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**Research Article**

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# Decolorisation of synthetic dye by Guava (*Psidiumguajava*) leaf powder – A statistical approach

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**ABSTRACT**

*Guava (Psidiumguajava) leaf powder was used as an adsorbent to remove a synthetic dye Coomassie Brilliant Blue. A two level full-factorial design of three factors namely, pH, adsorbent dosage and temperature were performed and the effects of each parameter was analysed by statistical techniques. A regression model was proposed and it was statistically significant. pH had a major negative effect on biosorption process and it had an interaction effect with temperature.*

**Keywords:** Biosorption, Guava Leaf Powder, Coomassie Brilliant Blue, Factorial Design, Design of experiments

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## INTRODUCTION

Synthetic dyes are used to color textile paper, leather, wood inks, food items and metals. Textile industries are the major source of releasing synthetic dyes into water bodies. By and large, these synthetic dyes contain aromatic and heterocyclic compounds and some of them are toxic and carcinogenic. These dyes are very difficult to degrade and thus pose an environmental threat [1].The presence these dyes in water bodies are not only highly toxic to aquatic life but also may cause various problems in human beings like, respiratory problems and gastrointestinal problems [2].

There are a lot reports on removal of dyes from textile waste effluents based on physical, chemical and biological methods[3,4]. The possible use of Guava (*Psidiumguajava*) leaf powder (GLP) as a cheap absorbent to remove methylene blue from waste water has been well established by [5]. Recently,equilibrium studies on biosorption of metalsby GLP have been investigated by [6,7].

In the conventional approach of “one variable at a time (OVAT)”, the significant process parameters are screened by altering only one variable at a time and by keeping all other factors constant. Since this approach involves many experiments, it is often time consuming and laborious. But astatistical screening method called “FFD” consists of performing minimum number of experiments at a particular factor level combination. In contrast to OVAT, the FFD involves changing all the significant process variables from one experiment to the next. Therefore, it is possible to find out the interaction between the process variables [8, 9].

Coomassie Brilliant Blue (CBB) is a synthetic dye which is the commercial name of two similar triphenylmethane dyes and is widely used in textile industry[10].The objective of this study was to remove CBB from waste water by using Guava leaf powder as an absorbent. A FFD with three factors pH, dosage and temperature were taken and adsorption studies were performed.

## EXPERIMENTAL SECTION

Pure CBB was purchased from Merck and used to prepare standard CBB stock solution with double distilled water. It has the following characteristics: $\lambda_{max}$ = 580nm; Chemical formula = $C_{45}H_{44}N_3NaO_7S_2$; F.W = 825.97 g/mol; C.I = 42660

**Preparation of adsorbent:**
Guava leaf powder (GLP) adsorbent was prepared according to the method described by [5].

**Preparation of dye solution:**
A stock solution of 1000 mg/l of dye was prepared by mixing 1g of CBB dye in one liter of double distilled water and different concentrations were prepared by proper dilution. The % dye removal (η) was calculated by using the formula:

$$\eta = \left(\frac{D_0 - D_t}{D_0}\right) 100$$

Where, $D_o$&$D_t$= Initial and final dye concentration respectively.

**Adsorption experiments:**
All the experiments were carried out by batch mode. Known amount of GLP was added to 100ml of dye solution in a 250 ml conical flask with required pH and was agitated in aincubated shaker at a constant speed of 200 rpm. After 2 hours of contacting time, the samples were centrifuged and the final concentrations were analysed using UV–visSpectrophotometer (Shimadzu, Japan). Eight experiments with combination of all variables were performed in duplicate and a matrix of high (+) and low levels ( – 1) are given in the Table 1.

