3

MATERIAL AND METHODS

The low cost activated carbon adsorbents used in this research study was prepared from the fruit pods of *Bauhenia racemosa* by treatment with Conc. Sulphuric acid / Phosphoric acid . The *Bauhenia racemosa* belongs to the family of Caesalpiniaceae and is abundantly found in Tamil Nadu, Kerala and Karnataka. *Bauhenia racemosa* is a road side shade tree, available plenty in number in Udumalpet and Pollachi areas of Tamil Nadu **(Figure 1).** The fruit pods were collected and converted into activated carbon by treatment with Conc. Sulphuric acid / Phosphoric acid.

Scientific Name : *Bauhenia racemosa*
Family : Fabaceae
Common Name : Apta Tamil name : ஆத்தி

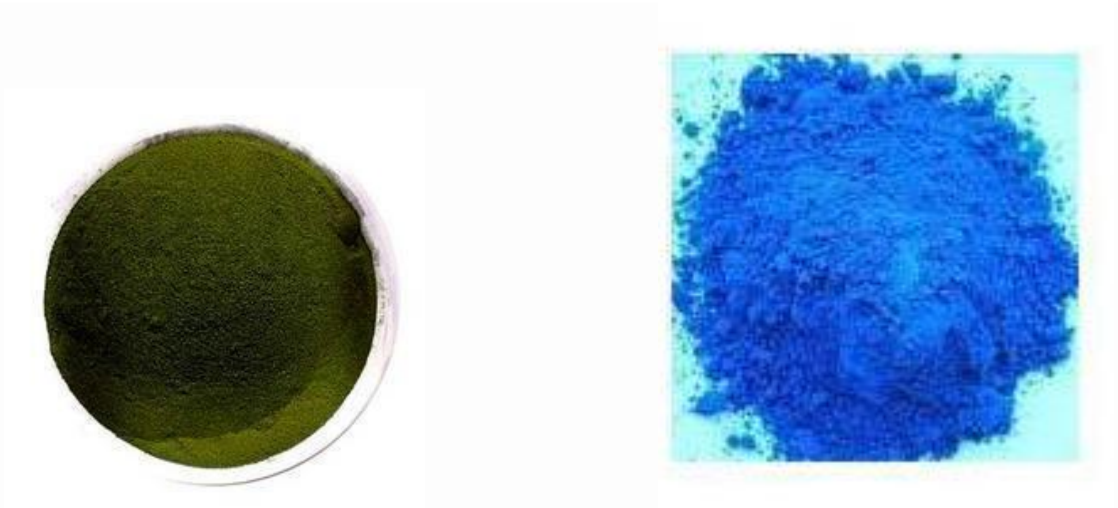
Description

Sub family : Caesalpiniaceae
Propagation : Seeds

Bauhenia racemosa is a medium size tree, native of India which is extremely drought resistant and has religious significance for Hindus.

This plant was authenticated by Botanical Survey of India, Southern Regional Centre, Tamilnadu Agricultural University, Coimbatore, Tamilnadu (**Appendix 1).**

Figure 1 *Bauhenia racemosa* Tree and Fruits



a) Crystal Violet (CV) **b) Acid Blue 110 (AB110)**

b) Remazol Black 5 (RB5) **d) Congo Red(CR)**

Figure 2 Dyes Used in this Study

3.1 PREPARATION OF THE ADSORBENTS

Adsorbent Sample 1: *Bauhenia racemosa* Fruit pods – Sulphuric Acid Activated Carbon

The fruit pods of *Bauhenia racemosa* were collected and washed repeatedly with water to remove dust and soluble impurities. It was then cut into small pieces, dried in sunlight for 10 days and further dried in hot air oven at 80℃ for 24 hours. The completely dried material was powdered well in mechanical grinder and chemically activated by treating it with concentrated sulphuric acid with constant stirring and kept for 24 hours at room temperature. It was then activated by heating at 110℃ in the hot air oven for 12 hours.

