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Optimitization of Crystal Violet Adsorption Performances on to H_3PO_4-Modified Mango Seeds Kernel using a Box-Behnken Experimental Design.

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Abstract: A biosorbent was produced over treatment of mango seeds kernel with a solution of phosphoric acid and used for Crystal Vipolet adsorption from aqueous solution. The adsorption capacity of the biosorbent was assess using a Box-Behnken experimental design (BBED). The pH value of 6 was considered as being convenient for this study. For this purpose, three key parameters were investigated namely, temperature, initial dye concentration and adsorbent mass. The amount of Crystal violet adsorbed was regarded as response function and was used for modeling and optimization. The results showed that responses of Crystal Violet adsorption were significantly affected by the interaction, singular and quadratic effects of temperature, adsorbent dosage and initial dye concentration. The optimal adsorbed dye amountof69.07 mg.g^{-1} was achieved with an initial Crystal Violet concentration of 75 mg.L^{-1} at 50°C, and adsorbent dosage of 0.1 g. Statistical analysis were performed in the form of the analysis of variance (ANOVA)and lack of fit adequacy which gave good interpretation of different interactions of experimental parameters, with highly significant with confidential level > 99%. The effect of mass of adsorbent is negative and it is in a 100 ordered of magnitude greater than the effect of temperature and initial dye concentration. However the quadratic effect of mass is in 5 ordered of magnitude greater than mass and is positive. This shows that H_3PO_4-mango seeds kernel is better adsorbent for Crystal Violet adsorption from aqueous solution than many others adsorbents including some activated carbons and biosorbents.
Key words: H_3PO_4-modified mango seeds kernel, Box Behnken experimental design, Crystal Violet, biosorbent, response surface methodology.

Introduction

Aqueous effluents are well known as massive industrial wastewater. The main pollution source of colored effluents comes from textiles, leather, printing, laundry, tannery, rubber painting etc. processes[1].

Untreated disposal of this colored water to a receiving water medium causes severe damages to aquatic life and to human being[2-5]. Removal of color from wastewater effluents is an important issue faced by textile dyes industries.

Many physical, chemical and biological treatment processes have been proposed for dyes removal from water[6-8]. Among these methods adsorption is one of the most popular nowadays[9-11]. This is due to the fact that adsorption is simple and efficient technique [12]. Activated carbon as the most widelyused adsorbent dyes removal from aqueous solution[13-17], but its high cost limits its application. Therefore, many studies have been carried out to develop alternative and low cost adsorbents[18-20]. The recent literature survey revealed that several industrial and agricultural wastes can be used for this purpose[21-32].

Recently, H_3PO_4-modified mango seeds kernelpowder (AMP), has been tested as a low cost adsorbent for dye removal from aqueous solution[24,30,31].Unfortunately, most of the above studies are carried out in one variable at a time method. This method does not give any information on the interaction among variables and best condition for optimal dye removal can not be found. Hence, the aim of this study is to use the Box-Behnken experimenntal design (BBED) to assess Crystal Violet adsorption from aqueous solution onto H_3PO_4-modified mango seeds kernel powder.

1. Experimental

1.1. Material

Mango seeds kernel used as adsorbent in this study were sampled in Ngaoundere a locality, in the Adamawa Region in Cameroon. Mango seeds kernel were treated by concentrated phosphoric acid (H_3PO_4), then washed with deionized water and dried at 110°C for 24 h. The dried mango seeds kernel were grounded to fine powder and sieved to a particle size lower than (50µm).

Crystal Violet is a commercial grade (M_F: $C_{25}H_{30}N_3Cl$, M_W: 408, λ_{max}: 586 nm) and it was used without further purification (Fig. 1). Stock solution of Crystal Violet was prepared by dissolving 1g of the CV powder in 1000 mL distilled water. For adsorption experiments this solution was diluted to the desired initial concentrations ranging from 50-300 $mg \cdot L^{-1}$.The initial solution pH was adjusted to the desired value by adding drop wise solution of HCl, 0.1M or solution of NaOH, 0.1M solutions.

