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Recent developments in preparation of activated carbons by microwave: Study of residual errors



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Abstract The application of microwave activated *Acacia nilotica* leaves (MVM) and chemically activated *A. nilotica* leaves (CVM) for the removal of crystal violet (CV) from aqueous solution was studied in a batch system. Equilibrium sorption isotherms and kinetics were investigated. The applicability of the Langmuir model for CV onto CVM and MVM was proved by the high correlation coefficient and non-linear regression such as, residual root mean square error (RMSE), chi-square (χ^2), sum of the square of the errors (ERRSQ), composite functional error (HYBRD), derivative of Marquardt's percent standard deviation (MPSD), average relative error (ARE), sum of absolute error (EABS) and average percentage error (APE). The adsorption of CV onto CVM and MVM follows pseudo-second-order kinetic model with intraparticle diffusion is one of the rate-limiting steps. The adsorption capacity of MVM is greater than CVM. The preparation of MVM is agreeing with the principles of green chemistry. Therefore, the eco friendly adsorbent MVM is expected to be the environmentally and the economically feasible adsorbent for the removal of CV from aqueous solution.

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1. Introduction

The textile industries are the greatest generators of liquid effluent due to the high quantity of water used in the dyeing process (Selen et al., 2008). Dyes used in textile industries may be toxic

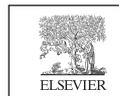
to aquatic organisms and can be resistant to natural biological degradation. Hence, the removal of color synthetic organic dye-stuff from waste effluents becomes environmentally important (Hameed, 2009). The valorization of agricultural wastes into valuable materials without generating pollutants is a big challenge and recommended for an industrial sustainable development in order to preserve the environment (Reffas et al., 2010).

Adsorption has been shown to be one of the most promising and extensively used methods for the removal of both inorganic and organic pollutants from contaminated water. Adsorption onto activated carbon is proven to be very effective in treating textile wastes. However, in view of the high cost and associated problems of regeneration, there is a constant search for alternate low-cost adsorbents (Madhavakrishnan

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et al., 2009). But those adsorbents were chemically activated and sometimes it may cause other environmental hazards.

Acacia nilotica is a species of *Acacia*, native to Africa and the Indian subcontinent. In Haryana, *A. nilotica* based agro forestry systems reduced the yield of wheat (Puri et al., 1995). The aim of the paper is to find out the more suitability and applicability of carbon prepared by different activation (microwave and chemical) of *A. nilotica* to uptake cationic dye (crystal violet) from simulated waste water. The microwave activation method was used because of its one-step simple process and the approach toward green chemistry principle.

Discharge of CV into the hydrosphere can cause environmental degradation, because CV is readily absorbed into fish tissue from water exposure and is reduced metabolically by fish to the leuco moiety, leucocrystal violet (LCV). Several studies by the National Toxicology Program reported that the carcinogenic and mutagenic effects of crystal violet in rodents. It has also been linked to increased risk of human bladder cancer. The leuco form induces renal, hepatic and lung tumor in mice.

There are two main objectives of this study: the first is to prepare an eco friendly microwave activated carbon based on *A. nilotica* leaves as an agricultural waste for the depollution of water effluents contaminated by dyes from textile industry; the second is to compare the adsorption properties of microwave activated carbon with chemically activated carbon based on *A. nilotica* leaves. Desorption was used to elucidate the nature of adsorption and to design the mechanism for recycling of the spent adsorbent.

2. Materials and methods

2.1. Preparation of adsorbent and adsorbate

The leaves of *A. nilotica* were dried in an oven and treated with conc. H_2SO_4 for 12 h and washed thoroughly with distilled water till neutral pH is attained and soaked in 2% $NaHCO_3$ overnight in order to remove any excess of acid present. Then the material (CVM) was washed with distilled water and dried. The raw sample is placed in a microwave oven (Samsung; Triple Distribution System) at 800 W for 2 min. The carbonized sample (MVM) was collected. Both CVM and MVM were preserved in an air tight container for further studies.

The commercial grade crystal violet (color index No. 42555) with molecular formula $C_{25}H_{30}ClN_3$, molecular weight 407.99 and λ_{max} 584 nm are obtained from Thomas baker (chemicals) Ltd., Mumbai, India (Fig. 1). All the chemicals used throughout this study were analytical-grade reagents and the adsorption experiments were carried out at room temperature ($27 \pm 2^\circ C$).

2.2. Characterization of CVM and MVM

The surface morphology and fundamental physical properties of the sorbents were obtained by the scanning electron microscope (LEO 435 VP model) and Fourier Transform Infrared (FTIR) analysis. Determination of zero point charge (pH_{zpc}) was done to investigate the surface charge of both chemically and microwave activated adsorbents for different solution pH.

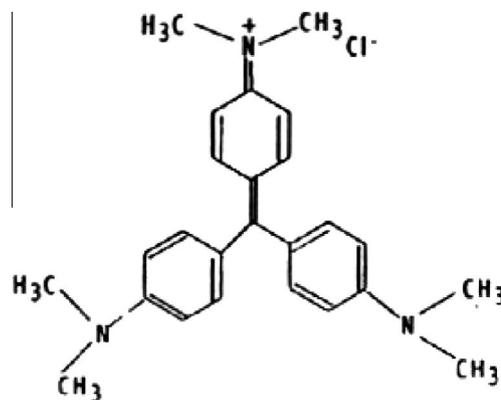


Figure 1 Structure of crystal violet dye.

2.3. Batch adsorption studies

The adsorption experiments were carried out in a batch process to evaluate the effect of pH, contact time, adsorbent dose, adsorption kinetics, adsorption isotherm and desorption of CV onto CVM and MVM.

2.3.1. Dye uptake experiments

For each experiment, a series of flasks were prepared with 50 mL of dye solution (25–200 mg/L, respectively) and the pH was adjusted from 2 to 8 using a pH meter (Deluxe pH meter, model-101 E). About 0.2 g of the sorbent was added, and the flasks were agitated at 160 rpm. The sorbent was removed by centrifugation and the supernatant was analyzed using a Systronic Spectrophotometer-104 at wavelength of 584 nm. Desorption studies were carried out by filtering the dye loaded adsorbent and the percentage of desorption was analyzed.

The amount of dye adsorbed at equilibrium onto carbon, q_e ($mg\ g^{-1}$) was calculated by the following mass balance relationship:

$$q_e = (C_0 - C_e)V/W \quad (1)$$

where C_0 and C_e are the concentrations ($mg\ L^{-1}$) of CV at initial and equilibrium, respectively. V is the volume (L) of the solution and W is the weight (g) of the adsorbent used.

2.3.2. Determining isotherm parameters by non-linear regression

Due to the inherent bias resulting from linearization, alternative isotherm parameter sets were determined by non-linear regression. This provides a mathematically rigorous method for determining isotherm parameters using the original form of the isotherm equation (Khan et al., 1996; Malek and Farooq, 1996; Seidel and Gelbin, 1988; Seidel-Morgenstern and Guichon, 1993). Non-linear analysis of isotherm data is an interesting mathematical approach for describing adsorption isotherms at constant temperature for water and waste water treatment applications and to predict the overall sorption behavior under different operating conditions (Hadi et al., in press). Indeed, as different forms of the equation affected R^2 values more significantly during the linear analysis, the non-linear analysis might be a method of avoiding such errors (Ncibi, 2008). Most commonly, algorithm based on the Levenberg–Marquardt or Gauss–Newton methods are used (Ho, 2004).

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