The carbonized material thus obtained was washed well with plenty of water several times to remove the excess acid present and then dried in hot air oven for 12 hours and in Muffle furnace at 550℃ for one hour. The adsorbent thus obtained was ground well and sieved through a 125-250 mesh and kept in air tight containers for further use **(Umadevi and Renuga devi, 2015; Theivarasu *et al.,* 2012**; **Sivakumar *et al.,* 2014).**

Sample II: *Bauhenia Racemosa* Fruit pods-Phosphoric Acid Activated Carbons

The fruit pods of *Bauhenia racemosa* were collected and cut into small pieces, dried in sunlight for 10 days and further dried in hot air oven at 80℃ for 24 hours. The completely dried material was powdered well and chemically activated by treating it with 60% phosphoric acid with constant stirring and kept for 24 hours at room temperature. It was then activated at 700℃ in muffle furnace for 30 minutes. The carbonized material thus obtained was washed well with plenty of water several times to remove excess of acid present and then dried at 110℃ to 120℃ in a hot air oven for 24 hours. The adsorbent thus obtained was ground well and sieved through a 125-250 mesh and kept in air tight containers for further use (**Umadevi and Renuga devi, 2018; Sivakumar *et al.,* 2014).**

The selected adsorbents - *Bauhenia racemosa* fruit pods - Sulphuric acid activated carbon and Bauhenia racemosa fruit pods - Phosphoric acid activated carbons are denoted as BR-SAC and BR-PAC respectively and the commercial activated carbon used in this study is denoted as CAC throughout this thesis work.

3.2 Analytical Procedure - Characterisation of the Adsorbents

3.2.1 Determination of pH

About 1.0g dried sample was weighed and transferred to 250ml beaker and 100ml freshly boiled (CO_2 free) and cooled distilled water is added and heated to boiling. After digesting for 5 minutes, the solution was filtered and cooled to room temperature. The supernatant solution was analysed for pH using Elico pH meter **(ISI: 1989).**

3.2.2 Determination of Moisture Content

About 5g dried sample was weighted in a Petri dish and it was placed in an electric oven maintained at 110±5℃ for about five hours. The dish was covered and cooled in a dessicator. Heating, cooling and weighing were repeated at 30 minutes interval until the difference between the two consecutive weights is less than 5mg. The loss in weight gives the moisture content **(ISI: 1989).**

Moisture content (%) = $\frac{M-X}{M} \times 100$

where M = Weight of the adsorbent (g)
X = Weight of the adsorbent taken after drying (g)

3.3.3 Ash Content

About 5g sample was weighed in Petri dish and it was dried in an electric oven maintained at 110±5℃ for about five hours. Two gram of dried sample was weighed accurately in a sintered porcelain crucible and the contents are ignited in an electric muffle furnace at a temperature of 800℃ for about 2 hours. The process of heating and cooling was repeated until constant weight was achieved **(ISI: 1989).**

Ash content (%) = $(10000\times M_1) / M_2(100 - X)$

where M_1 = Amount of ash (g)

M_2 = Amount of sample taken (g)

X = % of moisture content present in the sample (g)

3.3.4 Specific Gravity

Five gram oven dried carbon sample was placed in a small porcelain dish with about 50ml distilled water. The mixture was boiled for 3 minutes and cooled to 15℃. The carbon was transferred to a 100ml pycnometer using a small funnel. The pycnometer was filled with water stoppard, and weighed (W_c) **(ISI: 1989).**

$$\text{Specific gravity (S)} = \frac{weight\ of\ adsorbent\ (\mathrm{Wa})}{volume\ of\ displaced\ water\ (\mathrm{V})}$$

$$V = \frac{Wa+Wb-Wc}{Density\ of\ water}$$

where W_a = weight of adsorbent W_b = weight of pycnometer with water

W_c = weight of the dried pycnometer

3.3.5 Bulk Density

A specific gravity bottle of 50ml capacity was packed with sufficient amount of carbon sample using a trip balance with constant tapping and filled to the 50ml mark. After filling the cylinder the weight of the adsorbent was determined. The bulk density was calculated using the formula **(ISI: 1989)**

$$\text{Bulk density (D)} = \frac{weight\ of\ carbon\ sample(\mathrm{g})}{volume\ of\ carbon\ sample(\mathrm{ml})}$$

