



Decolorization of synthetic dye wastewater using packed bed electro-adsorption column



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ARTICLE INFO

Keywords:

Methylene blue dye
Bael shell
Activated carbon
Activated carbon electrode
Electro-adsorption
Packed bed column

ABSTRACT

The present study reports the electro-adsorption of Methylene blue (MB) dye from synthetic wastewater onto activated carbon in a packed bed electro-adsorption column and the break through data was analyzed for dye adsorption. The influences of bed height, flow rate and initial dye concentration with and without the presence of electrical potential on packed bed adsorption have been investigated and the possibility of regeneration of column was studied. The parameters such as bed capacity, mass transfer zone, bed utilization and adsorbent usage were evaluated from the break through curves. The columns characteristic design parameters have been evaluated using the adsorption models such as Bed Depth Service Time (BDST), Adam-boharts, Thomas and Yoon-Nelson models. The model parameters have been derived using origin software. Out of four adsorption models, Thomas and Yoon-Nelson models were found to be ideal to analyze the column performance with $R^2 \geq 0.80$. From the results, it is observed that electro-adsorption is more efficient than adsorption as the adsorption capacity is 3.18 folds higher and the amount of adsorbent required was much lesser for electro-adsorption. Also the saturation capacity is 0.17 mg/g for electro-adsorption whereas it is 0.069 mg/g for conventional adsorption at optimized condition, which confirms the stability of electro-adsorption column. It has been observed that modifications in electrode played a major role in dye waste water treatment processes.

1. Introduction

Water is essential major resource for all existence on this earth as well as an essential part of the global ecological system. In general, the quality and quantity of water available today is really a major concern all over the world. Specific contaminants leading to water pollution include a wide range of chemicals and microbes [1]. The textile manufacturing process is characterized by the high consumption of resources like water, fuel and a variety of chemicals in a long process sequence that generates a significant amount of waste. The treatment of spent dye wastewater is a growing concern for the textile industry because of aesthetic conditions, as well as eco toxicological issues regarding color and process wastewater impact of that affect the water streams. Different decolorization methods have been followed in literature, which includes physical, chemical and biological processes but all these techniques are not highly effective, produces secondary pollutants and costly too. Mostly, adsorption technology is used for textile dye wastewater treatment; since activated carbon adsorption was most simple and effective process. Normally, the commercial activated carbon has high surface area, good adsorption capacity and porous structure which seems to be the advantageous, but it remains as costly

process. Accordingly, unused agricultural recourses such as green coconut shell, peach stones, bamboo stem waste, areca nut shell, etc. used for manufacture of activated carbon founds to be economical and effective. As regulations become more stringent, the effectiveness and cost of treatment processes becomes more significant [2,3]. The science and technology used for the treatment of dye wastewater to upgrade its quality in order to meet specific water quality standards. Electro-adsorption is a hybrid process that combines elements from adsorption and electrochemistry [4]. Electrochemical principles dictate that the system must contain at least two electrodes that are connected via an external electrical circuit. Both must be in contact with an electrolyte [5–7]. In Ancient period the electro-adsorption work was carried out in 1946 by Grahame for potential controlled adsorption study of n-octanol and also in 1980 porous activated carbon electrode was introduced for desalination study with the help of column having vertical beds of activated carbon between two electrodes, one act as an anode and other act as a cathode. In recent years, a unique attention was given in theoretical and experimental understanding of statics and dynamics of electro-adsorption processes.

Initially, the study was carried out the experiment in batch mode with activated carbon electrode and carbon composite electrode as

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working electrodes and copper as counter electrode respectively for decolorization of Eosin-Y dye, a maximum of 97.12% dye removal was obtained at optimized conditions, subsequently isothermal and kinetic studies were performed to evaluate the maximum adsorption capacity and rate constants. Characterization of adsorbent and activated carbon electrode were done using Scanning electron microscopy, Energy dispersive X-ray spectroscopy, Fourier transform infrared, Thermogravimetric and BET analysis etc. The surface area and pore volume of activated carbon prepared from Bael fruit shell before activation was $87 \text{ m}^2/\text{g}$ and $0.098 \text{ cm}^3/\text{g}$, whereas the surface area and pore volume after H_3PO_4 activation for the same was $321 \text{ m}^2/\text{g}$ and $0.323 \text{ cm}^3/\text{g}$ respectively. In present scenario, electrode materials used in electrochemical processes are moderately expensive; hence low cost electrode material obtains more importance [3,8–13]. In electrodes, both charge and mass transfer limitations will occur. By inserting a carbon stick to centre of the activated carbon electrode increases the conductivity and surface adsorption properties.

