MATERIALS AND METHODS

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3.1 Preparation of the adsorbent

The pods of White lead tree (Leucaene leucocephala) were collected from Palakkad district, Kerala and these pods were cut into small pieces, dried in sunlight for 5 to 8 days and further dried in hot air oven at 60^0C for 24 hours. The completely dried material was powdered well. The powdered raw material was chemically activated by treating it with concentrated Sulphuric acid with constant stirring and kept for 24 hours. The carbonized material thus obtained was washed well with plenty of water for several times to remove the excess of acid present and then dried at 100°C-120°C in a hot air oven for 24 hours. The adsorbent thus obtained was ground well and sieved through a 250 mesh and kept in an air tight container for further use.

3.2 Description of the adsorbent

COMMON NAME	**: White lead tree**
BOTANICAL NAME	**: Leucaene leucocephala**
FAMILY	**: Fabaceae**
SUBFAMILY	**: Mimosoideae**

A low- cost and eco- friendly activated carbon was prepared from the pods of White lead tree and used in this study for the removal of Crystal Violet dye from an aqueous solution and also from a dyeing industrial effluent containing Crystal Violet dye.

Leucaene leucocephala

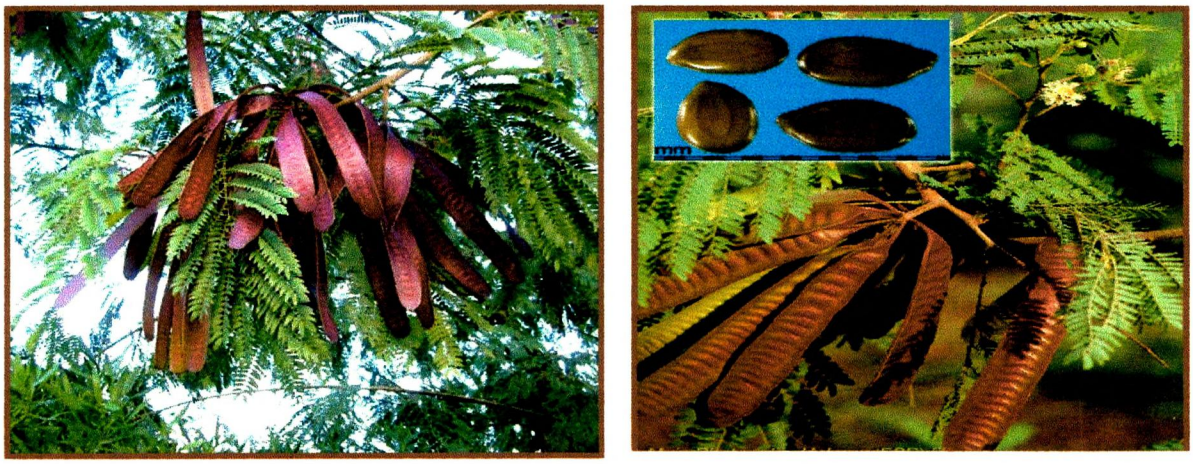

Figure- 3

3.3 Reagents

The Crystal Violet dye solution was prepared by dissolving 5g of Crystal Violet dye in distilled water and diluted to 1000ml.This prepared solution was taken as stock solution which was then diluted to appropriate concentrations.

Crystal Violet dye

Figure- 4

3.4 Equipments

- Elico pH meter was used to measure pH
- Photo Colorimeter (Model 1311) was used for Spectro Colorimetric work
- Orbital Shaker and Incubator Orbital Shaker were used for shaking of the solution containing adsorbent and adsorbate.