3.3.6 Porosity

Porosity is calculated from the specific gravity (S) and bulk density (D) values of carbon using the formula **(ISI: 1989)**

$$\text{Porosity (\%)} = \frac{(S-\mathrm{D})\times 100}{S}$$

3.3.7 Methylene Blue Value (Decolourising power)

Methylene Blue (MB) value of activated carbon indicates the maximum volume of standard MB solution (0.15%) decolourized by a fixed amount of activated carbon. Carefully weighed 0.1g activated carbon was added to a flask containing 5ml standard MB solution (0.15%) and shaken to decolourize. Definite volume (1ml) of MB solution was added subsequently to the flask till complete decolourization occurred within 5 min. The entire volume of MB added was noted and the MB value is reported in mg/g **(ISI: 1989)**

$$\text{Methylene Blue value} = \frac{1.5 \times V}{M}$$

where V = Volume of Methylene Blue solution consumed (mL)

M = Mass of the sample taken (g)

3.3.8 The pH of zero Point of Charge (pHzpc)

The pH of zero point of charge (pHzpc) of activated carbon was determined by the pH drift method **(Thitame *et al.*, 2016**). $CaCl_2$ solution (0.005 M) was boiled to remove CO_2 and cooled. The initial pH was adjusted between 2 and 12 for each 20ml of the above mentioned solution using 0.5 M HCl / NaOH. Activated carbon (0.06g) was added and the solutions were equilibrated for 24 h. Finial pH was measured and plotted against the corresponding initial pH. The point where this curve intersects the straight line passing through origin indicates pHzpc.

3.3.9 Water Soluble Matter

About 10g carbon material was accurately weighted into a flask and 100ml water was added into it. This mixture was heated with continuous stirring after fitted with condenser for one hour. Then the mixture was filtered and the flask was rinsed with three 25ml portions of water and the rinsing is also added to the filtrate. The filtrate was cooled at room temperature and quantitatively transferred into 250ml standard flask and made upto the mark. The extract (50ml) was transferred to an already weighed china dish and evaporated to dryness on a water bath and finally dried in a hot air oven maintained at 110± 5℃. Then it was cooled in desiccators and weighed. The procedure of drying and weighing was repeated for a period of 30 minutes intervals **(ISI: 1989).**

$$\text{Matter soluble in water (\%)} = \frac{20{,}000 \times M_1}{M_2\,(100 - \mathrm{X})}$$

where M_1 = Mass of the residue (g)

M_2 = Mass of the material taken for test (g)

X = Percent of moisture present in the material.

3.3.10 Acid Soluble Matter

About 10g carbon material was accurately weighed into flask and 100ml water was added in to it. Conc. HCl (10ml) was added to this mixture and heated with continuous stirring after fitted with condenser for one hour. Then the mixture was filtered and the flask was rinsed with three 25ml portion of water and the rinsings were also added to the filter. The filtrate was cooled to room temperature and quantitatively transferred into 250ml standard flask and made upto the mark. 50ml of the acid extract was transferred to an already weighed china dish and evaporated to dryness on a water bath and finally dried in a hot air oven maintained at 110±5℃. Then it was cooled in a desiccator and weighed. The procedure of drying and weighing was repeated for a period of 30 minutes interval **(ISI: 1989).**

Material soluble in acid (%) = $\frac{20{,}000 \times \mathrm{A}}{\mathrm{M}\ (100-\mathrm{X})}$

where A = Mass of the dried residue (g)
M = Mass of the material taken for test (g)
X = Percent of moisture present in the material.

3.3.11 Determination of Surface Area of the Adsorbent (BET)

Surface area, pore size and pore volume of the adsorbents used in this study (BR-SAC, BR-PAC and CAC) were measured using BET (Brunauer, Emmett and Teller) analyzer (1994-2012, Quantachrome Instruments Version 3.0) at Chemistry research centre, Bangalore Institute of Technology, Bangalore, India. BET surface area of the adsorbents BR-PAC and BR-SAC were found to be 1083.448m^2/g and 288.700 m^2/g respectively.

