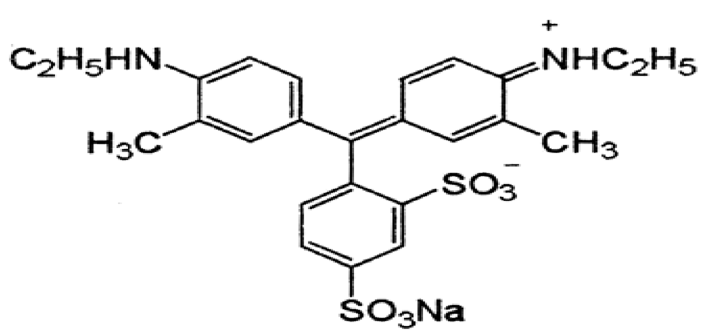

Figure 1: Molecular Structure of AB147 Dye.

Table 1: Factors and Levels used in the Factorial Design of Experiment for Adsorption of AB147.

Factors	Symbol	Low(-)	Center(0)	High(+)
pH	A	3	4	5
Initial AB147 concentration (mg/l)	B	20	40	60
Carbon dosage (g)	C	0.01	0.03	0.05
Contact time (mm)	D	30	45	60

Table 2: Design Matrix for the Adsorption of AB147 using 2^4 Full Factorial and Two Central Points.

Run	A:pH	B: AB147 Concentration (mg/l)	C:Carbon dosage (g)	D. Contact time (min)
1	4.00	40.00	0.03	45.00
2	5.00	20.00	0.01	60.00
3	3.00	20.00	0.01	60.00
4	3.00	60.00	0.01	60.00
5	4.00	40.00	0.03	45.00
6	3.00	20.00	0.05	30.00
7	3.00	60.00	0.05	30.00
8	5.00	60.00	0.05	30.00
9	5.00	60.00	0.01	60.00
10	5.00	20.00	0.05	60.00
11	3.00	20.00	0.05	60.00
12	5.00	60.00	0.01	30.00
13	5.00	60.00	0.05	60.00
14	5.00	20.00	0.01	30.00
15	3.00	60.00	0.01	30.00
16	3.00	60.00	0.05	60.00
17	3.00	20.00	0.01	30.00
18	5.00	20.00	0.05	30.00

Batch Adsorption Experiment

Working standard solutions of the dye were prepared by diluting the stock solution to obtain concentration specified in the factorial design (Table 1). The pH of the solution was adjusted using dilute hydrochloric acid and sodium hydroxide solutions. The experiment was performed at ambient temperature of 298K. Concentration of dye in solution at equilibrium was determined by taking absorbance reading on Jenway 6405 UV/vis spectrophotometer at λ_{max} 615nm. Removal efficiency (*R*) which was the response generated was calculated using Equation 1.

$$R(\%) = \frac{C_i - C_f}{C_i} \times 100 \qquad (1)$$

Where; C_i is the initial concentration of dye solution and C_f final concentration at equilibrium

RESULTS AND DISCUSSION

Removal Efficiency and Analysis of Variance for Adsorption of AB147 onto Activated Carbon

Presented in Table 3 is the variation of the selected factors affecting the adsorption process, and the corresponding percentage removal efficiency obtained after each experimental run was performed for the adsorption of AB147 dye onto the activated carbon. The results show that experimental run sixteen (16) gave highest removal efficiency value of 97.10% and the least value of 27.48% for the fourth run. The analysis of variance (ANOVA) values are presented in Table 4.

$$\text{Removal efficiency } (R_e)\% = 64.29 - 3.36A - 0.55B + 393.75C + 1.63D + 0.02AB + 118.75AC - 0.16AD + 16.44BC - 0.04BD - 21.88CD - 1.81ABC + 4.06 \text{ x } 10 - 3ABD - 0.13ACD + 0.54BCD - 0.03ABCD \quad (2)$$

Table 3: Design Matrix with Coded Values and Removal Efficiency for Adsorption of AB147 onto Activated Carbon.