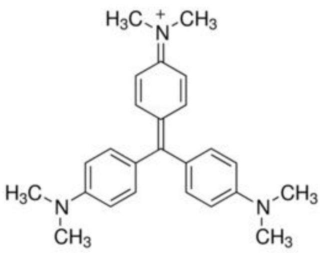

Figure 1: Crystal Violet molecule

1.2. Batch adsorption experiments

A predetermined amount of AMP was added into 100 mL conical flasks filled with 25 mL of CV solution of known concentration. The flasks were then placed in a shaking water bath (50 $cycles \cdot min^{-1}$) at room temperature for 24 hours. The samples were then withdrawn at predetermined time interval and the residual concentration of CV was determined by UV-Visible spectroscopy. The absorbance was measure at 586 nm with 1 cm optical path length quartz cell. TG Instrument T-70 UV-Vis spectrophotometer was used for this purpose.The concentration of CV was determined from calibration curve.

The dye removal efficiencywas expressed as:

$$q_t = (C_o - C_f)\frac{V}{m} \qquad (1)$$

where C_o is the initial concentration of dye ($mg\cdot L^{-1}$), $C_f$is the final concentration of dye ($mg\cdot L^{-1}$), m the adsorbent mass and V de volume of the solution.

1.3.Response surface methodology (RSM)

A 3-factor 3-level factorialBox–Behnken experimental design (BBED) matrix wasused to investigate the effects of selected parameters. BB designs are response surface methods (RSM), specially made to require only 3 levels, coded as −1, 0, and +1. BB designs are formed by combining two-level factorial designs with incomplete block designs that the geometry of this design suggests a sphere within the process space such that the surface of the sphere protrudes through each face with the surface of the sphere tangential to the midpoint of each edge of the space [33]. This procedure creates designs with desirable statistical properties but, most importantly, with only a fraction of the experiments required for a three-level factorial (3^{k-p}) designs. Since there are only three levels, the quadratic model is appropriate. The observations were fitted to a second order polynomial model as given below:

$$Y = a_0 + \sum a_i X_i + \sum a_{ij} X_i X_j + \sum a_{ii} X_i^2 \qquad (2)$$

where Y is the predicted response associated with each factor level combination, a_0 is the intercept term, a_i is linear effect, a_{ii} is the quadratic effect, and a_{ij} is the 2-waylinear by linear interaction effect. Additionally, X_i and X_j representthe real values of independent variables. The important variables chosen for the BBED are temperature,mass dosageand initial concentration of Crystal Violet designated as X_1, X_2 and X_3, respectively. The dependent variable (Yi) is theamount of adsorbed dye per gram of adsorbent (q). The range of experimentaldesign matrix is shown in Table 1.

Table 1: Factors and levels of the Box-Behnken design.

Variables	Factors	Levels		
		Low	Middle	High
X_1	Temperature (°C)	30	40	50
X_2	Masse (g)	0.1	0.2	0.3
X_3	Concentration ($mg.L^{-1}$)	25	50	75
Response				
Y	Adsorbed Amount(mg/g)			

1.4. Statistical analysis

The regression analysis, statistical significance and response surfaces were obtained to find the most suitable combination of factors resulting in maximumCrystal Violet adsorption capacity of AMP for predicting the responses. The statistical significance of variables was evaluated using the analysis of variance (ANOVA). Adequacy of the constructed models was investigated via lack of fit, coefficient of determination (R^2) and F-values. Statgraphics software package was used for the regression, residual and graphical analyses.

2. Results and Discussion

2.1. Physico-chemical properties of AMP samples.

Table 2 shows some physicochemical properties of AMP obtained after analysis.