The higher surface area of the adsorbent BR-PAC is due to weak oxidizing property of phosphoric acid, the walls of the existing micropores does not rupture to a large extend which results into comparatively higher surface area compare to that of sulphuric acid activated carbon **(Monal Dutta *et al.,* 2011).** In the case of sulphuric acid activation the lesser surface area of the adsorbent may be caused due to the strong oxidative nature of sulphuric acid which ruptures the walls of the existing micropores and breakdown the lignin from the inside of the adsorbent **(Mui *et al.,* 2010)** .The BET surface area results obtained were shown in **Appendix 2, 3 and 4.**

The characteristics of activated carbons BR-SAC, BR-PAC and CAC are presented in **Table 2.**

Table 2 Characteristics of Activated Carbons BR-SAC, BR-PAC and CAC

S.No	Parameters	BR-SAC	BR-PAC	CAC
1	pH	6.6	6.9	7.2
2	Moisture content (%)	2.18± 0.07	1.81±0.05	1.14±0.05
3	Ash content (%)	2.22±0.04	2.94±0.04	2.5±0.04
4	Specific gravity	1.49	1.32	1.25
5	Bulk density (g /ml)	0.725	0.675	0.689
6	Porosity %	51.34	48.86	44.88
7	Methylene blue adsorption (mg/g)	186	363	374
8	(pHzpc)	6.1	6.3	-
9	Acid soluble matter (%)	2.88	1.70	2.56
10	Water soluble matter (%)	2.10	1.62	1.54
11	BET Surface area (m^2/g)	288.700	1083.44	1137.962
12	Pore volume (cm^3/g)	0.183	1.113	0.319
13	Pore size (A^0)	12.20	15.19	12.273

Procedure for Effluent Analysis (Gobalan, 2009)

Colour

Preparation of Standard Solution:

K_2PtCl_6 (1.246 g) and $CoCl_2.6H_2O$ (1 g) dissolved in 400 ml of distilled water and 100 ml conc. HCl and made upto 1000 ml using distilled water. 1 ml of this has colour units of 0.5 Hazen unit.

Standard platinum solution (1 – 10 ml) was pipetted out into a set of ten 50 ml Nessler tubes. It is then diluted with distilled water upto the make and stirred. The sample water was taken in other Nessler tube and filled upto the mark. The colour was matched with colours of standard solution and the sample colour was fixed.

Turbidity

The turbidity was measured by Nephelometric method. The light source illuminates the sample and one or more photoelectric detectors are used to indicate the intensity of scattered light at right angle to the path of the incident light. The values are expressed in Nephelometric turbidity units (NTU)

Chemical Oxygen Demand (COD)

Water sample (50 ml) was taken in 500 ml conical flask. 25 ml of standard potassium dichromate (0.0125 N) and 50 ml conc. H_2SO_4 solutions were added with gentle mixing. To this mixture added 1 g silver sulphate and 1 g mercuric sulphate. The mixture was refluxed for 2 hours on the heating mantle. After cooling 0.5 ml ferroin indicator was added and unreacted dichromate was determined by titration with standard ferrous ammonium sulphate (0.012 N) taken in the burette. The end point is the colour change from blue to reddish brown. A duplicate was conducted simultaneously. The blank titration was performed using 50 ml of distilled water

$$\text{COD (mg L}^{-1}\text{)} = \frac{(V_2 - V_3) X\ N\ X 8\ X\ 1000}{V_1}$$

V_1 = Volume of water sample taken (ml)

V_2 = Blank titre value (ml)

V_3 = Titre value with the water sample (ml)

N = Normality of the ferrous ammonium sulphate solution

Biological Oxygen Demand (BOD)

Four BOD bottles (200 ml capacity) were filled with water sample upto the brim and tightly stoppered (No air bubbles should be present inside the bottles). The water sample in two bottles were used immediately to determine the dissolved oxygen. The other two bottles were seeded with 1ml waste water and placed in incubator at 20°C. After 5 days the dissolved oxygen in these two bottles were determined.

$$\text{BOD (mg l}^{-1}\text{)} = \text{DO (b)} - \text{DO (a)}$$

DO (b) Dissolved oxygen before incubation
DO (a) Dissolved oxygen after incubation

Determination of Chloride

Water (100 ml) was pipetted out into a conical flask. 1 ml of potassium chromate indicator was added. This mixture was titrated against standard silver nitrate solution (0.005 M) taken in burette. The end point is the appearance of reddish brown colour. The duplicate titration was conducted in the same way. The blank titration using 100ml de-ionised, chloride free water using 1 ml indicator was carried out. This value was subtracted from that obtained for the water samples for indicator correction.

