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Original research article

Adsorptive removal of Direct Red 81 dye from aqueous solution onto *Argemone mexicana*

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A B S T R A C T

A low cost adsorbent has been tested for its adsorption ability to remove a hazardous dye from textile wastewater. The present paper assesses the results pertaining to the investigation conducted on removal of a di azo sulphonated dye, Direct Red 81 by adsorption onto a natural adsorbent, *Argemone mexicana*. Adsorption studies were carried out in both batch and column mode to examine the influence of various parameters on the removal efficiency of the uncharted natural adsorbent. A fixed-bed column has been designed and necessary parameters have been calculated by applying mass transfer kinetic approach. The data obtained have been successfully used to equate different adsorption isotherm models. Isothermal data were found to fit well with Langmuir adsorption model. The estimated mean adsorption energy from the D–R isotherm model has been obtained as 1.34×10^4 J mol^{-1} further conferring to the chemical process of the adsorption. The adsorption interaction of direct dye on to *A. mexicana* obeyed pseudo second-order rate equation. Intraparticle diffusion studies revealed the dye adsorption interaction was complex and intraparticle diffusion was not only the rate limiting step. Column operations depicted good adsorption leading with 98% saturation of dye.

1. Introduction

Tremendous amounts of different synthetic dyes are being formulated globally which are remarkably consumed by color industry [1,2]. Amongst different kinds of dyes, azo dyes accounts for nearly half of the total dyes consumed in the textile industries [3–5]. During the dyeing process, about 10–15% of unfixed dye is lost in water which comes out as a colored effluent from the industries [6]. Hence it is essential to completely remove these toxic dyes from wastewater effluents [7]. Several researchers have embraced the adsorption approach to treat the textile wastewater by means of natural and synthetic adsorbents. Present study aimed at economic removal of Direct Red 81 using a low cost natural adsorbent by adopting adsorption technique. Direct Red 81, a toxic sulphonated azo-based dye, known for its carcinogenic nature and toxicity towards animals and humans is selected as a synthetic model dye solution for experimentation which is widely used in many industries [8–10]. Ingestion of significant amount of this dye results in gastro-intestinal discomfort producing nausea and vomiting. It also has harmful effect on skin and eyes. Presence of double azo linkage along with sulphonic acid group, makes the dye easily soluble in water [11,12]. Earlier researches have been carried out with modified Silk Maze [13], native and citric acid modified bamboo sawdust [14], Chamomilla plant [15] and *Sonchus* fruit plant [16] for removal of Direct Red 81 using these modified agricultural wastes. *Argemone mexicana* seeds utilized in this work belongs to the family of Papaveraceae which is widely available all over the globe. It is commonly known as Mexican prickly poppy. In India it is known as 'Satyanashi' meaning devastating and is phytotoxic to many other crop species [17,18]. The present investigation is aimed at studying the kinetics and adsorption isotherms of the adsorption of Direct Red 81 with an objective to explore optimum conditions for the removal of the dye by adsorption technique using *A. mexicana* as a novel low cost adsorbent. Since very few research have been carried out with unmodified natural material to treat Direct Red 81, present study will unveil the pristine low cost natural adsorbent in the field of textile wastewater treatment.

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2. Experiments

2.1. Materials and methods

Direct Red 81, a sulphonated azo dye, disodium; (3E)-7-benzamido-4-oxo-3-[[4-[(4-sulfonatophenyl)diazenyl]phenyl]hydrazinylidene]naphthalene-2-sulfonate, having molecular formula $C_{29}H_{19}N_5Na_2O_8S_2$ and molecular weight 676 g mol^{-1} was procured from Sigma–Aldrich. The structure is shown in Fig. 1. Maximum absorption wavelength of the dye is 508 nm. Dye stock solution of concentration 10^{-3} M was prepared in distilled water. All solutions were prepared by diluting the stock solution with distilled water.

2.2. Material development

For experimentation, the natural adsorbent opted for this study was collected from Lavale road, near the Symbiosis International University Campus Pune, were it was available in plenty. The seed capsules were separated from the thorny plant carefully and dried in sunlight for about 48 h. Seeds were separated from the capsules and were further dried in a Labline Hot air oven for 4 h at 110 °C. Moisture free seeds were then cleaned and finally grounded in a grinder to get a fine powder. Subsequently the fine powder was sieved by using a sieve set and then was collected in the range of 600–425, 425–300 and 300–125 μm particle size respectively. The particle size of the adsorbent was determined by sieve analysis using Standard test sieves as per IS-460 (Indian standards). Fourier transform infrared spectroscopy (FTIR) analysis was also carried out in order to find out the morphology and characterization.

