

PRODUCTION AND CHARACTERIZATION OF A NOVEL NON-CONVENTIONAL LOW-COST ADSORBENT FROM *EUPHORBIA ANTIQUORUM L*

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ABSTRACT

Activated carbon samples are characterized by their high adsorption capacities. Although the highly active surface properties of the activated carbon are attributed to the chemical functional groups. Surface morphology also plays a significant role in the adsorption properties along with surface functional groups. In this study, the influence of activation methods on the preparation of activated carbon from *Euphorbia antiquorum L* wood was studied. The resulting samples were analyzed for their physico-chemical characteristics. The results indicate that an activated carbon with high adsorptive properties can be conveniently prepared from *Euphorbia antiquorum L* wood. Impregnation with H₃PO₄ solution process followed by activation at 800° C yields activated carbon with the highest surface area and more developed porosity. Suitability of the potential adsorbent was analyzed for the various categories of synthetic dye solutions.

Keywords: *Euphorbia antiquorum L* wood, Activated carbon, Carbonization process, Surface area, Adsorption.

INTRODUCTION

Water pollution due to the discharge of dyeing industry effluent is a major concern now days. Dyeing industrial waste water adversely affects the environment due to its high toxicity and non-biodegradable nature. Many techniques like electrochemical coagulation, Reverse osmosis, nano filtration, adsorption using activated materials etc., are used for the removal of dye from waste water. Adsorption has been found to be an efficient and economic process to treat dyeing industry effluent. In recent years, many naturally occurring waste materials have been investigated to evaluate their suitability and ability to be used as an adsorbent.

Recently some agricultural wastes and forestry products have been developed as adsorbents. The costs of these biomaterials are negligible when compared to the cost of commercial activated carbon and also renewable sources of raw materials for the production of activated carbon. Activated carbon prepared from different materials like agricultural wastes¹, coir pith², oil palm waste³, coconut shell waste⁴, tropical wood⁵, saw dust⁶, pinus pinaster bark⁷, corncob⁸, eucalyptus bark⁹, pistachio shells¹⁰, babool wood¹¹, etc., Basically there are two different processes for the preparation of activated carbon: physical and chemical activation¹². Physical activation involves carbonization of precursor followed by activation of the resulting char in presence of activating agents such as CO₂ or steam. Chemical activation on the other hand, involves the carbonization of precursor in presence of chemical agents¹³. In the chemical impregnation methods, first impregnation of precursor with the selected chemical followed by carbonization and activation. In case of Acid process, charring of the material done with acids like H₂SO₄, HCl and HNO₃ followed by activation. The purpose of carbonization and charring is to remove the volatile matter and increase the carbon content. Chemical activation has more advantages¹⁴ over physical activation with respect to higher yield, more surface area and better development of porous structure, oxygenated surface complexes in carbon.

In this investigation, an attempt is made to prepare activated carbon from *Euphorbia antiquorum L* wood by various physical and chemical activation processes. About the precursor, it is widespread throughout peninsular India, can be found growing up to an altitude of 800 m. One of the largest armed tree Euphorbias with an average height of 5 - 7 m., it has been known to attain gigantic proportions if left

undisturbed. The odour of its latex is pungent and lingering. Easily propagated from seed or vegetatively and the most important point about this plant is it does not have any economical importance.

EXPERIMENTAL

Euphorbia antiquorum(L) wood used as precursor for the preparation of activated Carbon. The wood was cut into pieces of 2 cm to 3 cm size, dried in sunlight for 10 days, and used for the preparation of activated carbon by various processes. The carbonized material was sieved to 300 μm to 850 μm (20-50 ASTM mesh) size and characterized using physico-chemical methods and used for adsorption studies. Table 1 shows the list various activated carbons and their preparation methods.

Carbonization procedures¹⁵

Acid process

The dried material was treated with excess of sulphuric acid and Hydrochloric acid respectively. Charring of the material occurred immediately, accompanied by evolution of heat and fumes. When the reaction subsided, the mixture was left in an air oven maintained at 140-160°C for a period of 24 hours. At the end of this period, the product was washed with large volume of water to remove free acid, dried at 110° and finally activated at 800°C.