Chloride in water sample (mg l^{-1}) = $\frac{35.45 \, X \, V_2 \, X \, M \, X \, 1000}{V_1}$

V_1 = Volume of water sample (ml)
V_2= Corrected titre value for water sample titration (ml)
M= Molarity of $AgNO_3$

Total Dissolved Solids (TDS)

A clean 150 ml beaker was placed in an air oven at 103 -105° C for 1 hour. The beaker was removed from the oven and placed in a desiccator till it cool's and then weighed (w_1 g). 100 ml of sample water was taken in a beaker after filtering through 0.45 µm filter paper. The water sample was evapourated on a water bath to reduce the volume of sample to approximately 10 ml.It is placed in hot air oven at 103 -105℃ for 1 – 1 ½ hours. After cooling in the desiccator the weight was determined (w_2 g). The procedure was repeated for concordant value.

$$\text{TDS} = [(w_2 - w_1)/100] \times 10^6 \text{ mg / l}$$

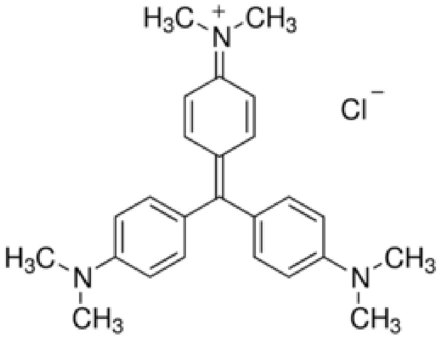
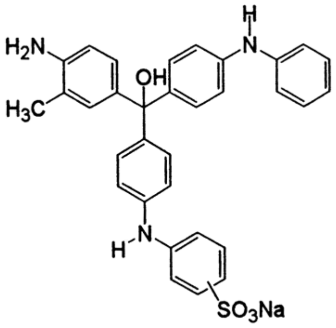
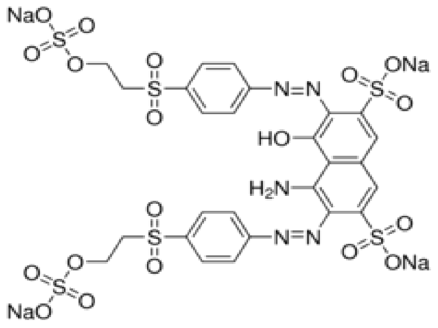
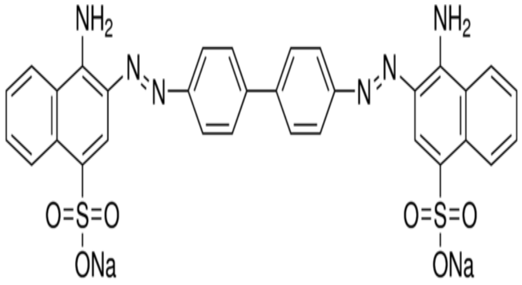
w_2 = Weight of beaker after filtration
w_1 = Weight of beaker before filtration

Total Suspended Solids

Two filters were washed with double distilled water and dried separately in a hot air oven at 104° C for 30 minutes. It is then placed in desiccators for cooling, and weighed. 100 ml sample water was passed through each pre-weighed filters and the filters were placed in oven at 104° C for 1 hour. After cooling the filters in desiccators it is weighed.

TSS (mg/L) = Average weight two filters after passing the sample (g) - Average weight of two filters before passing the sample (g)(1000mg/l) / Sample volume in (l)

Table 3 List of Dyes Used

Name of the Dye	Structure of the Dye
Crystal Violet **Molecular Formula : $C_{25}H_{30}N_3Cl$** **Molecular Weight : 407.98** **C.I. No : 20505** **CAS Number : 548-62-9** **λmax : 590 nm**	
Acid blue110 **Molecular Formula : C32H28N3NaO4S** **Molecular Weight : 573.64** **C.I. No : 42750** **CAS Number : 62152-67-4** **λmax : 603 nm**	
Reactive Black 5 **Molecular Formula : C26H21N5Na4O19S6** **Molecular Weight : 991.82** **C.I. No : 20505** **CAS Number : 17095-24-8** **λmax : 597 nm**	
Congo Red **Molecular Formula : C32H22N6Na2O6S2** **Molecular Weight : 696.66** **C.I. No : 22120** **CAS Number : 573-58-0** **λmax : 498nm**	

3.4 Preparation of Adsorbate

The dyes Crystal Violet (CV), Acid Blue 110 (AB110), Remazol black 5(RB5) and Congo red (CR) used in the present study **(Figure 2)** were procured commercially from SD-Fine chemicals, Mumbai and used without further purification. All the chemicals used in this study were of analytical grade. The chemical structure of the dyes used in this study is shown in **Table 3.** The stock solutions were prepared by dissolving 1g of dye in 1000ml of distilled water.