**Table 1: Levels of factors studied**

| Factor | Low value ( – 1) | High value ( + 1) |
|---|---|---|
| pH | 2 | 8 |
| Adsorbent dosage (g/L) | 1 | 5 |
| Temperature (°C) | 25 | 40 |

**Full Factorial Design: (FFD)**
One of the screening methods available to determine significant process variables is two-level full factorial design (FFD). In this method, all the factors are fixed at only 2 levels, high (+) and low (–). In FFD, $2^k$ number of experiments is carried out at different combinations of the factors. Even though no single pair of conditions is replicated, the main effect of each factor is measured at 2 levels of the other factor. This hidden replication in this factorial design increases the accuracy of the results. Moreover, it is possible to find out the interaction effect among the factors in this design [8, 9].

In the present study, three factors (pH, dosage and temperature) were taken and $2^3$ experiments (number of experiments = 8) were performed in duplicates. The coded and uncoded values with the % dye removal were given in the Table 2. The results were analysed by using MINITAB 15 Software.

## RESULTS AND DISCUSSION

The experimental results with the factors (coded and uncoded) are shown in the Table 2.

**Table 2: Coded and uncoded values of the factors with % dye removal**

| Coded Values | | | Uncoded Values | | | Dye Removal (%) | |
|---|---|---|---|---|---|---|---|
| pH | D (g/L) | Temp (°C) | pH | D (g/L) | Temp (°C) | Trial 1 | Trial 2 |
| -1 | -1 | -1 | 2 | 1 | 25 | 86.32 | 84.83 |
| 1 | -1 | -1 | 8 | 1 | 25 | 76.55 | 79.33 |
| -1 | 1 | -1 | 2 | 5 | 25 | 84.31 | 84.77 |
| 1 | 1 | -1 | 8 | 5 | 25 | 78.99 | 79.48 |
| -1 | -1 | 1 | 2 | 1 | 40 | 81.85 | 81.52 |
| 1 | -1 | 1 | 8 | 1 | 40 | 81.14 | 85.23 |
| -1 | 1 | 1 | 2 | 5 | 40 | 87.47 | 83.21 |
| 1 | 1 | 1 | 8 | 5 | 40 | 80.11 | 82.94 |

For this $2^3$ FFD, the model equation in terms of coded values is given by,

$$\eta = 82.378 - 1.907\ pH + 0.282\ D + 0.556\ T - 0.373\ pH*D + 1.328\ pH*T + 0.217\ D*T - 0.956\ pH*D*T$$

**Table 3: Estimated Effects and Coefficients for % Dye Removal (coded units)**

| Term | Effects | Coefficients | Standard Error | T | P |
|---|---|---|---|---|---|
| Constant | | 82.378 | 0.4567 | 180.38 | 0.000* |
| pH | – 3.814 | – 1.907 | 0.4567 | – 4.18 | 0.003* |
| D | 0.564 | 0.282 | 0.4567 | 0.62 | 0.554 |
| T | 1.111 | 0.556 | 0.4567 | 1.22 | 0.258 |
| pH x D | – 0.746 | – 0.373 | 0.4567 | – 0.82 | 0.438 |
| pH x T | 2.656 | 1.328 | 0.4567 | 2.91 | 0.02* |
| D x T | 0.434 | 0.217 | 0.4567 | 0.47 | 0.648 |
| pH x D x T | – 1.911 | – 0.956 | 0.4567 | – 2.09 | 0.07 |