Table 2: Properties of AMP

Designation	Value
pH_{pzc}	5.4
Mass loss (mg)	71.4
BET surface ($m^2.g^{-1}$)	44.57
Volume ($cm^3.g^{-1}$)	10.24

2.2 Development of Regression model equation for Crystal Violet removal

The complete design matrix and values of both responses obtained from the experimental works are given in Table 3. BBED is a modeling technique used to evaluate the correlation between the experimental variables to the adsorption of CV in adsorbent and corresponding response of dye adsorbed quantity from aqueous solution. The maximum adsorbed amount of dye was found to be >59mg.g^{-1}. The second order polynomial equation fitted between the responses representsadsorbed quantities (Y) and the input variable of temperature (X_1), mass (X_2)and initial concentration of dye (X_3). The empirical model in terms of actual factors for adsorption of Crystal Violetin adsorbent (Y) is given in Eq. 3:

$$Y = - 37.87 + 2.99X_1 - 102{,}64X_2 + 0.26X_3 - 0.027X_1X_1 - 4.37X_1X_2 + 0.011X_1X_3 + 475.50X_2X_2 - 0.79X_2X_3 - 0.0037X_3X_3 \quad (3)$$

In order to develop the regression model that is statically significant, the insignificant terms in the Eq. 3 are eliminated. The terms with p values more than 0.05 are significant and hence removed to obtain the regression model

$$Y = - 17.60 + 1.42X_1 - 111.66X_2 + 0.67X_3 - 4.37X_1X_2 + 498.05X_2X_2 - 0.79X_2X_3 \quad (4)$$

The effect of mass of adsorbent is negative and is 100 times greater than the effect of temperature and initial dye concentration. However the quadratic effect of mass is 5 times greater than mass and is positive. The equation has been used to observe the effects of experimental factors on removalefficiency. The experimental and predicted values ofadsorbed amount of Crystal Violet are given in Table 3. Experimental values are measured for a particular run, and the predicted values are evaluated from the model and are generated by using the approximating functions. The predicted R^2is99.97%. The correlation coefficients might have resulted from the insignificant terms in Table 4, and most likely due to four different variables selected in wide ranges with a limited number of experiments as well as the non-linear influence of the investigated parameter on process response.

Table 3: Experimental and predicted values of the BBEDmatrix for Crystal Violet uptake.

Run	X_1	X_2	X_3	Adsorbed Amount ($mg.g^{-1}$)		
				Experimental	Predicated	Residue
1	30	0.30	50	17.01	17.52	0.51
2	50	0.20	25	15.12	15.92	0.80
3	40	0.10	75	59.94	60.96	1.02
4	50	0.30	50	19.58	19.80	0.22
5	40	0.30	75	31.35	31.65	0.30
6	30	0.20	25	10.36	10.87	0.51
7	40	0.10	25	31.85	31.55	-0.30
8	50	0.10	50	54.40	53.89	-0.51
9	30	0.20	75	31.16	30.36	-0.80
10	30	0.10	50	34.35	34.14	-0.22
11	50	0.20	75	47.85	47.34	-0.51
12	40	0.30	25	11.17	10.15	-1.02
13	40	0.20	50	28.66	29.28	0.62
14	40	0.20	50	29.63	29.28	-0.35
15	40	0.20	50	29.55	29.28	-0.27

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2.4. Analysis of variance (ANOVA) BBED for Crystal Violet uptake