2.3. Batch adsorption studies

To access the propensity of adsorption process, batch studies were carried out by varying different physiochemical conditions. Batch adsorption studies were conducted by taking 25 mL of dye solution in 100 mL of volumetric flask at 30, 40 and 50 °C using suitable sieve size and definite amount of adsorbent. The test solution was intermittently shaken with a speed of 100 rpm in an orbital shaker BTI-05 for 1 h and then was kept for 24 h for saturation. Thereafter the supernatant liquid was filtered through Whatman filter paper no. 41 (pore size 20 μm) and final equilibrium concentrations of the dye in solution were measured spectrophotometrically at a wavelength of 508 nm, using a Systronic double beam spectrophotometer model no. 2203. Amount of dye adsorbed is calculated as per Eq. (1) below.

$$q_e = \frac{(C_o - C_e)V}{m} \tag{1}$$

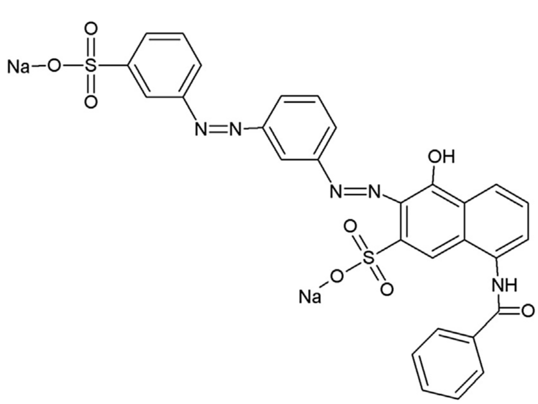

Fig. 1. Structure of Direct Red 81 dye.

The experiment was performed by varying several parameters like pH, dye concentration, contact time, amount of adsorbent, particle size, and temperature.

2.3.1. pH

pH is one of the pivotal factors influencing not only the surface charge (ionization/dissociation) of the adsorbent, but also the solution dye chemistry. The effects of initial pH on Direct Red 81 solution using *A. mexicana* seeds were evaluated within a range between 1 and 10. The pH of the test solutions were adjusted by using HCl and NaOH with an Equiptronics digital pH meter EQ-610.

2.3.2. Amount of adsorbent

In order to study the variation in adsorption of Direct Red 81 on the basis of amount of adsorbents, various amounts ranging from 0.02 to 0.12 g of the adsorbent was taken in 100 mL volumetric flask and experiments were performed at three different temperatures 30, 40 and 50 °C, keeping other parameters constant.

2.3.3. Particle size

Another influential factor during adsorption is size of the particle. In order to study the effect of particle sizes, batch adsorption experiments were conducted at three sieve sizes, i.e., 600, 425 and 300 μm.

2.3.4. Concentration

The effect of concentration on adsorption of Direct Red 81 by *A. mexicana* seeds was investigated keeping other experimental parameters (pH, particle size and adsorbent dosage) constant. After undergoing meticulous experimental procedures, a desired concentration range of 1×10^{-5} to 10×10^{-5} M was selected and fixed amount of adsorbent (0.1 g) was added to these solutions.

2.3.5. Contact time

Contact time study aided in acquiring the optimum time duration for adsorption during the experiment. Fixed amount of adsorbent was taken in single concentration solution for duration of about 360 min and shaken at a speed of 100 rpm at three different temperatures i.e., 30, 40 and 50 °C. The supernatant solution was observed after every 30 min.

2.4. Adsorption isotherms

For optimization of adsorption process, the results obtained from the adsorption of Direct Red 81 dye onto *A. mexicana* seeds were analyzed by well-known isotherm models of Langmuir, Freundlich, Dubinin–Kaganer–Radushkevich (D–R) and Temkin. In the present paper adsorption isotherm models were extended to describe experimental isotherm data and identify the mechanism of the adsorption process.

2.4.1. Langmuir isotherm

According to the Langmuir adsorption isothermal model, maximum adsorption corresponds to the formation of a saturated monolayer of dye molecules on adsorbent surface which stipulates the chemisorption of the process and aids in assessing the adsorption capacity of the adsorbent [19]. Langmuir model, may be expressed as Eq. (2)

$$\frac{1}{q_e} = \frac{1}{Q_o} + \frac{1}{bQ_oC_e} \tag{2}$$

The key feature of Langmuir adsorption isotherm can be exhibited in terms of a dimensionless constant separation parameter (R_L), expressed by the following Eq. (3) [20].