3.5 Equipments

Elico -110 pH meter was used measure pH. The pH meter is standardized using buffer 4 and 7.

Digital Spectrophotometer (Deep Vision, Model 2306) was used to measure the residual dye concentration.

Horizontal Electrical Bench Shaker (Lab-Line 3527 Bench Top Orbit Shaker) with temperature control was used for agitation of the solution containing the adsorbate and adsorbent.

3.6 Batch Adsorption Experiments

Batch adsorption studies were carried out to study the adsorption capacity of the adsorbents BR-SAC and BR-PAC. These experiments are simpler and very effective for assessing the basic parameters affecting the adsorption process.

3.6.1 Effect of Variation of Initial Concentration of the Dye on Adsorption

Batch mode experiments were performed with BR-SAC **/** BR-PAC adsorbent by varying the initial dye concentration at pH 6.8±0.2 and at room temperature (32±2℃). Different initial concentration (30, 50, 70 and 100 mg/l) of dye solutions (100ml) were taken in Pyrex bottles having 100mg BR-SAC / 50 mg BR-PAC adsorbent. Then the adsorbent and adsorbate mixture was agitated at 120 rpm speed on a horizontal electrical bench shaker for various time intervals (10, 20, 30, 40, 50, 60, 80, 100, 120 and 180 minutes). The adsorbate was separated from the adsorbent by centrifugation and the residual dye concentrations were measured using spectrophotometer at 590 nm for Crystal Violet dye, at 603nm for Acid Blue 110 dye, at 597nm for Reactive Black 5 dye and at 498 nm for Congo Red dye solutions.

3.6.2 Effect of Variation of pH on the Adsorption of Dye:

The influence of pH on dye adsorption was studied by changing the pH from 2 to 10. To explore the influence of pH, 100ml dye solution of initial concentration of 100mg/l were taken in Pyrex bottles containing 100mg BR-SAC **/** 50mg BR-PAC adsorbent. The pH of the dye solutions were adjusted using 0.1N H_2SO_4 or 0.1N NaOH. These solutions were agitated for a time interval of 120 minutes. The adsorbate was separated from the adsorbent by centrifugation and the residual dye concentration was measured using spectrophotometer.

3.6.3 Effect of Variation of Adsorbent Dosage on the Adsorption of Dye:

The influence of adsorbent dosage on the removal of dyes (CV, AB110, RB5 and CR) was studied at pH 6.8±0.2 and at temperature 32 ± 2℃. The dye solution (100ml) of initial concentration 100 mg/l was taken in Pyrex bottles containing 50, 100, 150 and 200 mg of BR-SAC adsorbent **/** 25, 50, 75 and 100 mg BR-PAC adsorbent. The adsorbent and adsorbate mixture was shaken using horizontal electrical bench shaker at room temperature by varying the contact time from 10 to 150 minutes. The adsorbate was separated from the adsorbent by centrifugation and the residual dye concentration was measured using spectrophotometer.

3.6.4 Effect of Variation of Temperature on the Adsorption of Dye:

To investigate the influence of temperature on the adsorption of dyes used in this study, 100ml dye solution of initial concentration 100mg/l was taken in Pyrex bottles containing 100mg BR-SAC **/** 50mg BR-PAC adsorbent at pH 6.8±0.2. These solutions were agitated in a temperature controlled water bath cum rotary shaker at different temperatures (27°C, 37°C and 47℃) by varying the contact time from 10 to 120 minutes. The adsorbate was separated from the adsorbent by centrifugation and the residual dye concentration was measured using spectrophotometer.