Run	A:pH	B: AB147 Concentration (mg/l)	C:Carbon dosage (g)	D. Contact time (min)	*R*: Removal efficiency (%)
1	0	0	0	0	**88.90**
2	+1	-1	-1	+1	**73.20**
3	-1	-1	-1	+1	**87.50**
4	-1	+1	-1	+1	**27.48**
5	0	0	0	0	**88.90**
6	-1	-1	+!	-1	**94.05**
7	-1	+1	+1	-1	**96.50**
8	+1	+1	+1	-1	**93.46**
9	+1	+1	-1	+1	**31.85**
10	+1	-1	+1	+1	**84.15**
11	-1	-1	+1	+1	**95.05**
12	+1	+1	-1	-1	**33.90**
13	+1	+1	+1	+1	**93.98**
14	+1	-1	-1	-1	**61.70**
15	-1	+1	-1	-1	**33.48**
16	-1	+1	+1	+1	**97.10**
17	-1	-1	-1	-1	**70.85**
18	+1	-1	+1	-1	**89.90**

Table 4: ANOVA of the Factorial Model for the Adsorption of AB147 onto Activated Carbon.

Source	Sum of Squares	DF	Mean Square	F Value	Prob. > F	Remark
Model	10524.89	15	701.6593	63660000	< 0.0001	Significant
A	100	1	100	63660000	< 0.0001	
B	1383.84	1	1383.84	63660000	< 0.0001	
C	6569.103	1	6569.103	63660000	< 0.0001	
D	16.81	1	16.81	63660000	< 0.0001	
AB	86.49	1	86.49	63660000	< 0.0001	
AC	0.4225	1	0.4225	63660000	< 0.0001	
AD	4	1	4	63660000	< 0.0001	
BC	2120.603	1	2120.603	63660000	< 0.0001	
BD	57.76	1	57.76	63660000	< 0.0001	
CD	35.4025	1	35.4025	63660000	< 0.0001	
ABC	23.5225	1	23.5225	63660000	< 0.0001	
ABD	15.21	1	15.21	63660000	< 0.0001	
ACD	2.1025	1	2.1025	63660000	< 0.0001	
BCD	109.2025	1	109.2025	63660000	< 0.0001	
ABCD	0.4225	1	0.4225	63660000	< 0.0001	
Curvature	462.25	1	462.25	63660000	< 0.0001	Significant
Pure Error	0	1	0			
Cor. Total	10987.14	17				

Std. Dev.	0.000	R^2	1.0000
Mean	74.57	R^2_{adj}	1.0000
C.V.	0.000	CL	95%

From the Results of ANOVA, model F-value of 63660000.00 implies that the model is significant. There is only a 0.01% chance that a "Model F-Value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant. "Prob > F" value for all the terms was < 0.0001, in this case A, B, C, D, AB, AC, AD, BC, BD, CD, ABC, ABD, ACD, BCD, ABCD are significant model terms. The "Curvature F-value" of 63660000.00 implies there is significant curvature (as measured by difference between the average of the center points and the average of the factorial points) in the design space. There is only a 0.01% chance that a "Curvature F-value" this large could occur due to noise. Removal efficiency mean of 74.57 %, Coefficient of Variance. 0.000, Std. Dev of 0.000, $R^2$1.0000, and R^2_{adj}1.0000 were obtained at 95% confidence limit. The equation that defines each of the response obtained in the factorial design for the adsorption of AB147 onto activated carbon is given by Equation 2.

The Interaction Effects of Two Variables on the Adsorption of AB147 Dye from Solution

Carbon Dosage and Initial AB147 Concentration: The interaction effect of carbon dosage and initial AB147 concentration on the adsorption process is presented in Figure 2. The results indicate that with a carbon dosage of 0.05g, initial dye concentration of 60mg/l at pH 4 and 45 minutes contact time, 95.26% of the dye can be adsorbed from solution. Observation of Figure 2 showed that, if carbon dosage and initial dye concentration were the only factors affecting the adsorption process, then in application carbon dosage will be the most critical factor that requires serious attention due its influence on the removal of the dye from solution.

From the results, increase in the initial dye concentrations when carbon dosage was set at 0.01g indicates a decrease in percentage adsorption. Conversely, at a carbon dosage of 0.05g an increase in dye concentration resulted in an increase in amount of dye removed from the liquid stream. This finding is In agreement with the theory that increase in carbon dosage means increase in surface area, which provides more sites for uptake of the adsorbate. Also, it can be seen clearly that with a carbon dosage of 0.05g the adsorption profile is above the factorial model design point, while at 0.01g it is below the factorial model design point.