ANOVA of Crystal Violet removal obtained in the study is presented in the Table 4. In general terms, ANOVA explains any variation in the statistically derived model and significance of the model parameters.The degrees of freedom (DF), sum of squares (SS) quadratic mean standard deviation probability and Fratio are calculated and tabulated in the ANOVA table. Normally, a lower P value of a model term in ANOVA indicates good significance of theterm over others. In this study, the models obtained for Crystal Violetdye removal were able to explain the experimental results on color removal. The regression model, linear model and square model allfitted well with 100% confidential level. The interaction of the parameter temperature, adsorbent dosage and initial concentration of Crystal Violetadsorption is also highly significant withconfidential level> 95%.Figure 2 shows predicted versus actual plots for adsorbed amount, demonstrating that the actual values are distributed relatively near to the straightline. This indicates that the models are adequate for predicting the color removal efficiency within the range of the variables studied.The test of lack of adequacy is used to determine if the selected model can be used to describe experimental data or if another complicated one should be chosen. If the probability for lack of fit is bigger than 0.05 there is evidence that the quadratic linear regression model is more appropriate to explain the relationship of the parameters on CV dye adsorption. In the present study, the observation is true where probability of lack of fit is lower than that of that of pure errors sum of square and quadratic mean.

Table 4 : Analysis of variance (ANOVA) for RSM for Crystal Violet uptake

Run	Sum of Square	Quadratic Mean	Standard Deviation	Degree of Freedom	Proba.	F ratio
X_1	242.77	242.77	0.31	1.00	0.00	837.43
X_2	1286.01	1286.01	0.38	1.00	0.00	4436.03
X_3	1295.41	1295.41	0.38	1.00	0.00	4468.45
X_1X_1	26.92	26.92	0.38	1.00	0.01	92.85
X_1X_2	76.39	76.39	0.56	1.00	0.00	263.50
X_1X_3	35.58	35.58	0.54	1.00	0.01	122.74
X_2X_2	83.48	83.48	0.54	1.00	0.00	287.97
X_2X_3	15.64	15.64	0.56	1.00	0.02	53.96
X_3X_3	0.77	0.77	0.54	1.00	0.24	2.67
Lack of adequacy test	4.67	1.56		3.00	0.16	5.37
Pure Error	0.58	0.29		2.00		
Total (corr.)	3076.00			14.00		

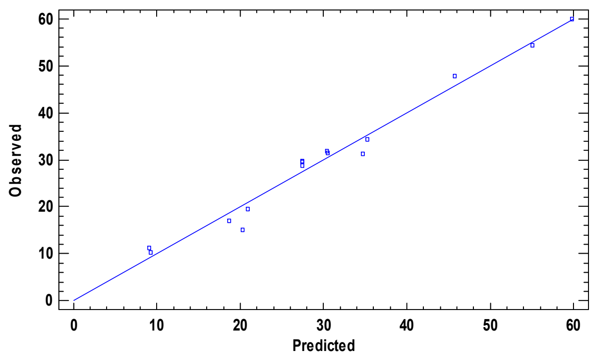

Figure 2. Experimental vs. Predicted of Crystal Violet adsorbed.

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2.4. Estimation of Quantitative Effects of the Factors

The three dimensional response surface and contour plot obtained from second polynomial equations are shown in Figs.3-5. The optimum values of the variable were obtained as the response is maximized using the RSM technique. The best response range can be obtained by analyzingthe response surface plots. The combined effect of temperature and adsorbent dosage, at constant initial concentration of dye (50 mg.L^{-1}) and pH (6),for the adsorption of Crystal Violet from aqueous solution are shown in Figure 3. The adsorbed quantity decreased with increasing temperature and initial dye concentration. This is due to the decrease in the number of binding sites on the adsorbent surface. It is clear from this figure that the adsorbed amount decreases with increase of adsorbent dosage from 0.1 to 0.3g. It is noted that the maximum adsorption at all the temperature takes place at 0.1g. The maximum adsorbed amount is 54.4 mg.g^{-1} was observed at constant temperature (50°C), initial dye concentration (40.0mg.L^{-1}) and pH (6).