Hence, this present study focused on continuous electro-adsorption of dye from wastewater using bael shell activated carbon beads as adsorbent [14]. The effect of process parameters like bed height, initial dye concentration and flow rate under the influence of electrical potential in packed bed adsorption column have been investigated [15,16]. Development of novel carbon electrode with large surface area in the forms of porous circular plates minimized the diffusional limitations and hence electro-adsorption has better dye removal efficiency compared to conventional packed bed adsorption [17–19].

2. Experiment

2.1. Materials

Bael fruit shells utilized in the present study were obtained from Erode District, western part of Tamilnadu in India. The raw materials were cleaned with tap water to remove the impurities, dried at 110°C for 24 h and granulated using a pulverizer and sieved using sieve shaker in the range between 0.25 mm and 0.5 mm and stored in sealed plastic containers to use as adsorbent [11,20–22].

2.2. Preparation of activated carbon

The pulverized bael shell material (adsorbent) was activated chemically using H_2SO_4 , HCl, KOH, NaCl, NaOH and H_3PO_4 . Out of which H_3PO_4 activated adsorbent was selected for experiments due to its microporous and mesoporous structure along with high specific surface area. Later chemically activated adsorbent was heated to 600°C for a period of 1.5 h in muffle furnace, then submerged in 2% NaHCO_3 to remove the residual acid which overlay on the adsorbent and washed several times with distilled water to remove the impurities from the surface of the adsorbent. Then the adsorbent was kept for drying at 110°C for 2 h and kept in desiccator to remove the moisture content if any. The prepared activated adsorbent was stored in the air tight container for use [15,20,23–29].

2.3. Development of activated carbon beads

The activated carbon beads were prepared using a binding material (w/w) poly (vinylidene fluoride) PVDF. To prepare the beads, a known amount of activated carbon prepared from Bael shell was mixed with PVDF in a weight ratio of 9: 1 and the mixture was constantly stirred under warm conditions (50°C) until the PVDF gets completely dissolved. Spherical shape beads of 0.3 mm diameter were prepared by rolling with hands. Subsequently, the beads were soaked in water at ambient conditions for 48 h to evaluate upon the bead adhesion capabilities. The effect of PVDF composition (5–20%) in bead formation was taken into considerations. From the results, it was proven that the beads prepared with 15% PVDF retained their texture even after being

exposed to water for more than 40 h. There by the prepared beads with 15% PVDF were dried in a hot air oven for 6 h at 70°C to remove the moisture. Finally, the beads were packed in airtight polyethylene bags to further use as adsorbent for continuous electro-adsorption studies [14,26,30–33].

2.4. Preparation of synthetic dye wastewater

Methylene blue (MB) dye was procured from Sisco research laboratories pvt.ltd. 1gm of dye was taken in 1000 ml of distilled water to prepare a stock solution of 1000 mg/L.

2.5. Continuous packed bed electro-adsorption column

Column studies were conducted in acrylic column with an internal diameter and length of 2 cm and 20 cm respectively. A porous copper circular plate of 1 mm thickness was placed in between the flanges of top and bottom of column as electrode and distributor for continuous even flow. The gap between the two plates/electrodes was filled with activated carbon beads. An electrical potential of 5 V was applied between the two circular copper plates [2,16,34–38]. A solution of known MB dye concentration was passed through the column using a peristaltic pump (Model: PP-20-EX – Miclins, India) in an up-flow mode to avoid channeling and clogging. The schematic diagram of continuous packed bed electro-adsorption was shown in Fig.10. The effluent samples were collected at specific intervals and analyzed for the MB concentration using spectrophotometer in the visible region at 664 nm.

2.6. Effect of operating parameters

The electro-adsorption of MB wastewater onto the bael shell activated carbon beads was investigated at different operating parameters as follows.

2.7. Effect of bed height

The breakthrough curves of the MB dye adsorption were obtained at 3, 6 and 9 cm bed heights by maintaining the flow rate, MB concentration and potential constant at 3 mL/min, 5 mg/L and 5 V potential respectively.

2.8. Effect of flow rate

Investigation over the effect of influent flow rate on the removal of MB dye was carried out at three different flow rates 3, 6 and 9 mL/min. For each test, the bed height, influent MB dye concentration and potential were held constant at 6 cm, 5 mg/L and 5 V respectively.

2.9. Effect of initial dye concentration

The adsorption performance of bael shell activated carbon beads was analyzed by varying the inlet concentration of MB dye solution range from 5 to 15 mg/L at a constant bed height of 6 cm, flow rate of 3 mL/min and potential of 5 V.

2.10. Data analysis of electro-adsorption column

The electro-adsorption column of adsorbate till the breakthrough point and exhaustion point were calculated using the following equation:

$$Q_{total} = \frac{Q}{1000} \int_{t=0}^{t_{total}} C_{ad} dt \quad (1)$$

$$M_{total} = \frac{C_0 Q_{total}}{1000} \quad (2)$$

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