Carbonization with H₃PO₄

The material to be carbonized is impregnated with a boiling solution of 10 % H₃PO₄ for 2 hrs and soaked in the same solution for 24 hours. At the end of 24 hours, the excess solution decanted off and air dried. Then the material was carbonized in muffle furnace carbonized at 400°C. The dried material was powdered and activated in a muffle furnace at 800°C for a period of 10 minutes. Then the material was washed with plenty of water to remove residual acid, dried and powdered.

Carbonization with chloride salts

The material to be carbonized is impregnated with a boiling solution of 10 % zinc chloride for 2 hrs and soaked in the same solution for 24 hours. At the end of 24 hours, the excess chloride solution decanted off and air dried. Then the material carbonized in muffle furnace at 400°C. The dried material was powdered and activated in a muffle furnace kept at 800°C for a period of 10 minutes. After the activation, the carbon washed sufficiently with 4N HCl to remove the cations. Then the material was washed with plenty of water to remove residual acid, dried and powdered.

Carbonization with sulphate salts

In this method the precursor material was soaked in 10% solution of sodium sulphate for a period of 24 hours. After impregnation, the liquid portion was decanted off and then dried. The dried mass was subjected to carbonization process at 400°C, powdered well and finally activated at 800°C for a period of 10 minutes.

Direct pyrolysis

The precursor material was subjected to carbonization process at 400°C, powdered well and finally activated at a temperature of 800°C for a period of 10 minutes. After the activation, the material was repeatedly washed with plenty of distilled water.

Dolomite process

Sufficient quantity of dried material taken over a calcium carbonate bed and the upper layer of precursor also covered with a layer of calcium carbonate. The whole material was carbonized at 400°C, powdered well and followed by the thermal activation at 800°C. After the activation, the material was repeatedly washed with excess water to remove calcium carbonate and dried at 110°C.

Chemical activation with H₂SO₄+H₂O₂

In this method 1 part of the material and 1.5 parts of H₂SO₄ were mixed with 0.4 parts of H₂O₂ and kept in muffle furnace at 120°C for 12 hours. At the end of this period, the product was washed with large volume of distilled water to remove free acid, dried at 110°C and finally activated at 800°C.

Characterization of the carbons

Physico-chemical characteristics of the activated carbon samples were studied as per the standard testing methods.^{16,17&18} The N₂ adsorption-desorption isotherms of activated carbon were measured at 77K using

N₂ gas sorption analyzer (Nova 1000, Quanta Chrome Corporation) in order to determine the surface area using the BET equation. Fourier Transform Infrared (FT-IR) measurements were carried out using AVATOR-360 FT-IR (Thermo Nicolet-USA) over the range 4000 – 400cm⁻¹. Carbon samples (0.33 wt%) were stirred with dry KBr (Merk, spectroscopy grade) and then pressed to form appropriate tablets. The Morphological characteristics of the samples were studied using JSM-5610LV (Make: JEOL-JAPAN) Scanning Electron Microscope(SEM).

RESULTS AND DISCUSSION

General Properties

The characteristics of the activated carbon prepared from *Euphorbia antiquorum* L stem in various methods were listed in Table 2. The pH value of the carbon prepared by acid processes (EAC1, EAC2, EAC3 and EAC5) were acidic. This may be due to the introduction of acidic groups on to the activated carbon surface. Except the above said four all the remaining five carbon were basic in nature. Most of the commercial activated carbon are basic in nature¹⁹. EAC9 exhibits a higher pH of 7.80 may be caused by residual KOH in the carbon structure. The details of functional groups present in the surface of carbon is not clear, however the possible reactions during chemical activation of pistachio-nut shells using KOH are reported by Yang and Lua²⁰. M.Radika and K.Palanivelu²¹ reported the pH of the activated carbon above 7.0 during the activation of coconut shell carbon by KOH.