pH meter

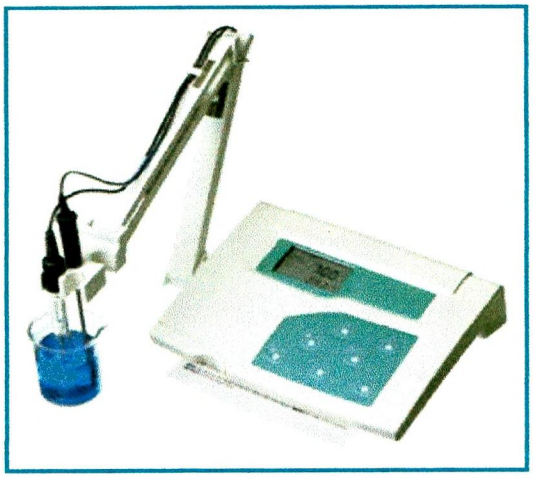

Figure – 5

Photo Colorimeter

Figure - 6

Orbital Shaker

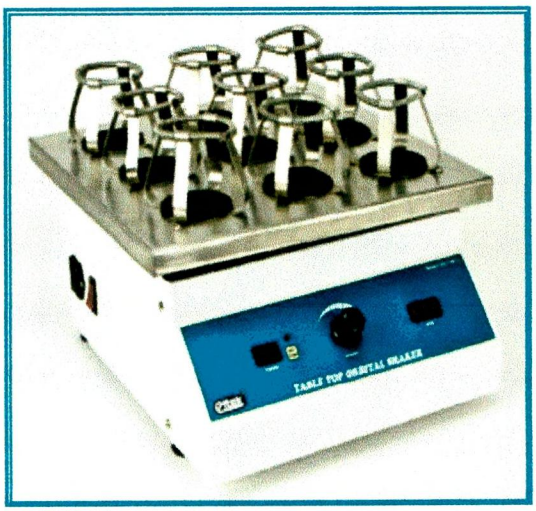

Figure- 7

Incubator Orbital Shaker

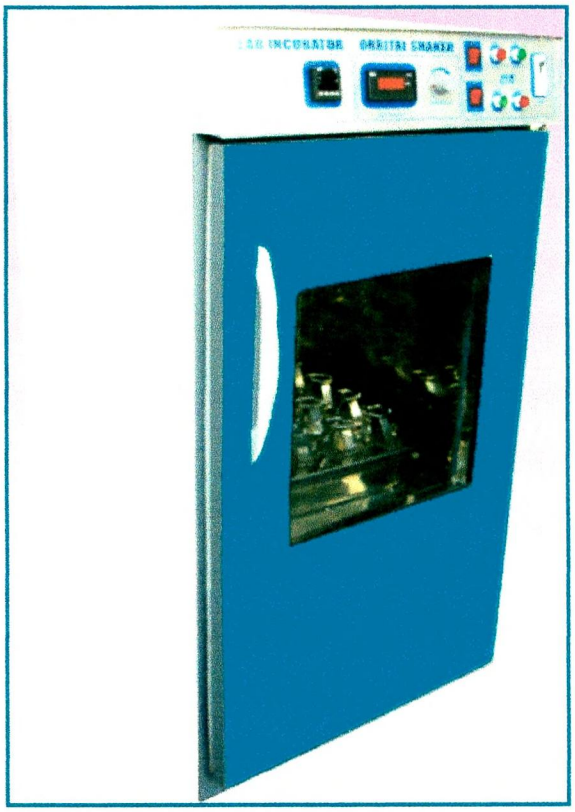

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Figure- 8

3.5 BATCH EXPERIMENTS

The present investigation have been carried out to compare the efficiency of a low-cost and eco-friendly adsorbent prepared from the pods of Leucaene leucocephala with that of Commerical Activated Carbon for the removal of a basic dye, Crystal Violet by adsorption technique. Batch adsorption studies using two adsorbents namely Activated Leucaene leucocephala adsorbent (ALL) and Commerical Activated Carbon adsorbent (CAC) were carried out by varying the initial concentration of dye solution, initial pH, adsorbent dosage and temperature. The industrial effluent containing the Crystal Violet dye was collected from a dyeing industry (Kanjikode, Kerala) and its removal by adsorption also studied in this work.

