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Adsorptive removal of direct textile dyes by low cost agricultural waste: Application of factorial design analysis

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ABSTRACT

This research work involved the use of factorial experimental design technique to investigate the biosorption of Everdirect Orange-3GL and Direct Blue-67 dyes from aqueous solution on rice husk. A 5^3 full factorial experimental design was employed to study the effect of three factors: initial dye concentration, biosorbent dose and pH at five levels. The efficiency of rice husk for dyes removal has been determined after 3 h of treatment at 30 °C using suitable size of biosorbent (0.255 mm). Main and interaction effects were analyzed by analysis of variance (ANOVA), *F*-test and *P*-values to define most important process variables affecting the dyes biosorption. The statistical model was best fitted with high coefficient of determination (R^2 = 0.999) for Everdirect Orange-3GL and Direct Blue-67 dyes. The most significant variable was thus found to be pH for both the dyes. FT-IR studies showed that hydroxyl functional groups participated in the biosorption of dyes.

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1. Introduction

Synthetic dyes are widely used in textile, paper, leather, food, plastic, cosmetic industries, etc. to color the final products to fulfill the demand of the people [1,2]. Direct dyes have highly conjugated molecular structure containing one or more anionic sulfonate groups which are responsible for the solubilization of dye molecules in water. Benzidine based direct dyes or their metabolites are highly toxic and carcinogenic [3]. Colored dye effluents are highly toxic to ecosystems and aquatic life. It is estimated that 12–14% of textile dyes are discharged into the water stream during dyeing process. The presence of dyes in water inhibits the penetration of light and photosynthesis activity of water plants [4,5]. Large number of diseases such as allergy, skin irritation, dermatitis and cancer are caused due to synthetic dyes [6]. Therefore, treatment of hazardous industrial wastewaters containing dyes is essential for sustainable life and development [7–10].

Wastewater containing dyes is normally treated by physical or chemical treatment processes like flocculation, membrane filtration, electrokinetic coagulation, ion exchange, irradiation, precipitation and ozonation. However, these technologies are generally ineffective in color removal, expensive and less adaptable to a wide range of dye wastewater [11,12].

Biosorption is the cost effective technique for the dyes removal [13]. Researchers have used different biosorbents such as biogas waste slurry [10], tree-fern [14], moss [15], bacteria [16] and coffee wastes [17]. But the search for excellent and efficient biosorbent is still continuing.

Experimental design is an excellent tool for studying the individual and interaction effects of all parameters simultaneously [18,19]. The analysis in which the evaluation of more than one factor can be done is called full factorial analysis. In full factorial multivariate experiment, all main factors and their interactions are compared with one another [20]. Rice is one of the important crops grown in Pakistan. Milling of rice crop produces rice husk as agriculture waste material. It consists of silica, cellulose, hemicellulose and lignin and is available in excess. Initial dye concentration, pH and biosorbent dose are important parameters in biosorption study. This research paper illustrates the use of rice husk as low cost biosorbent for the removal of Everdirect Orange-3GL and Direct Blue-67 textile dyes and the optimization of various parameters influencing the sorption capacity using 5^3 full factorial design.

2. Materials and methods

2.1. Preparation of rice husk sorbent

Fresh rice (*Oryza sativa*) husk was purchased from local rice mills. The biomass was washed several times with distilled water to remove dust and other foreign particles. The cleaned biomass was dried in sunlight for 8 h and then for 24 h at 60 °C in the oven. The dried biomass was ground with food processor (Moulinex, France) and sieved using Octagon sieve (OCT-DIGITAL 4527-01) to various mesh sizes from <0.250, 0.250–0.355, 0.355–0.500, 0.500–0.710

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and 0.710–1.000 mm. Finally the biomass was stored in plastic bottles for further use.