Figure 4 shows the three-dimensional response surface for the interaction effect of temperature and initial dye concentration on adsorption of CV at constant adsorbent dosage (0.2g) and pH(6). Increasing the temperature from 30 to 50 °C facilitated the removal efficiency of CV, probably due to the increase in the mobility of the large dye ions. The increase in amount of dye sorption increases with temperature also due to either higher affinity of sites for dye or an increase in number of binding sited on absorbent. A maximum adsorption of dye(47.84 mg.g^{-1}) was observed at fixed concentration of dye (50 mg.L^{-1}) and temperature (50°C). Enhancement of adsorption capacity of CV at higher temperature may be the possibility of an increase in the porosity and in the total pore volume of the adsorbent [34]. The interactive effect of adsorbent dosage and concentration of the solution on the removal of Crystal Violet at constant pH (6) and temperature (40.0°C) is shown in Figure 5. The adsorbed amount decreases with the increased of adsorbent dosage and initial concentration. This is due to the decrease in the number of binding sites on theadsorbent surface. It is clear from figure 5 that the adsorptionof Crystal Violet is efficient at initial dye concentration of 75mg.L^{-1} for 0.1g of adsorbent dosage.

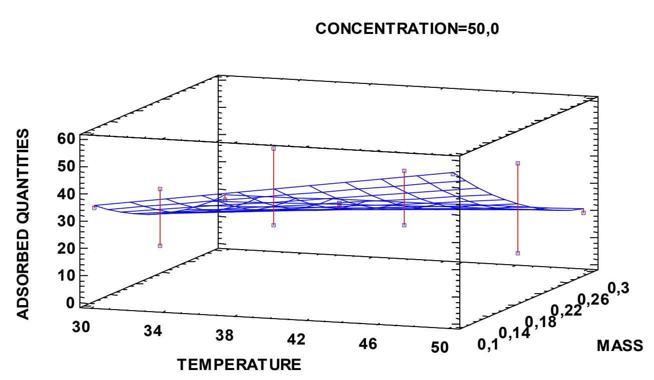

Figure 3. Effect of temperature and adsorbent dosage, on the adsorption of CV at constant initial concentration of dye (50 mg·L^{-1}) and pH (6).

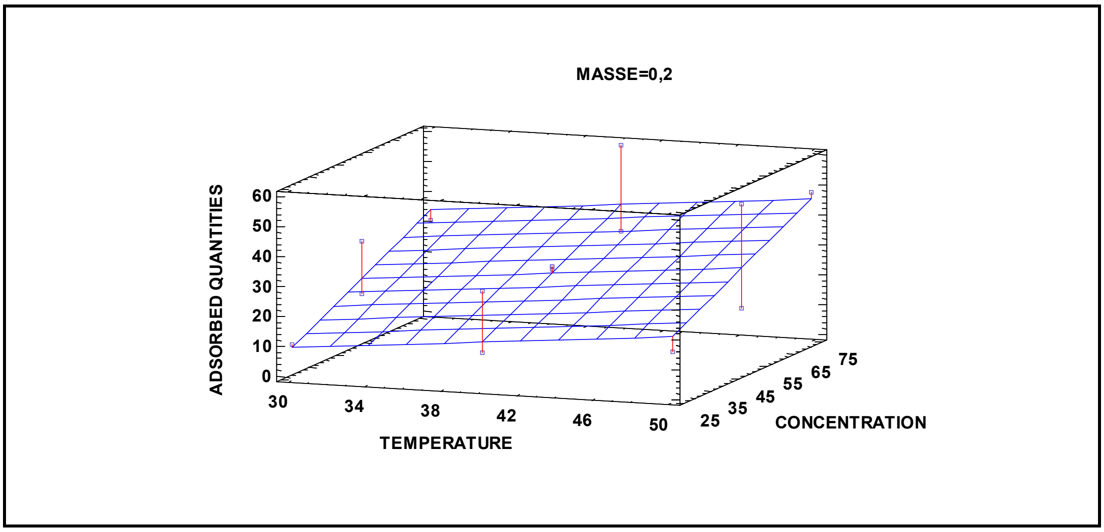

Figure 4. Effect of temperature and initial concentration, on the adsorption of Crystal Violet at constant adsorbent dosage (0.2g) and pH (6).