3.5.1 Effect of variation of initial concentration of the dye on the adsorption

Batch experiments for the adsorption of Crystal Violet dye with both the adsorbents (ALL and CAC) used in this study were conducted by varying the initial concentration of Crystal Violet dye solution.100 ml of dye solution containing 15, 20, 25 and 30 mg of dye were prepared from the stock solution and taken in Pyrex bottles containing 200 mg of the adsorbent ALL. Similarly 100 ml of the dye solution containing 15, 20, 25 and 30 mg of dye were prepared and treated with 50 mg of the adsorbent CAC. The Pyrex bottles containing the dye solutions and the adsorbents were agitated at 150 rpm (rotation per minute) speed on a Orbital Shaker at 32^0 C for various time intervals (10, 20, 30, 40, 50, 60, 90, 120, 150 and 180 minutes) . These solutions were filtered and the dye concentration of the filtrates were estimated colorimetrically at 590 nm using Photo Colorimeter.

3.5.2 Effect of variation of pH on the adsorption of dye

Batch experiments were performed by varying the initial pH from 5 to 9 (5, 6, 7, 8, and 9) using 0.1 N HCl or 0.1 N NaOH solution. pH measurements were carried out using Elico pH meter. 100 ml of the dye solutions containing 30 mg of the Crystal Violet dye were prepared from the stock solution and treated with 200 mg of the adsorbent ALL taken in Pyrex bottles. Similarly 100 ml of the dye solutions containing 30 mg of the dye were prepared and treated with 50 mg of the adsorbent CAC. The pH of the above

solutions was adjusted to a desired pH from 5 to 9 and batch studies were conducted. These solutions were shaken using Orbital Shaker at 32^0 C for various time intervals (10, 20, 30, 40, 50, 60, 90, 120, 150 and 180 minutes).These solutions were filtered and the filtrates obtained were analyzed colorimetrically to find out the percentage amount of dye adsorbed.

3.5.3 Effect of variation of adsorbent dosage on the adsorption of dye

The effect of adsorbent dosage on the adsorption of Crystal Violet dye was studied at pH 9.0 ± 0.02.100ml of the dye solutions containing 30 mg of the Crystal Violet dye were taken in Pyrex bottles and batch adsorption studies were conducted by varying the adsorbent dosage (200, 300, 400 to 500 mg) of the adsorbent ALL. Similarly 100ml of the dye solutions containing 30 mg of the Crystal Violet dye were taken in Pyrex bottles and treated with 50, 75, 100 and 125 mg of the adsorbent CAC. The Pyrex bottles containing adsorbent and adsorbate were shaken in a Orbital Shaker at 32^0 C for various time intervals (10, 20, 30, 40, 50, 60, 90, 120, 150 and 180 minutes).These solutions were filtered and the filtrates were analyzed colorimetrically using Photo Colorimeter to find the percentage amount of dye adsorbed.

3.5.4 Effect of variation of temperature on the adsorption of dye

The effect of temperature on the adsorption process was studied at 295, 305 and 315 K. Dye solutions containing 30 mg of the Crystal Violet dye were taken in Pyrex bottles containing 200 mg of the adsorbent ALL. Similarly 100ml of the dye solutions containing 30 mg of the Crystal Violet dye were treated with 50 mg of the adsorbent CAC and the adsorption studies were conducted at pH 9.0 ± 0.02. The Pyrex bottles containing the adsorbent and the adsorbate were taken in an Incubator Orbital Shaker and agitated for various time intervals (10, 20, 30, 40, 50, 60, 90, 120, 150 and 180 minutes).These solutions were filtered and the filtrates were analyzed colorimetrically using Photo Colorimeter to find the percentage amount of dye adsorbed.