EAC4, EAC7 and EAC8 showed higher conductivity, EAC3 and EAC4 also found with moderate conductivity. This fact may be due to the development of exchangeable sites on the surface of activated carbon¹⁵. However, the conductivity of EAC5 was very low, indicates the absence of more exchangeable sites. Carbon derived by KOH activation and pyrolysis have high moisture content of 10.54 % and 10.51 % respectively. Even though moisture content of the carbon has no effect on its adsorptive power, it dilutes the carbon and necessitates the use of additional weight carbon during treatment process¹⁵. Moisture content of the other carbons were similar to the commercial activated carbon as well as comparable with the results of earlier researchers^{13 & 22}.

Ash content of an activated carbon gives an indication about the amount of inorganic constituents present in a carbon. EAC3 and EAC9 has more ash content, which may be incorporated by the activating agents. Carbon prepared by pyrolytic method (EAC6) shows least ash content, which supports the above said assumption. High ash content ultimately reduces the fixed carbon percentage.

Activated carbon derived from most of the naturally occurring plants and agro products have high percentage of volatile matter. This is due to the presence of more volatile organic compounds in the precursor. Solubility studies of carbon in water and acid were performed separately to evaluate the amount of impurities present in the carbon prepared by different carbonization processes, this may affect the quality of treated water during the treatment process. From the data it was clear that all the carbon have very low level of water soluble matter and acid soluble matter.

Generally, an adsorbent with higher bulk density need not be regenerated frequently because it can hold more adsorbate per unit weight²³. Bulk density values of all carbons found only in a narrow range. Indicating that activating agents have least role in altering the bulk density, as well as it depends on the nature of precursor.

Adsorptive properties are directly related to the porosity of activated carbon, as the highly porous carbon can adsorb relatively large amount of organic compounds. EAC5 is one of the highly porous carbons among the nine varieties. The above said factor was supported by the specific surface area that H₃PO₄ activated carbon has highest specific surface area of 918 m² g⁻¹. Iodine number can be correlated with the ability of a carbon to adsorb low molecular weight substances. As iodine molecule is relatively small and so provides a measure of surface area or capacity to adsorb small adsorbates. Iodine number for commercial adsorbent ranges from 300 to 1200 mg/g¹³. Methylene blue number and Phenol number is an indication of ability of a carbon to adsorb high molecular weight substances like dye molecule. EAC3, EAC4 and EAC5 shows methylene blue number of greater than 200, indicates that the carbon is good for dye adsorption.

The level of sodium is high in the case of EAC4 and EAC7. High level of potassium noticed in KOH impregnation method. This can be taken as an advantage for ion exchange process, for the removal of cations in wastewater. High charring nature of HCl and H₂SO₄ produces less ash and ultimately results more fixed carbon. High yield obtained in the dolomite process followed by H₃PO₄ impregnation process. The results of characterization studies indicate that *Euphorbia antiquorum* L stem is a good precursor material for the production of activated carbon of high efficiency. Especially ZnCl₂ and H₃PO₄ impregnation methods give more porous and high surface area carbon suitable for wastewater treatment.

FT-IR Characteristics

IR measurements of *Euphorbia antiquorum* L stem activated carbon (Fig. 1a to 1i) showed the presence of the following groups. A sharp peak at 3732 cm⁻¹ found in EAC5. This is due to the introduction of surface hydrogen bonded strong -OH groups by H₃PO₄. Absence of most of the -CH and -NH peaks proves that the solid surface of carbon homogeneous and does not have any specific functional groups other than -OH group. In the case of EAC5 surface area and porosity plays a major role in adsorption rather than surface functional groups. Except EAC5 and EAC8 all the other carbons shows a broad peak around 3450 cm⁻¹ due to -OH str vibrations. This is attributable to the surface hydroxyl groups and chemisorbed water^{24 & 25}.

Except EAC8 all the remaining eight carbons shows a peak at 2850 cm⁻¹ to 2920 cm⁻¹ for -CH str vibrations. -CH bending at 1400 cm⁻¹ and -C-C- str at 1050 cm⁻¹ exhibited by all the carbons. Presence of -CH def. were noticed in EAC1, EAC2, EAC3 and EAC4 carbons around 840 cm⁻¹. The peak around 600 - 700 cm⁻¹ may be attributed to the carbon-halogen bond vibrations.