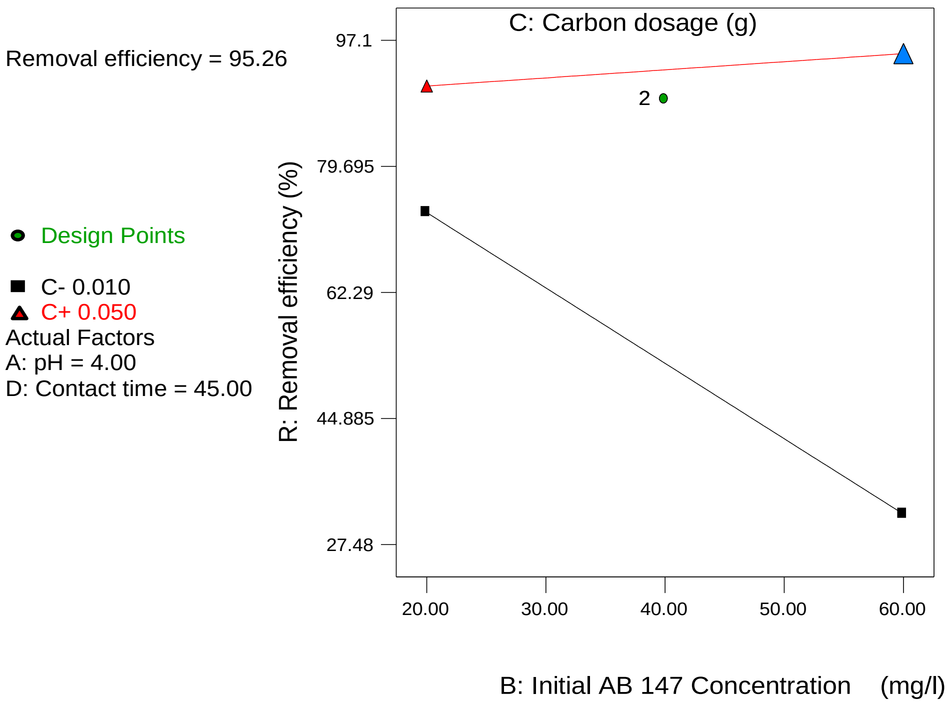

Figure 2: Interaction Effect of Carbon Dosage and Initial AB147 Concentration on the Adsorption Process.

Carbon Dosage and Contact Time: The interaction effect of contact time and carbon dosage on the removal of the dye from solution is presented in Figure 3. From the results obtained increase in contact time and carbon dosage produced an increase in removal efficiency. This means that as more time was allowed dye molecules occupied more of the adsorption sites in the adsorbent. Optimum amount of dye removed was 93.47% at 0.05g carbon dosage, contact time of 30 min, pH 4 and initial dye concentration of 40.0mg/l. Conditions that allows adsorption efficiency of ≥ 80% is said to be good (Thomas *et al*., 2008). Considering both factors, the results showed that 30 min contact time was sufficient for a very good removal of the dye from solution. With a carbon dosage of 0.05g the adsorption profile was above the factorial model design point. This result also indicate that carbon dosage is the most critical of all the factors influencing the adsorption of this dye.

Carbon Dosage and pH: The interaction effect of carbon dosage and pH on the adsorption process is presented in Figure 4. The results indicate that with a carbon dosage of 0.05g, initial dye concentration of 40mg/l at pH 3 and 45 minutes contact time, 95.675% of the dye can be adsorbed from solution. Observation of Figures 4 showed that, at higher pH values percentage removal of the dye from solution decreased. The dye is best removed from solution when the medium is acidic. If we assume carbon dosage and pH were the only factors affecting the adsorption process, then carbon dosage will be the most important. Again, it can be seen clearly that with a carbon dosage of 0.05g the adsorption profile is above the factorial model design point, while at 0.01g it is below the factorial model design point.