** denotes significance at 95% confidence level (P<0.05)*

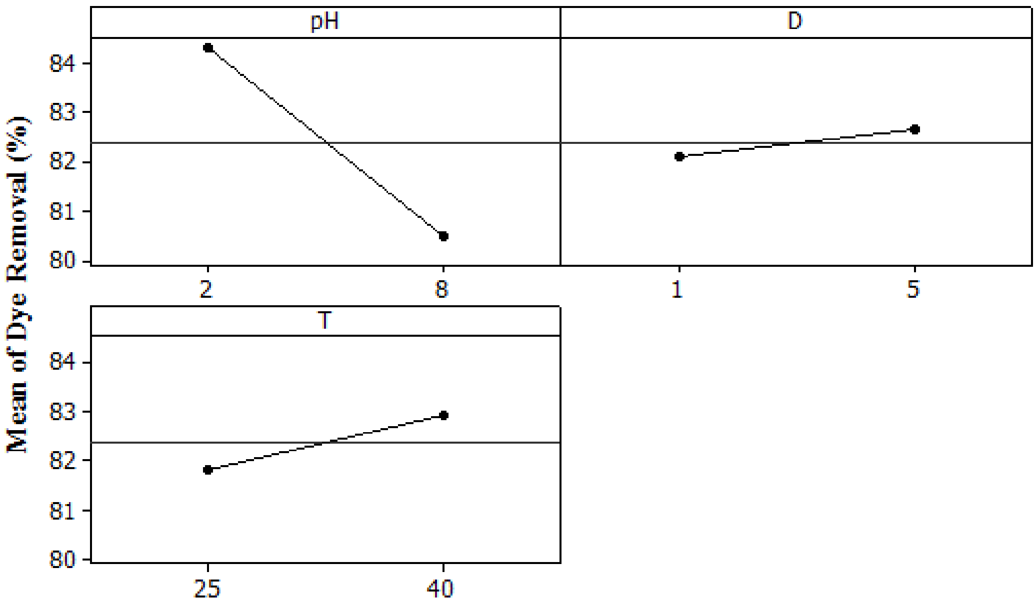


**Figure 1: Main effect of factors on % dye removal**

This regression model was significant at 95% confidence level. The effects, regression coefficients, standard errors, T and P are shown in Table 3. The effect or main effect representsaverage deviations between high and low levels for eachone of the factor.When the effect of a factor is positive, increase in % dye removal occurs as the factor is changed from low to high levels.In contrast, if the effectis negative, a decrease in % dye removaloccurs for high level of the same factor [9].Fig.1 shows the main effect plot of three factors on % dye removal, in which except pH all other factors show positive effect.

**Pareto chart:**
The significance of individual and interaction effects is given by Pareto chart (Fig 2). Based on Student's t-test, for eight degrees of freedom (DF) and 95% confidence level ($\alpha = 0.05$) the t – value is equal to 2.31. The absolute effects were compared with this t-value. From this chart it is clear that only the factor pH (A) and the interaction between pH and temperature (AC) are significant at 95% confidence level.