3.6.5 Characterization of Industrial Waste Water

The textile dyeing effluent containing CV dye (Sample 1), AB110 dye (Sample 2), RB5 dye (Sample 3) and CR dye (Sample 4) were collected from Balamurugan bleaching and dyeing unit Tirupur, the **"Banian City"** of the South India, Tamil Nadu and Sri Ganga bleaching and dyeing unit Udumalpet, Tirupur district, Tamil Nadu. Batch adsorption experiments were conducted with BR-SAC **/** BR-PAC adsorbents for effluent samples 1-4 and characteristics such as Colour, Odour, Turbidity, pH, BOD, COD, Total suspended solids, Total dissolved solids and Chloride content of effluent samples before and after adsorption studies were determined from Analytical and Advisory unit of Tamil Nadu Agricultural University, Department of Environmental Sciences, Coimbatore. **(Appendix 5 & 6)**

3.6.6 Batch Adsorption Studies with Adsorbent CAC

Commercial activated carbon was purchased from SD Fine Chemicals, Mumbai, India and used as such for the present work. To compare the adsorption capacities of BR–SAC and BR–PAC with Commercial Activated Carbon (CAC) batch adsorption experiments were conducted after adjusting the pH of the effluent to 6.8 ± 0.2 using 0.1N H2SO4. The diluted effluent (100ml) of dye concentration 100mg/l with 100mg of the adsorbent (CAC) were agitated using horizontal electrical bench shaker for predetermined time intervals of 10 to 180 minutes at 32 ± 2°C. The solutions were centrifuged and the centrifugate was analysed to find out the residual dye concentration using spectrophotometer.

3.7 Kinetic, Thermodynamic and Adsorption Isotherm Studies for the Removal of Dyes

The results of the experimental studies obtained above were used to find out the rate constants for the adsorption of the dyes from aqueous solution using Lagergren rate equation, Intraparticle diffusion rate equation and Elovich rate equation. Thermodynamic parameters (ΔS, ΔG and ΔH), Langmuir and Freundlich adsorption isotherms were also assessed for the adsorption of CV, AB110, RB5 and CR dyes using the low cost adsorbent prepared and used in this study.

3.8 Field Emission Scanning Electron Microscope

The surface morphology of the adsorbents BR-SAC and BR-PAC before and after adsorption of the dyes were observed using Field Emission Scanning Electron Microscope. **(Model: MIRA3 TESCAN).** FESEM works with electrons liberated by a field emission source and accelerated in a high electrical field gradient. Within the high vacuum column these primary electrons are focused and deflected by electronic lenses to produce a narrow scan beam that bombards the object. As a result secondary electrons are emitted from each spot on the object. The angle and velocity of these secondary electrons relates to the surface structure of the object. A detector catches the secondary electrons and produces an electronic signal. This signal is amplified and transformed to a video scan image that can be seen on a monitor or to a digital image that can be saved and processed further **(Figure 3).** Thus FESEM is employed to study very small topographic details on the surface of the object **(www.vcbio.science.ru.nl/public/pdf/fesem_info_eng.pdf.).** The surface morphology of the adsorbents BR-SAC and BR-PAC were determined before and after adsorption of the dyes using Field Emission Scanning Electron Microscope.