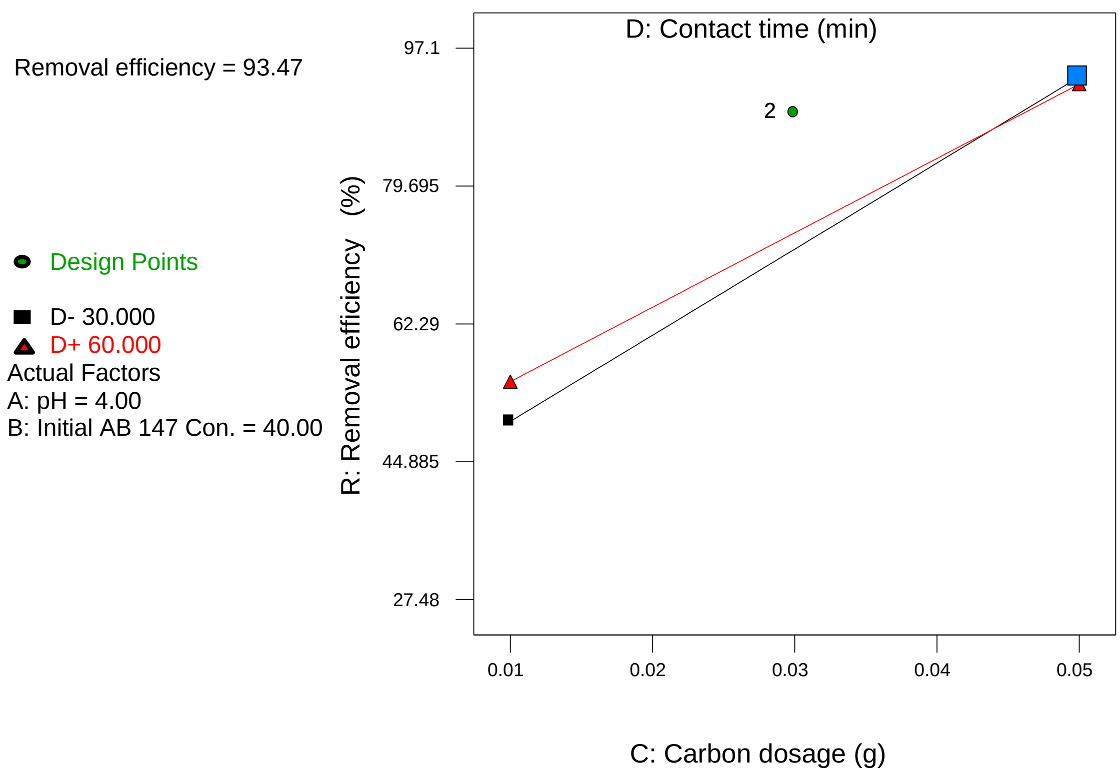

Figure 3: Interaction Effect of Carbon Dosage and Contact Time on the Adsorption Process.

