



Adsorption of Eriochrome black-T azo Dye from Aqueous solution on Low cost Activated Carbon prepared from *Tridax procumbens*

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Available online at: www.isca.in, www.isca.me

Received 31st January 2015, revised 10th March 2015, accepted 16th March 2015

Abstract

*In the current study, low cost activated carbon was prepared from the stem and flowers of widespread weed *Tridax procumbens*. Chemical activation method was used to prepare activated carbon; sulphuric acid was used as activating agent. The surface morphology was studied by field emission scanning electron microscopy. The variations in the functional groups were identified by means of Fourier transform Infrared analysis. Adsorption of environmentally hazardous anionic Eriochrome Black-T (EBT) azo dye onto the activated carbon was studied with various parameters such as effect of contact time, dosage, pH, initial dye concentration. From the result it reveals that the activated carbon prepared from *Tridax procumbens* is an excellent adsorbent for the azo dyes at lower pH.*

Keywords: *Tridax procumbens*, activated carbon, adsorption, Eriochrome Black-T, morphology.

Introduction

World is facing serious problem of waste water from textile industries. Textile industries contain various kinds of manmade dyes which are considered as chief sources of environmental pollution in terms of both the volume of dye discharged and the sewage composition¹. In the present days, people find simple way to prepare new carbonaceous materials which can be extensively applicable in different fields in order to overcome energy shortage, environmental crisis and developing customer demands².

Currently, low cost, non-conventional adsorbents like bottom ash, deoiled soya, hen feathers, bagasse, coir pith, wool, orange and banana peels, rice husk and neem saw dust have attracted greatly. To remove the colour from waste water adsorbents play a very important role. Good adsorption capacity can be observed by activated carbon having appropriate pore size distribution. Therefore, efficient tool to remove dyes from textile effluents is adsorption by low cost adsorbents which is simple and cost effective³. Due to wonderful structure and easy availability carbonaceous materials like activated carbons with high surface area and porosity have been applied in different industries for several decades⁴.

The problems like chemical oxygen demand (COD) by the water body, and an increase in toxicity causes mainly due to discharge of colored waste water without proper treatment. Trough out the world annually there are more than thousand varieties of commercial dyes and pigments exist and over 735 tones of synthetic dyes are produced. It is calculated that about 10–15% of the dyes are lost in the effluent during the dyeing

processes. Lowering light penetration, photosynthesis and damage to aesthetic nature of the water surface are the chief problems associated with colored effluent⁵.

Majority of the dyes are toxic, mutagenic and carcinogenic in nature besides they are very stable to light, temperature and microbial attack, making them intractable compounds. From an environmental point of view, the removal of synthetic dyes is of great concern. Among several chemical and physical methods, adsorption process is the effective method that has been successfully employed for color removal of synthetic dyes from effluent. Many adsorbents have been tested to decrease dye concentrations from aqueous solutions. Activated carbon is regarded as an effective adsorbent⁶.

Tridax procumbens, a novel medicinal plant material used as low cost adsorbent for successive removal of Cu, cadmium(II), nitrite from synthetic industrial waste water⁷⁻⁹. *Tridax procumbens* (asteraceae), an herb found throughout India is employed as indigenous medicine for a large number of ailments including jaundice. It is commonly used in Indian traditional medicine as anticoagulant, antifungal and in dysentery. *Tridax procumbens* is known for its wound healing activities. Whole plants is made into paste and applied on fresh cuts. It is used for the treatment of asthma, ulcer, piles, and urinary problems^{10,11}. Conventionally it is used for the treatment of various diseases like bronchial catarrh, dysentery, malaria, stomachache, diarrhoea, high blood pressure etc, and also to check haemorrhage from cuts, bruises, wounds and to prevent falling of hair. It possesses antiseptic, insecticidal, parasiticidal and hepatoprotective properties and has marked depressant action on respiration¹².

Tridax procumbens possesses a wide variety of medicinal properties, but there is no satisfactory literature relating to preparation and adsorption property against environmentally hazardous organic azo dyes. This creates our interest to prepare and study the adsorption activities using *Tridax procumbens* activated carbon. In this article we present the preparation of low cost activated carbon from the stem and flowers of *Tridax procumbens* and studied its adsorption behavior by varying different parameters.

Material and Methods

Commercially pure sulphuric acid (0.1N H₂SO₄ AR 99 %, Merck), sodium hydroxide (0.1 N NaOH AR 99 %, Fisher Scientific), Hydrochloric acid (0.1 N HCl AR 99 %, Fisher Scientific), EBT azo dye (AR 99 %, Fisher Scientific), All glassware used in the present study was Pyrex quality manufactured by Borosil works limited. Water used in all experiments was double distilled water.