2.2. Preparation of aqueous dye solutions

In the present investigation, two direct textile dyes Everdirect Orange-3GL and Direct Blue-67 (gifted by Sandal Dye Stuff Industry, Faisalabad, Pakistan) were used. Stock solutions of both dyes were prepared by dissolving 1.0 g of each dye in 1000 mL of double distilled water. The experimental solutions of different concentrations ranging from 25 to 150 mg/L for Everdirect Orange-2GL and from 25 to 300 mg/L for Direct Blue-67 were made by further dilutions. Standard curves were developed at λ_{max}, 415 and 570 nm for Everdirect Orange-3GL and Direct Blue-67 dyes, respectively through the measurement of the dye solution absorbance by UV/Visible Spectrophotometer (Hitachi U-2001).

Table 1
Levels of factors for Everdirect Orange-3GL.

Factors	Levels				
	1	2	3	4	5
Dye conc. (mg/L)	25	50	75	100	125
Biosorbent dose (g/L)	0.05	0.07	0.09	0.11	0.13
pH	1	2	3	4	5

this work, initial dye concentration, biosorbent dose and pH were taken as independent variables while the other variables like the particle size (0.255 mm mesh size), temperature (30 °C) and shaking speed (100 rpm) were kept constant. Two replicates of 5^3 full factorial design having 125 experiments (with one replicate) were studied. The three factors and the five levels for Everdirect Orange-3GL and Direct Blue-67 are shown in Tables 1 and 2, respectively.

Following equation was used to explain the design:

$$
\begin{aligned}
Y = {} & X_o+A[1]+A[2]+A[3]+A[4]+B[1]+B[2]+B[3]+B[4]+C[1]+C[2]+C[3]+C[4]+A[1]B[1]+A[2]B[1]+A[3]B[1]+A[4]B[1]+A[1]B[2]+A[2]B[2]+A[3]B[2]+A[4]B[2] \\
& +A[1]B[3]+A[2]B[3]+A[3]B[3]+A[4]B[3]+A[1]B[4]+A[2]B[4]+A[3]B[4]+A[4]B[4]+A[1]C[1]+A[2]C[1]+A[3]C[1]+A[4]C[1]+A[1]C[2]+A[2]C[2]+A[3]C[2] \\
& +A[4]C[2]+A[1]C[3]+A[2]C[3]+A[3]C[3]+A[4]C[3]+A[1]C[4]+A[2]C[4]+A[3]C[4]+A[4]C[4]+B[1]C[1]+B[2]C[1]+B[3]C[1]+B[4]C[1]+B[1]C[2]+B[2]C[2] \\
& +B[3]C[2]+B[4]C[2]+B[1]C[3]+B[2]C[3]+B[3]C[3]+B[4]C[3]+B[1]C[4]+B[2]C[4]+B[3]C[4]+B[4]C[4]+A[1]B[1]C[1]+A[2]B[1]C[1]+A[3]B[1]C[1]+A[4]B[1]C[1] \\
& +A[1]B[2]C[1]+A[2]B[2]C[1]+A[3]B[2]C[1]+A[4]B[2]C[1]+A[1]B[3]C[1]+A[2]B[3]C[1]+A[3]B[3]C[1]+A[4]B[3]C[1]+A[1]B[4]C[1]+A[2]B[4]C[1]+A[3]B[4]C[1] \\
& +A[4]B[4]C[1]+A[1]B[1]C[2]+A[2]B[1]C[2]+A[3]B[1]C[2]+A[4]B[1]C[2]+A[1]B[2]C[2]+A[2]B[2]C[2]+A[3]B[2]C[2]+A[4]B[2]C[2]+A[1]B[3]C[2]+A[2]B[3]C[2] \\
& +A[3]B[3]C[2]+A[4]B[3]C[2]+A[1]B[4]C[2]+A[2]B[4]C[2]+A[3]B[4]C[2]+A[4]B[4]C[2]+A[1]B[1]C[3]+A[2]B[1]C[3]+A[3]B[1]C[3]+A[4]B[1]C[3]+A[1]B[2]C[3] \\
& +A[2]B[2]C[3]+A[3]B[2]C[3]+A[4]B[2]C[3]+A[1]B[3]C[3]+A[2]B[3]C[3]+A[3]B[3]C[3]+A[4]B[3]C[3]+A[1]B[4]C[3]+A[2]B[4]C[3]+A[3]B[4]C[3]+A[4]B[4]C[3] \\
& +A[1]B[1]C[4]+A[2]B[1]C[4]+A[3]B[1]C[4]+A[4]B[1]C[4]+A[1]B[2]C[4]+A[2]B[2]C[4]+A[3]B[2]C[4]+A[4]B[2]C[4]+A[1]B[3]C[4]+A[2]B[3]C[4]+A[3]B[3]C[4] \\
& +A[4]B[3]C[4]+A[1]B[4]C[4]+A[2]B[4]C[4]+A[3]B[4]C[4]+A[4]B[4]C[4]
\end{aligned}
$$