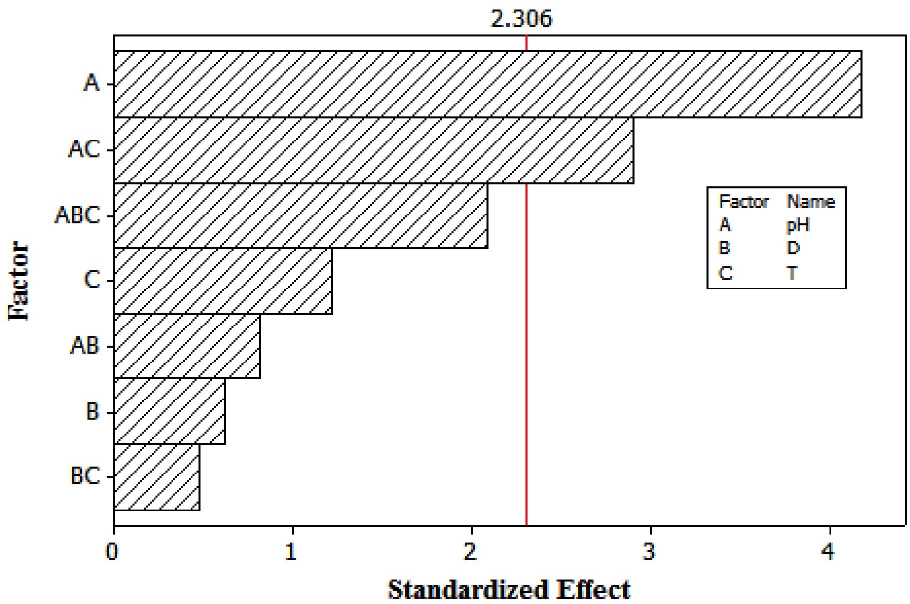


**Figure 2: Pareto chart of standardized effects on the % dye removal**

**Analysis of variance (ANOVA):**
In Table 4,the sum of squares (SS), mean squares (MS), F-ratios and P values are shown. The value of $F_{crit} = F(0.05,1,8) = 5.32$, and therefore all the effects having F value greater than 5.32 are statistically significant. From the table the factor pH and the interaction between pH and temperature are significant. Moreover these have a value of P $< 0.05$, and hence the null hypothesis can be rejected. Fig.4 shows normal probability plot of residuals which is a straight line and therefore all the errors are normally distributed.

**Table 4: Analysis of Variance for % Dye Removal (coded units)**

| Source | DF | Sum of Squares | Mean Square (MS) | F-ratio | P |
|---|---|---|---|---|---|
| pH | 1 | 58.179 | 58.1788 | 17.43 | 0.003* |
| D | 1 | 1.271 | 1.2713 | 0.38 | 0.554 |
| T | 1 | 4.94 | 4.9395 | 1.48 | 0.258 |
| pH x D | 1 | 2.228 | 2.2276 | 0.67 | 0.438 |
| pH x T | 1 | 28.223 | 28.2227 | 8.46 | 0.02* |
| D x T | 1 | 0.753 | 0.7526 | 0.23 | 0.648 |
| pH x D x T | 1 | 14.612 | 14.6115 | 4.38 | 0.07 |
| Residual Error | 8 | 26.697 | 3.3371 | | |
| Total | 15 | 136.901 | | | |