Preparation of low cost activated carbon: The plant *Tridax procumbens* was collected at places in and around Rajanukunte, Bengaluru. Stem and flower parts of the plant were broken down into small pieces, dried in daylight until the water content evaporated. Further it is dried in hot air oven at 50°C for 12 hours. The dried materials were used for the preparation of activated carbons using chemical activation method.

Chemical activation method is used for the preparation of activated carbon^{13,14}. In this method the dried materials are treated with excess of sulphuric acid. Charring of the materials occurred immediately with evolution of fumes and heats. After the reaction completed, the materials were left in air oven at 140-160°C for a period of 24 hours. The dried masses were washed with excess of distilled water to remove the free acid residues. They were dried at 110°C, and finally activated at 600°C.

An accurately weighed amount of the EBT dye was dissolved in de-ionized water to prepare stock solution (10 mg/l). Further

experimental solutions of preferred concentration were obtained by successive dilutions.

Characterization techniques: Prepared product was characterized by FE-SEM performed on a ZEISS scanning electron microscope. The FT-IR spectrum was recorded on a Perkin Elmer Spectrometer (Spectrum 1000) with KBr pellet method in the range of 400-4000 cm⁻¹. UV-Vis spectrum was recorded using Elico SL-210 UV-Vis spectrophotometer. Kemi centrifuge was used to separate dye solution from adsorbent.

Results and Discussion

FT-IR spectroscopic studies: The FT-IR spectrum of activated carbon is shown in figure-2. The broad band at 3400 cm⁻¹ is a characteristic of the stretching vibration of hydrogen bonded hydroxyl groups of the activated carbon. The band at 2345 cm⁻¹ refers to the presence of an aliphatic -CH stretching. The spectrum shows a pronounced band at 1590 cm⁻¹, that can be assigned to the C=C stretching vibration in the structure of the activated carbon. The band at 1100-1300 cm⁻¹ is usually found with oxidized carbons and has been assigned to C-O stretching in acid¹⁵. The FT-IR spectroscopy result indicates that the prepared activated carbon is rich in surface functional groups. Peaks in the region of wave numbers lower than 800 cm⁻¹ could be attributed to N-containing bioligands.

UV-Vis spectroscopy studies: In order to determine the optical property, the UV-Vis absorption spectrum was recorded. Figure-3 shows the UV-Vis absorption spectrum. It shows a strong absorption peak (λ_{max}) at 218 nm at the UV region. This can be ascribed to photo excitation of electrons from the valence band to the conduction band.

Morphological analysis: The morphology of prepared activated carbon was investigated using field emission scanning electron microscopy. Figure-4(a-c) shows FE-SEM image of activated carbon at different magnifications. It revealed that the morphology of the sample is a flake like and has micro pores, heterogeneous structure¹⁶.



Figure-1(a)
Tridax procumbens plant

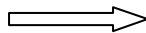


Figure-1(b)
Activated carbon prepared from Tridax
procumbens Preparation of EBT azo dye solution