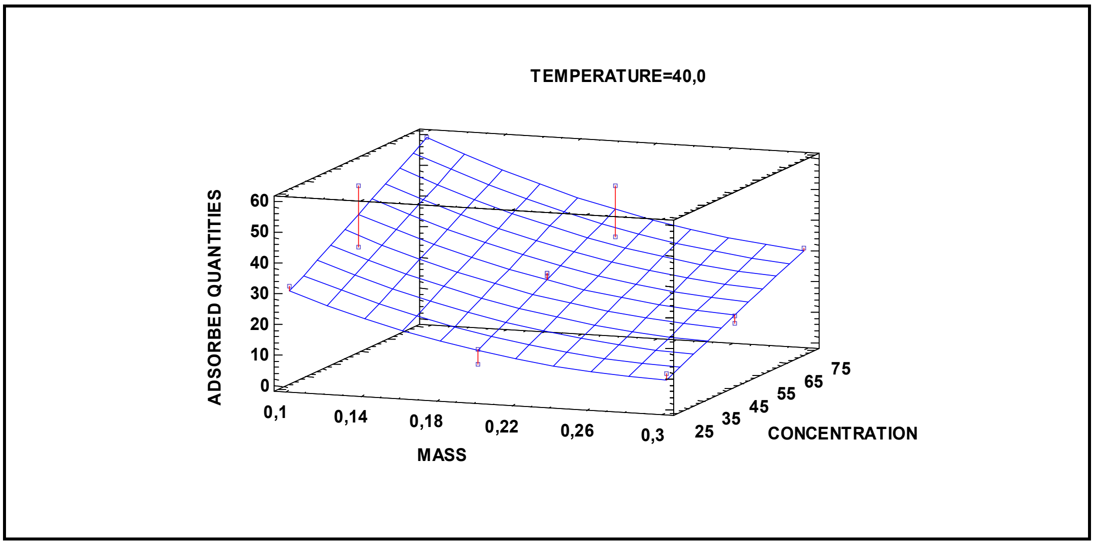

Figure 5. Effect of adsorbent dosage and initial concentration, on the adsorption of Crystal Violet at constant temperature (40.0°C) and pH (6).

Table 5 : Crystal Violet adsorption capacity of some adsorbents

Adsorbents	q ($mg.g^{-1}$)	References
H_3PO_4 activated carbon (PAAC)	60.42	[35]
H_2SO_4 activated carbon (SAAC)	85.84	[35]
Carbon from Jute fibers	27.99	[36]
Kaolin	47.27	[37]
Coniferous pinus bark powder (CPBP)	32.79	[38]
Semi-IPN hydrogel	35.09	[39]
Nano magnetic composite	81.70	[40]
H_3PO_4-modified mango seeds kernel	69.07	This study

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Conclusion

The adsorptionefficiency of H_3P0_4- mango seeds kernel was assessed using Crystal Violet dye solution. Adsorption experimental process parameters namely temperature, initial concentration and adsorbent mass were considered to optimizethe removal of Crystal Violetusing response surface methodology.it was found that adsorbent dosage was the key factor for Crystal Violet removal. Interactions among variables are low as showed by BBED analysis. The regression analysis, statistical significance and response surface well predicted the responses in experimental regions. The multiple correlation coefficient of determination $R^2$was 99.97%, showing that the actual data fitted well with the predicted data. The lack of adequacy test showed that this model fit very well experimental data. The optimal condition for Crystal Violet adsorption is using H_3P0_4- mango seeds kernelis 50°C, 75 mg L^{-1} and 0.1 g respectively for temperature, initial concentration and adsorbent dosage. For this optimal condition, the amount of Crystal Violet adsorbed is 69,07 mg.g^{-1}. This study showed us that H_3P0_4- mango seeds kernels have good potential for the removal of Crystal Violet as compared with other adsorbents in table 5.

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