Scanning Electron Microscope

Examination of the SEM micrographs (Fig. 2a to 2i) of the clean activated carbon particles showed smooth areas with long ridges and rough areas with micropores and more number of edges. An electron microscope study reveals highly porous and branched particles particularly in EAC3, EAC5, EAC7 and EAC9. Small cavities, pores and more rough surfaces on the carbon sample indicate the presence interconnected porous network. Tubular pores and cavities will increase the surface area of the adsorbent by many fold. EAC5 with 2000x magnification clearly shows the morphology of highly porous activated carbon sample. Most of the particles are having very fine pores of <1 μm size in the cavity walls.

Dye adsorption Characteristics

Acid, reactive and direct dyes have anionic character, where as basic dyes are cationic in nature. In this study Basic Red 29, Acid Blue 92, Reactive Red 4 and Direct Blue 53 are selected for the analysis of adsorption characteristics of the activated carbon prepared by nine different preparation methods. Fig. 3 shows the structure of selected dyes and Fig. 4 shows the amount selected dyes adsorbed by the nine different activated carbon prepared from *Euphorbia antiquorum* L. The amount of basic Red 4 adsorption is very high in all the nine carbons. Especially EAC3, EAC4 and EAC5 shows excellent adsorption.

The difference in uptake of cationic dye can not be explained on the basis of surface area alone, as EAC1, EAC2, EAC7 and EAC8 has smaller surface area and absorbs considerable amount of cationic dye. As the carbons prepared by different preparation procedures, these carbons are expected to have different chemical structures and textural properties on their surface. Many researchers in the past¹⁵ have proved that the surface chemical structure of carbon can also influences the dye adsorption. In general basic red 4 shows good adsorption with acid treated samples. This may be due to the presence of surface acid groups (mainly carboxylic, anhydride, lactones and phenolic groups etc.). The acidic surface functional groups are good anchoring sites for basic dyes as a result of electrostatic interactions.

All the anionic dyes show comparatively lesser adsorption than basic red 4. Amount of Acid Blue 92 adsorption is slightly higher than Reactive Red 4 and Direct Blue 53. This may be due to its smaller molecular size. Reactive Red 4 and Direct Blue 53 are high molecular weight dyes; hence, they occupy more area in the activated carbon. This leads to the lower amount of adsorption on the carbon surface. While analyzing the pH of all the nine variety of carbon (nearly neutral), role of surface groups on the

adsorption anionic dyes are also not a prominent factor. Only the area of micro and meso pores plays a major role in the adsorption of anionic dyes.

The chemical structure of carbon surface is due to the presence of associated oxygen which is present in the form of two types carbon-Oxygen surface groups; one which is evolved as CO₂ and the second is evolved as CO on degassing. In order to examine the influence of these type of surface groups on the adsorption (Cationic and anionic dyes more clearly), activated carbons were oxidized with H₂SO₄ and H₂O₂ solutions. These oxidative treatments are known to enhance the amount of these Carbon-Oxygen surface groups. But the adsorption data clearly indicates that enhancement of dye adsorption by carbon-oxygen surface groups is very poor for the selected dyes. In general EAC3, EAC4 and EAC5 has best performance in absorbing all categories of dyes (anionic and cationic).

CONCLUSIONS

From the results of the present investigation, we can conclude that:

1. Activated carbon can be conveniently and economically prepared from *Euphorbia antiqorum* L.
2. The extensive characterization studies of the different activated carbon prepared by various processes reveal that the carbons obtained from *Euphorbia antiqorum* L can be assessed as superior grade carbon.
3. Porosity plays a major role in the selected adsorbent-adsorbate system rather than surface area and surface functional groups.
4. For the four categories of dyes tested, activated carbons with different surface characteristics can remove a maximum of 90.06 % of dye from its aqueous solutions.
5. ZnCl₂ and H₃PO₄ activation process produces highly active carbon with very high surface area.
6. Hence, the activated carbon prepared from *Euphorbia antiqorum* L using ZnCl₂, Na₂SO₄ and H₃PO₄ can be used for the treatment any category of dyeing industry effluent.
7. A detailed study on the kinetics and Isotherms of the above said four dyes is under progress.