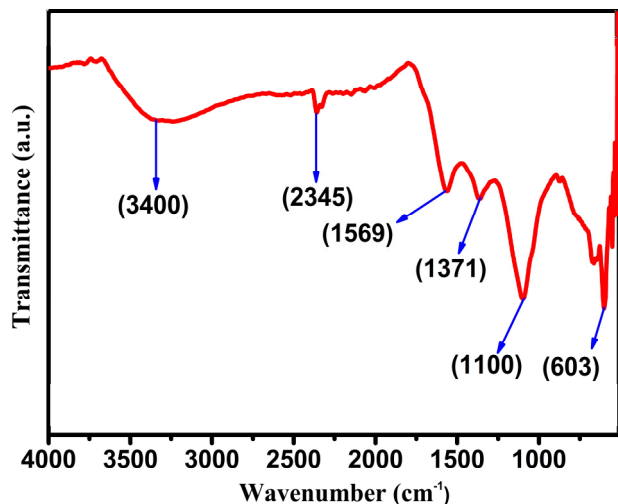


Figure-2
FT-IR spectrum of activated carbon

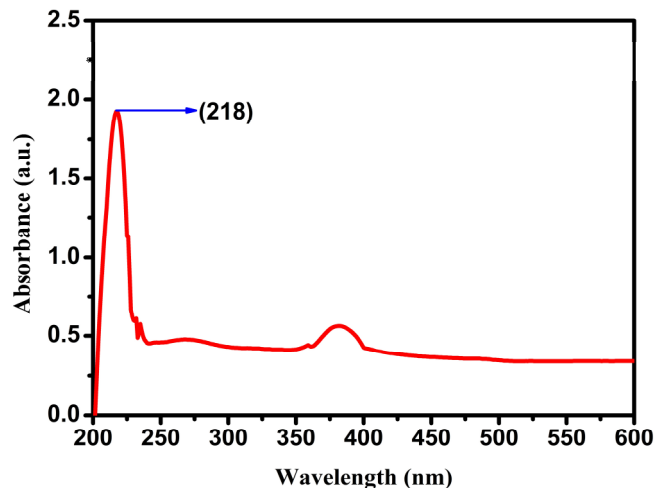


Figure-3
UV-Vis spectrum of activated carbon

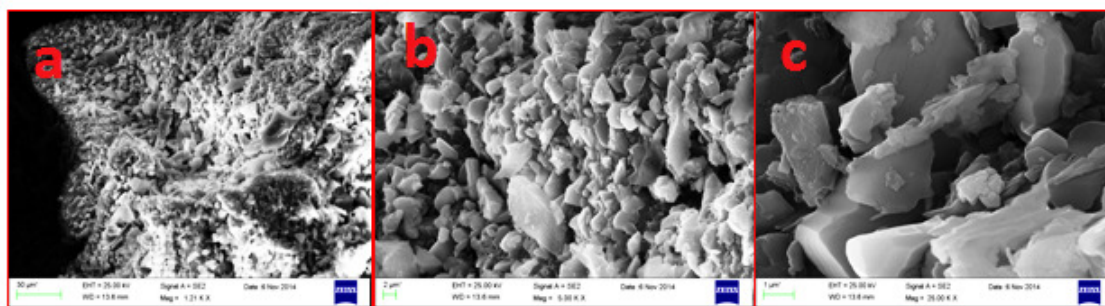


Figure-4
(a-c) FE-SEM images of activated carbon at different magnifications

Adsorption studies: Adsorption experiments were performed using organic hazardous anionic EBT dye. EBT is an azo dye with a molecular weight 461.38. IUPAC name is 1-[1-hydroxy-2-naphthol azo]-6-nitro -2-Naphthol- 4-sulphonic acid sodium salt with molecular formula $C_{20}H_{12}N_3NaO_7S$. EBT has a high solubility in acidic organic solvents but less in water. Chemical structure EBT dye is shown in figure-5.

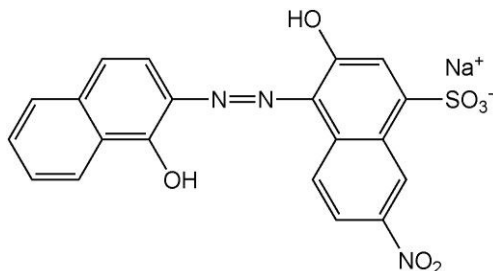


Figure-5
Chemical structure EBT dye

Effect of contact time: Effect of contact time on the adsorption of EBT onto activated carbon was studied. It can be observed from the Figure-6 that the adsorption increases with the

increasing of stirring time up to 90 min. The rate of adsorption is initially quite rapid with most of the dye being adsorbed within the first 90 min. It was found that more than 70 % adsorption of dye occurred in the first 90 min; thereafter the rate of adsorption was found to be slow. This shows that equilibrium can be assumed to be achieved after 90 min. It is mainly due to saturation of the active sites which does not permit further adsorption to take place¹⁷.

Effect of adsorbent dose: Adsorption is strongly influenced by the dose of the adsorbent. Adsorption of EBT onto activated carbon was studied with changing the amount of adsorbent from 10 mg to 90 mg/L at a constant stirring rate of 90 minutes with optimum dye concentration of 10 ppm. It is observed from the Figure-7 that with increase in the dose, increases adsorption up to optimum quantity of adsorbent. Maximum of 75 % dye adsorbed at the dose of 60 mg of adsorbent. Further increase in adsorbent dose decreases the adsorption percentage. This is due to the over-lapping of adsorption sites resulting in decrease in total surface area available to EBT¹⁸.

Effect of pH: pH has an immense effect on the adsorption efficiency of organic azo dyes. Effect pH on EBT adsorption

onto the activated carbon was carried out at 10 ppm of initial dye concentration with 60 mg mass of adsorbent at 90 min of stirring rate at lab temperature. As given in the figure-8, activated carbon shows maximum of 78 % adsorption at the pH of 4 which decreased to 35 % at pH of 9. This confirms that the low pH (7–9) was unfavourable for EBT adsorption by activated carbon. This is because, as initial pH of dye solution increased the number of positively charged adsorbent sites decreased and negatively charged sites increased which did not favour the adsorption since the EBT is anionic dye results in electrostatic repulsion^{19,20}.

carbon was found to be less (35 %). This experimental result clearly explains that availability of dye molecules to interact with the adsorbent should be in the optimum range. Figure-9 shows the effect of initial concentration of the dye.