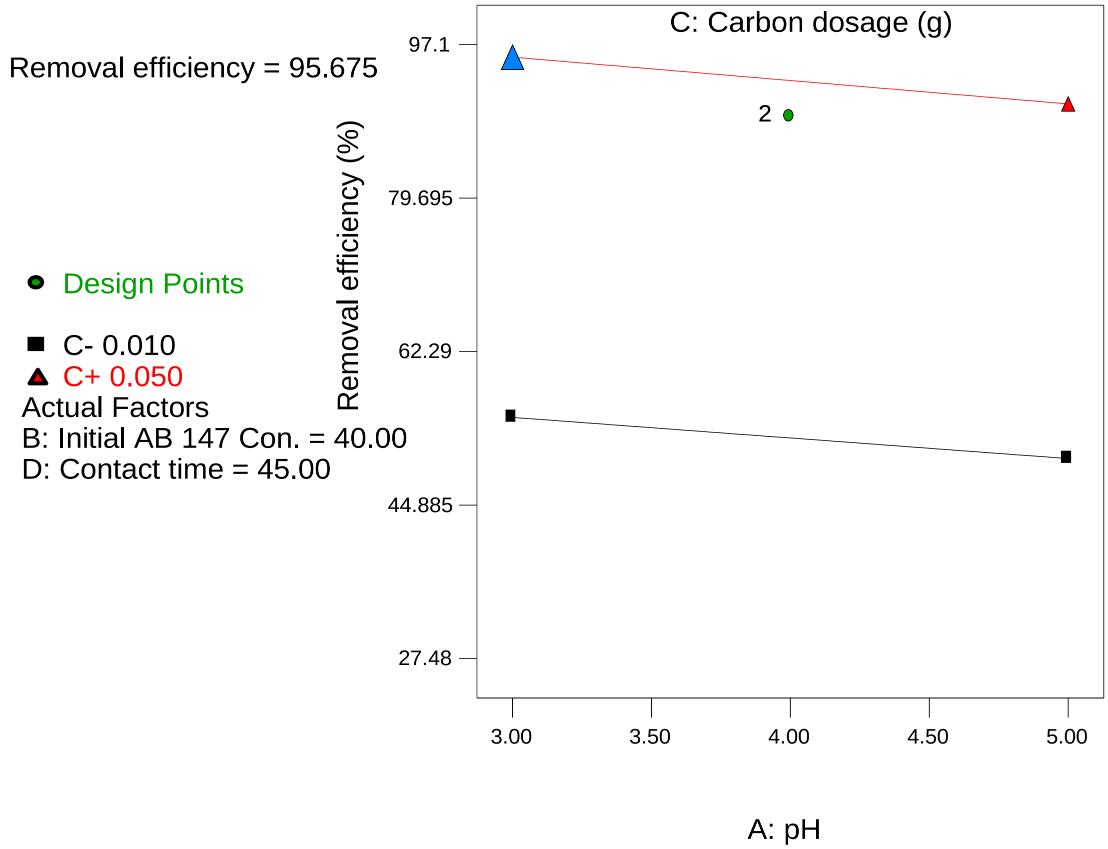

Figure 4: Interaction Effect of Carbon dosage and pH on the Adsorption Process.

The Interaction Effects of Three Variables on the Adsorption of AB147 Dye from Solution

Carbon Dosage, Initial AB147 Concentration and pH: The interaction effect of carbon dosage, initial dye concentration and pH on adsorption of AB147 from solution is presented in Figure 5. From the cube plot, removal efficiency of the adsorption process was 96.80% at contact time of 45 minutes when the three variables were C+, B+ and A-. This condition of high carbon dosage and initial dye concentration with low pH provided for a better removal of the dye from solution.

Considering these three variables the least amount of dye (30.48%) was adsorbed at B+, A- and C- with contact time of 45 minutes. This poor adsorption was observed when C was low and factors A and B remained unchanged. This again support the fact that adsorption is enhanced as carbon dosge increases, making it the most infuencial variable for the adsorption of AB147 unto the activated carbon. A close observation of the cube plot indicate percentage adsorption greater than 80 on the points where carbon dosage is high (C+). Values obtained at these points are 87.03, 93.72, 94.55, and 96.80%.