3.5.5 Effect of variation of initial concentration of the dye on the adsorption (obtained from a dyeing industrial effluent) with the adsorbents ALL and CAC

Batch adsorption studies of Crystal Violet dye obtained from a dyeing industrial effluent were performed with 600 mg of the adsorbent ALL by varying the initial concentration of Crystal Violet dye solution (100, 150 and 200 mg/L). Similar experiments were conducted with 150 mg of the adsorbent CAC by varying the initial concentration of Crystal Violet dye solution (100, 150 and 200 mg/L) obtained from a dyeing industrial effluent. These solutions were taken in the Pyrex bottles and shaken in an Orbital Shaker, agitated for various time intervals (10, 20, 30, 40, 50, 60, 90, 120, 150 and 180 minutes). The adsorbate and the adsorbents were separated and analyzed colorimetrically.

3.6 KINETICS AND ADSORPTION ISOTHERM STUDIES FOR CRYSTAL VIOLET DYE REMOVAL

The results of the above mentioned studies were used to determine the rate constant for the adsorption of Crystal Violet dye using Lagergren rate equation, Elovich rate equation and Intraparticle Diffusion rate equation. Langmuir and Freundlich adsorption isotherms were also evaluated for Crystal Violet dye adsorption with both the adsorbents ALL and CAC.

3.7 SCANNING ELECTRON MICROSCOPE (SEM) ANALYSIS

The Scanning Electron Microscope (SEM) is a type of electron microscope that produces images of a sample by scanning it with a focused beam of electrons. The electrons interact with electrons in the sample, producing various signals that can be detected and contain information about the sample's surface topography and composition. The electron beam is generally scanned in a raster scan pattern, and the beam's position is combined with the detected signal to produce an image. SEM can achieve resolution better than 1 nanometer. Specimens can be observed in high vacuum, low vacuum and in environmental. SEM specimens can be observed in wet conditions also **(http://en.wikipedia.org/wiki/Scanning_electron_microscope).**

Schematic diagram of a SEM

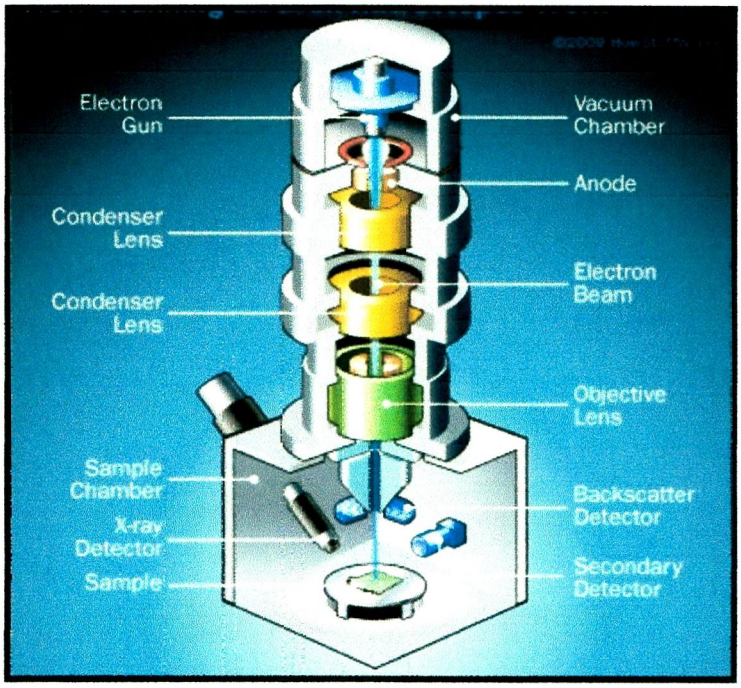

Figure- 9

The SEM photographs of the following were taken in this study:

- Adsorbent ALL prepared from the pods of Leucaene leucocephala.
- Commercial Activated Carbon adsorbent (CAC).
- Adsorbent ALL with the adsorbed Crystal Violet dye species from an aqueous solution.
- Adsorbent CAC with the adsorbed Crystal Violet dye species from an aqueous solution.
- Adsorbent ALL with the adsorbed Crystal Violet dye species from a dyeing industrial effluent.
- Adsorbent CAC with the adsorbed Crystal Violet dye species from a dyeing industrial effluent.