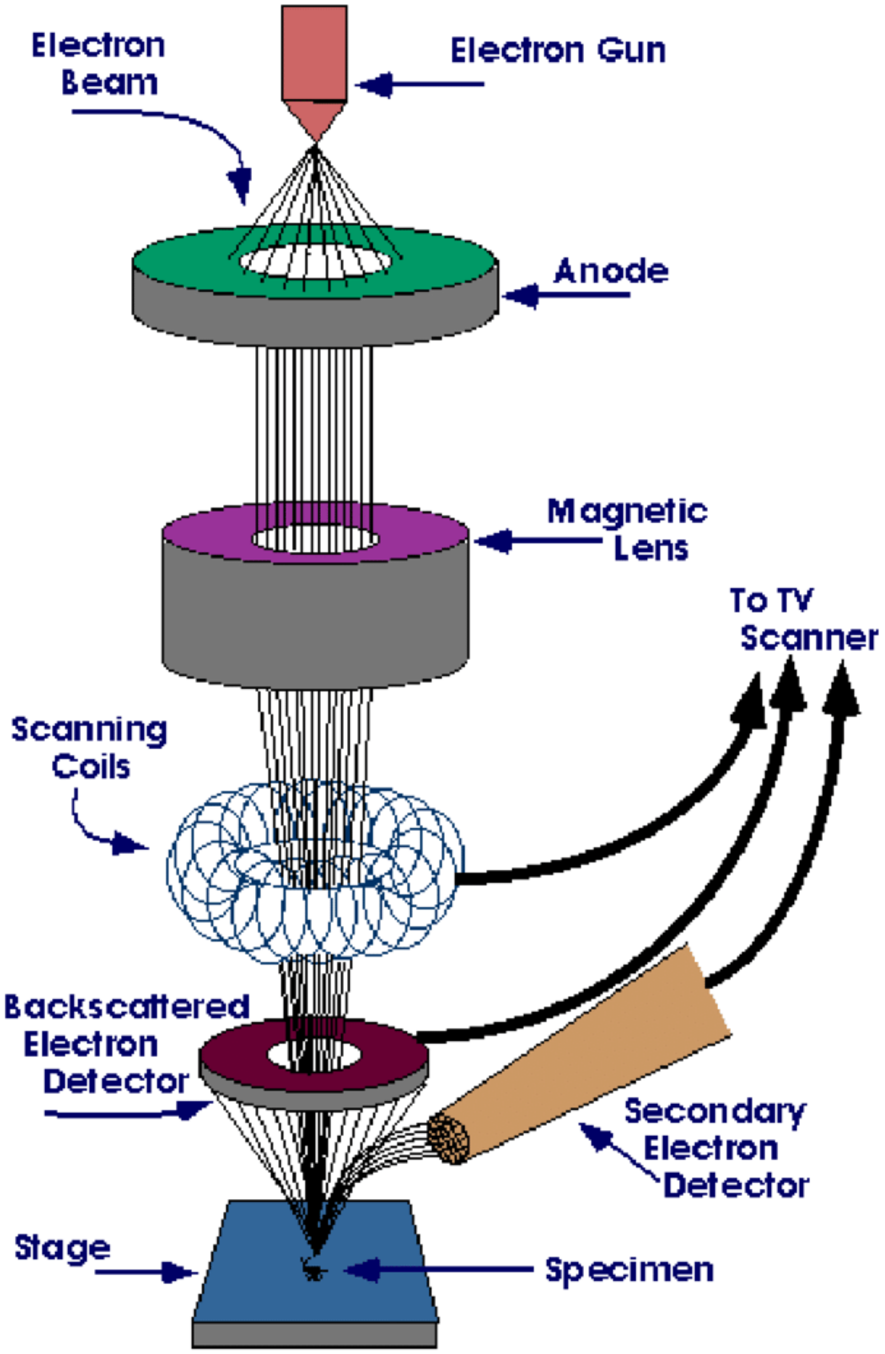


Figure 3 Field Emission Scanning Electron Microscope (FESEM)

3.9 FTIR Spectral Analysis of the Prepared Carbon Adsorbents

The carbon matrix does not consist of carbon atoms alone but is also formed by other hetero atoms like oxygen, nitrogen, halogen, sulphur, phosphorus etc. which are bonded to the edges of carbon layers which governs the surface chemistry of the activated carbon **(Yakout and Sharaf El-Deen, 2016; Arvind kumar and Hara Mohan Jena, 2016)**. The chemically bonded hydrogen and oxygen atoms present in the activated carbon are derived from the starting material and stay behind because of imperfect carbonization or they chemically bonded to the carbon surface during activation. Oxygen combines with carbon to form oxides and these oxides undergo hydration to form surface hydroxyl groups **(Karthikeyan, 2007).**

Infrared spectroscopic analysis is used for the identification of the organic functional groups present on the surface of the activated carbon. The infra red spectrum of the adsorbents BR-SAC and BR-PAC were obtained using a Fourier transform infrared spectrometer using KBr pellet technique **[SHIMADZU MIRacle10 (ZnSe)].**

3.10 Energy Dispersive X-ray Analysis (EDX)

Energy Dispersive X-ray Analysis (EDX) is an effective analytical technique used to determine the elemental composition of a sample when combined with imaging tool like Scanning Electron Microscopy. In EDX spectrum the electron beam strikes the surface of the sample and produce X-rays from the irradiated substance that are characteristic of the elements present in the sample. The elemental composition of the two adsorbents BR-SAC and BR-PAC obtained from EDX studies were studied using **MIRA3 TESCAN FESEM coupled with EDX**. APEX software has been used for data processing and analysis **(Kibami *et al.*, 2017).**

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