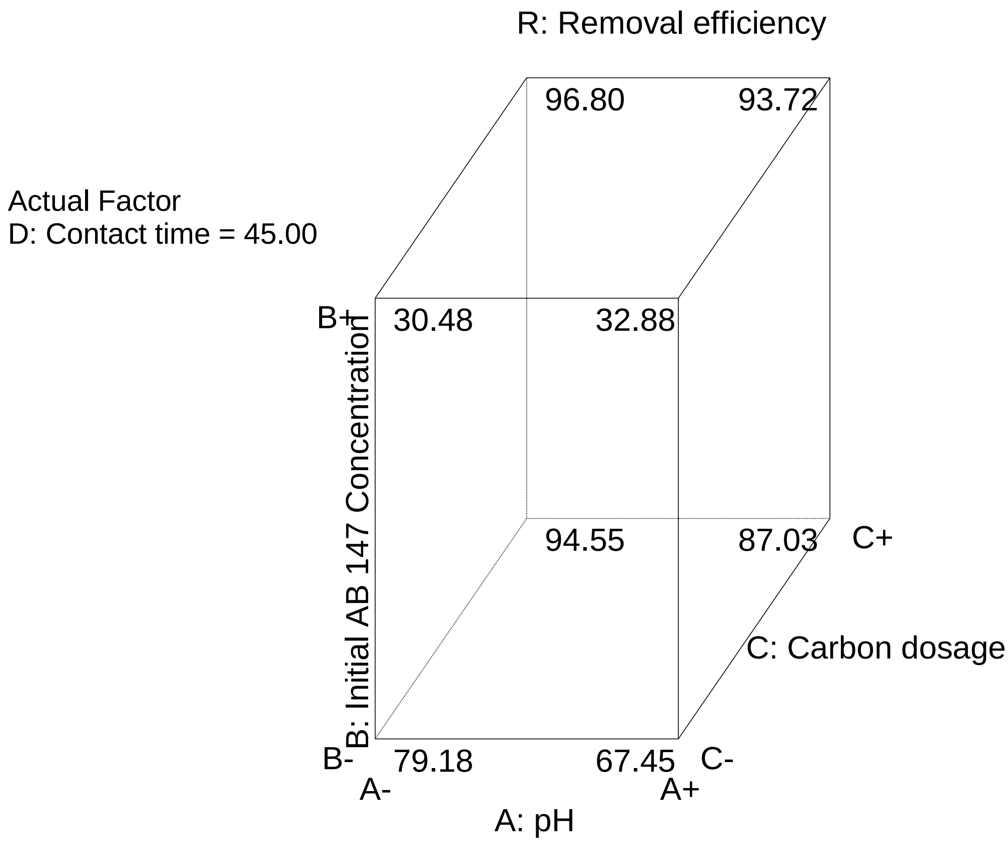

Figure 5: Interaction Effect of Carbon dosage, Initial AB147 Concentration and pH on Removal Efficiency.

Carbon Dosage, Initial AB147 Concentration and Contact Time: Figure 6 is the cube plot for the interaction effect of carbon dosage, initial dye concentration and contact time on adsorption of AB147 from solution. At pH 4, removal efficiency of the adsorption process was greater than 80% at the points where carbon dosage was high (C+). The highest value of 95.54% was obtained at C+, B-, and D-. and the lowest value of 29.67% at D+, B+, and C-. Despite the high initial AB147 concentration and high contact time, because of low carbon dosage the removal of the dye from solution was very poor with a value <30%.

Initial AB147 Concentration, pH and Contact Time: The interaction effect of initial dye concentration, pH and contact time on adsorption of AB147 from solution is presented in Figure 7. From the cube plot, removal efficiency of the adsorption process was 91.20% at carbon dosage of 0.03g when the three variables were A-, B- and D+. Considering these three variables change in pH value from 3 to 5 at B+ and D+ did not affect the amount of dye adsorbed because percent removed was 62.29% at both point. Observation of the cube plot indicate percentage adsorption >80 on the points (A-, B-, and D-) with the value of 82.45% and (A-, B-, and D+) of 91.28%. This observation suggest that at low initial dye concentration and pH increase in contact time without change in carbon dosage will increase amount of dye removed from solution