** denotes significance at 95% confidence level (P<0.05)*

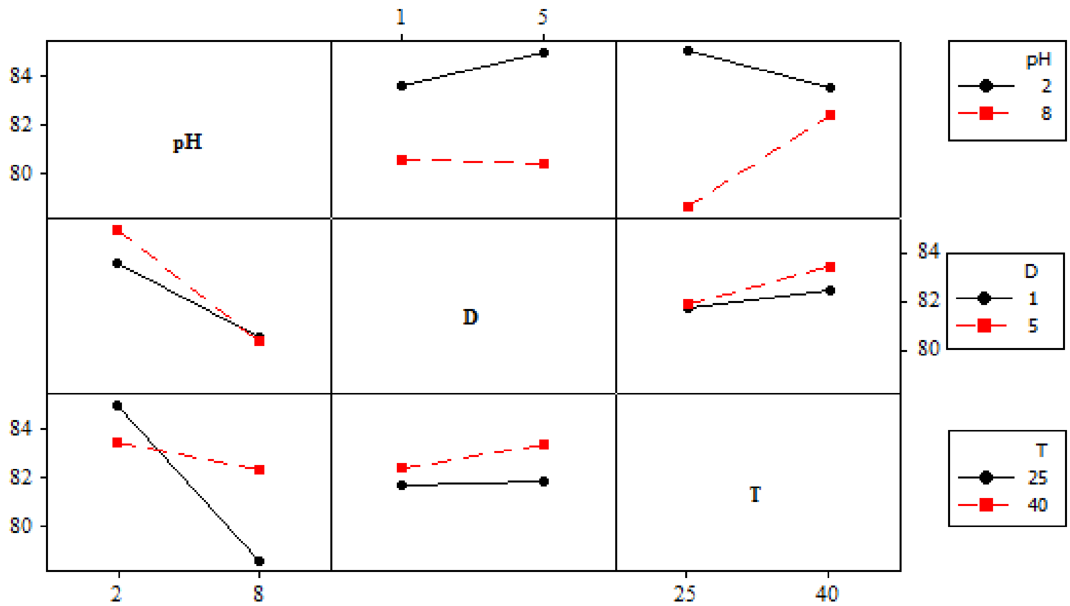


**Figure 3: Interaction effect of factors on % dye removal**

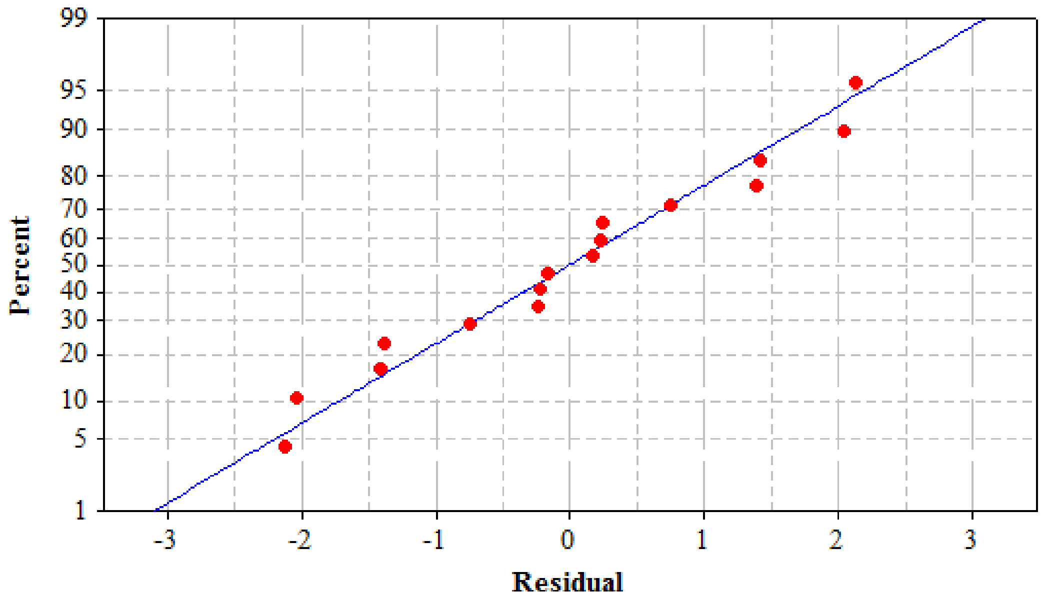


**Figure 4: Normal probability plot of residual values of % dye removal vs. predicted values**

**Effect of pH:**
The physical parameter pH dominates the dye removal process. As mentioned in the Table 3, pH has a negative effect. Increasein pH from 2 to 8 decreases the adsorption efficiency by about3.814%. A decrease in pH favors the adsorption of CBB on GLP. Similar kinds of results were reported by [11].

**Effect of adsorbent dosage:**
As the adsorbent dosage increases, the % dye removal increases because of the availability of more GLP particles[12]. In this study, adsorbent dosage has a positive effect on % dye removal.

**Effect of temperature:**
Generally increase in temperature increases adsorption to some extent and decreases further because of desorption[13]. The increased adsorption may be due to the increase in porosity of the GLP particles with the increase in temperature. In the temperature range studied (25 to 40°C), temperature has a positive effect on dye removal.

**Interaction between pH and temperature:**
From the interaction plot (Fig 3), we can infer that when the pH is increased from 2 to 8 at a constant temperature of 40ºC, the % dye removal increases from 78.59 % to 82.36%. In contrast to this, when the pH is increased from 2 to 8 at a constant temperature of 25ºC, the % dye removal decreases from 85.06 % to 83.51%. Since the effect of pH depends on the levels of the temperature, there is a strong interaction between pH and temperature[8, 9]. The slope of the % dye removal *vs* pH depends on the value of temperature and vice versa.

## CONCLUSION

From the results, it is clear that GLP can be used as an adsorbent to remove CBB from waste water. pH plays a major role and is significant in the adsorption process. The interaction effect between pH and temperature is also shown. It has been found that a low value of pH, high value of the adsorbent dosage and temperature increases the % dye removal.

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