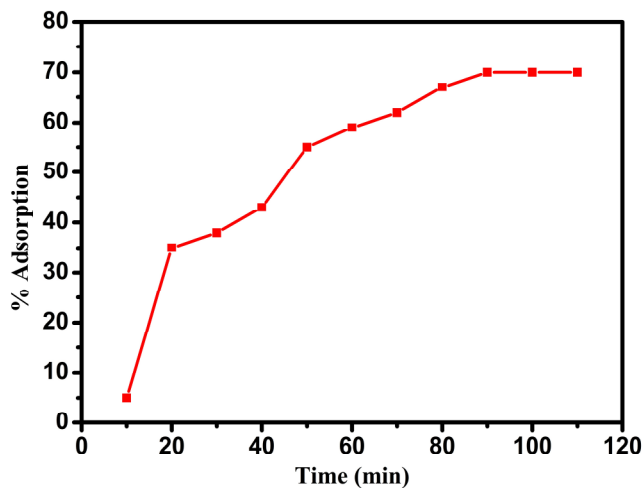


Figure-6
Effect of contact time

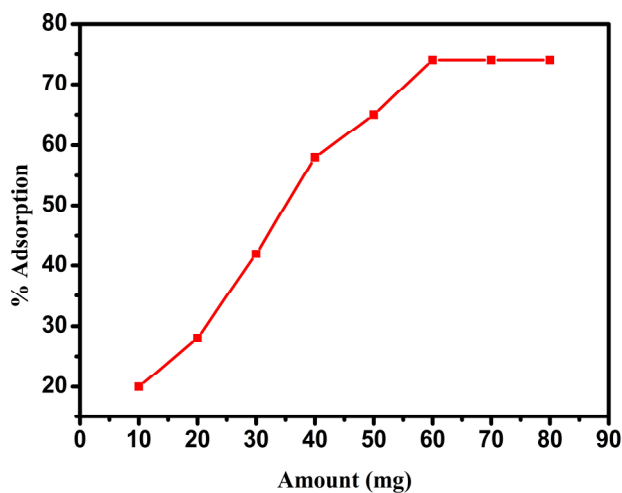


Figure-7
Effect of dosage

Initial concentration effect: It is very exciting to note that the adsorption percentage for 5 ppm dye solution was very low (25 %) since the availability of dye molecules to the adsorbent was poor. With increasing the concentration of dye to the 10 ppm, percentage of adsorption increases to 75 %, further for 15 ppm of EBT concentration, percentage of adsorption onto activated

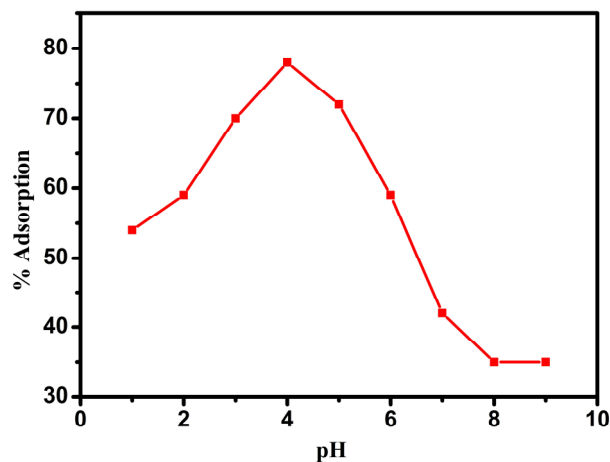


Figure-8
Effect of pH

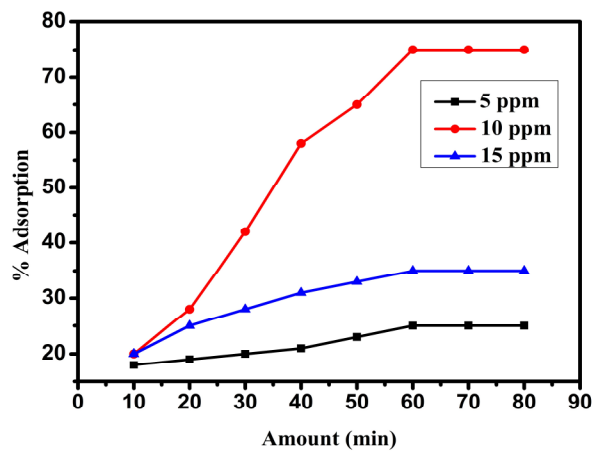


Figure-9
Effect of Initial concentration of dye

Conclusion

In the current study, activated carbon was prepared from *Tridax procumbens* by chemical activation method using H_2SO_4 . The various characterization methods used in this study show that physical and chemical nature of activated carbon. No impurity peaks were observed other than carbon in the FTIR spectrum. Flake like morphology of the activated carbon helps to better adsorption of dye. Since the EBT is anionic dye maximum adsorption occurs at lower pH.

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