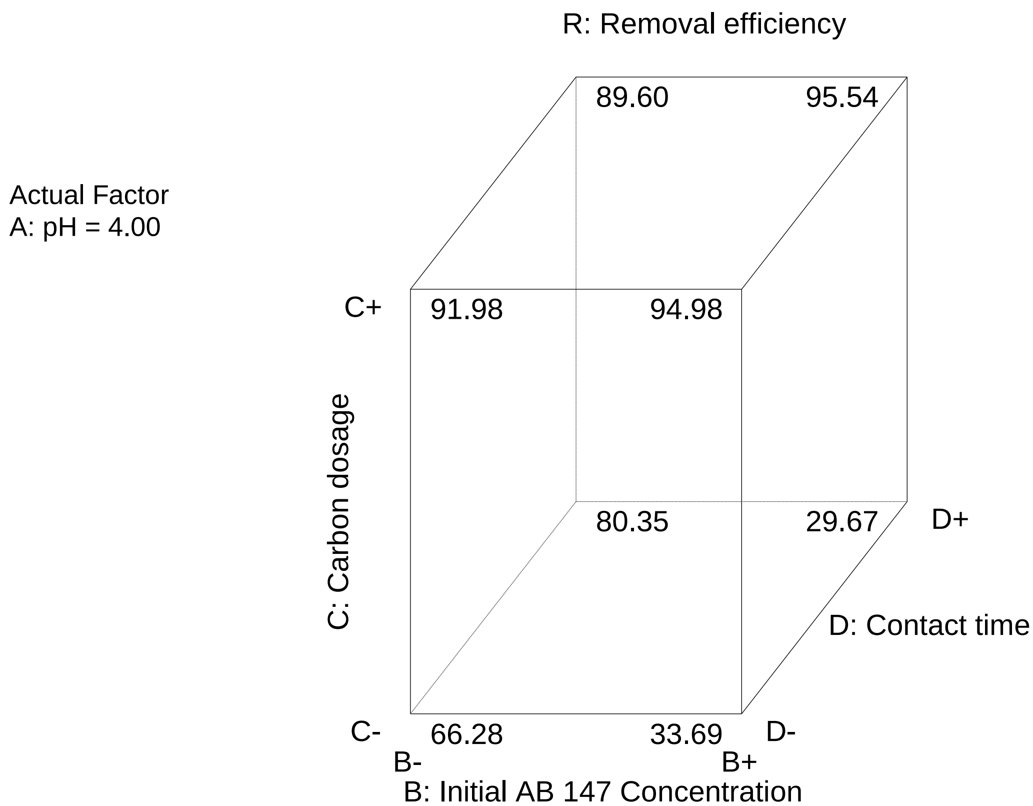

Figure 6: Interaction Effect of Carbon Dosage, Initial AB147 Concentration and Contact time on Removal Efficiency.

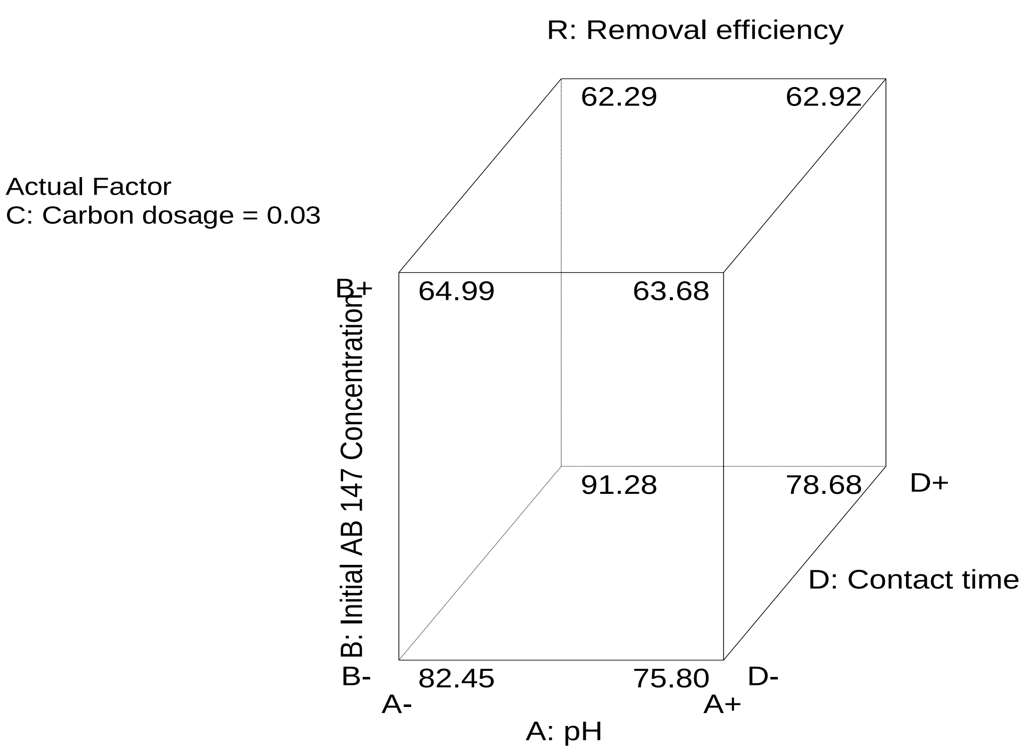

Figure 7: Interaction Effect of Initial AB147 Concentration, pH and Contact Time on Removal Efficiency.

CONCLUSION

The carbon adsorption at 615nm and 298K of AB147 dye was successfully investigated by statistical experimental design using full factorial model at two levels and four factors. Results obtained revealed that the main factor affecting the carbon adsorption process is carbon dosage. The major finding of this work is that as carbon dosage increases percentage removal of AB147 dye from solution also increases.

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SUGGESTED CITATION

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