Y is the predicted response (amount of dye sorbed mg/g), X_o is the global mean, A is the initial dye concentration (mg/L), B is the biosorbent dose (g/L) and C is the initial solution pH.

2.3. Batch biosorption studies

The effect of important parameters such as pH, biosorbent dose and initial dye concentration on the biosorption of Everdirect Orange-3GL (C.I. no, Orange-39 and λ_{max}, 415 nm) and Direct Blue-67 (C.I. no, 27925 and λ_{max}, 570 nm) was studied at 30 °C. Biosorption experiments were conducted in 250 mL conical flasks containing 50 mL of dyes solution of known pH, concentration and biosorbent dose. The flasks containing the dyes solution were shaken in orbital shaking incubator (PA250/25H) at 100 rpm and 30 °C for 3 and 4 h, respectively. Blank solutions were run using same conditions except the addition of biosorbent material. To study the effect of pH, various pH's (1–8 for Everdirect Orange-3GL and 3–8 for Direct Blue-67) were adjusted using 0.1 M HCl and NaOH solutions. After specific time, the samples were taken out and centrifugation was performed at 2000 rpm for 20 min. The aliquots were used for the residual concentration of each dye.

The amount of dye biosorbed per unit mass was calculated using the following equation:

$$q = \frac{(C_o - C_e)V}{W} \tag{1}$$

where q is the amount of dye biosorbed on the biosorbent (mg/g), C_o and C_e are the initial and equilibrium concentrations of dye solution, respectively. V is the volume of dye solution (cm^3) and W is the amount of the rice husk (g). The % sorption was measured by the following equation:

$$\%\ \text{sorption} = \frac{C_o - C_e}{C_o} \times 100 \tag{2}$$

All the experiments were conducted in duplicate and results are reported as mean value.

2.4. Full factorial design of experiments

The factorial design describes which factor shows more impact and influence the variation of one factor on the other factors [21]. In

3. Results and discussion

3.1. Analysis of variance (ANOVA)

There are several important problems with the conventional approach of changing one or two variables in a run. It may take several rounds of experiments to find the optimum point. In cases where variables must be changed in large steps, the optimum may not be found at all. An experiment is called a factorial experiment if the treatments consist of all possible combinations of several levels of factors. It reveals the effect of interaction of process variables and improves process optimization. The interaction effects are easily estimated and tested through the usual analysis of variance (ANOVA). ANOVA is a statistical method that partitions the total variation into its component parts each of which is associated with a different source of variation [22].

The ANOVA results for Everdirect Orange-3GL and Direct Blue-67 are shown in Tables 3 and 4, respectively. The sum of the squares used to estimate factors effects and Fisher's F-ratios and P-values are also shown in the respective tables. The results calculated using ANOVA predicted that the main factors such as initial dye concentration, biosorbent dose, pH and the interaction effects werehighly

Table 2
Levels of factors for Direct Blue-67.

Factors	Levels				
	1	2	3	4	5
Dye conc. (mg/L)	25	50	75	100	125
Biosorbent dose (g/L)	0.1	0.2	0.3	0.4	0.5
pH	3	4	5	6	7

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