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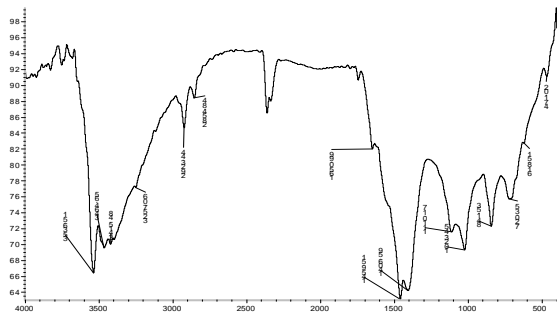


Fig. 1a FTIR spectra of EAC1

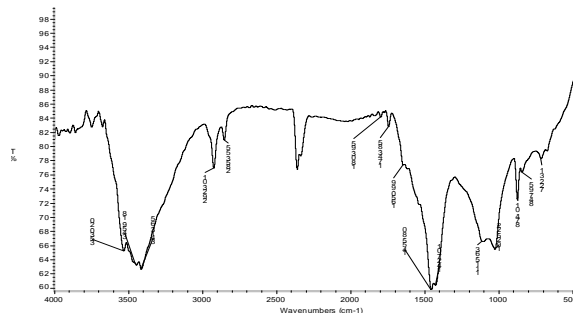


Fig. 1b FTIR spectra of EAC2

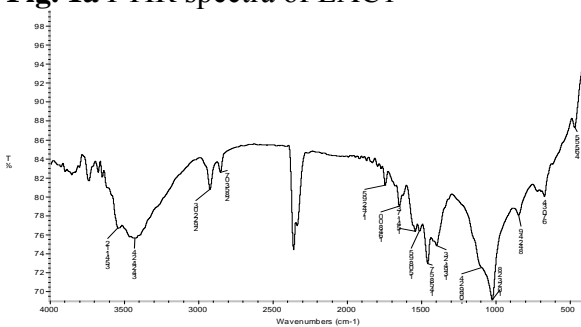


Fig. 1c FTIR spectra of EAC3

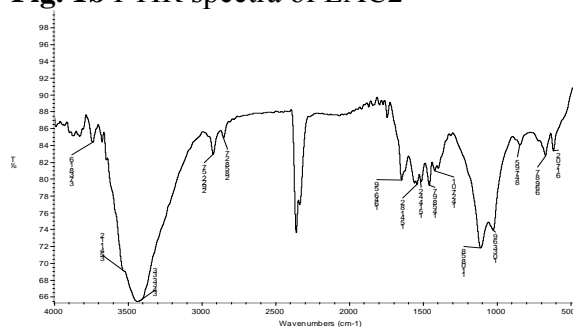


Fig. 1d FTIR spectra of EAC4

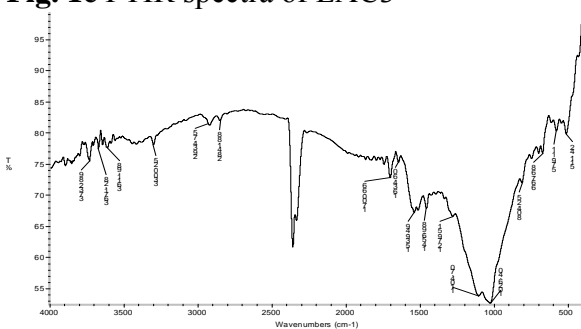


Fig. 1e FTIR spectra of EAC5

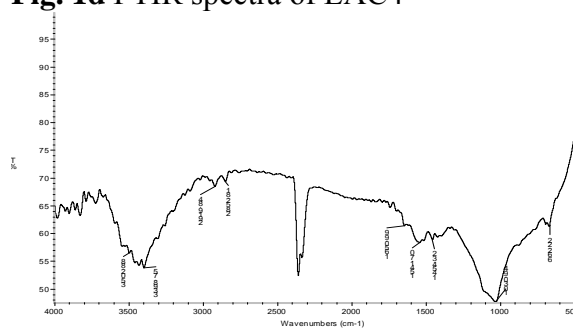


Fig. 1f FTIR spectra of EAC6

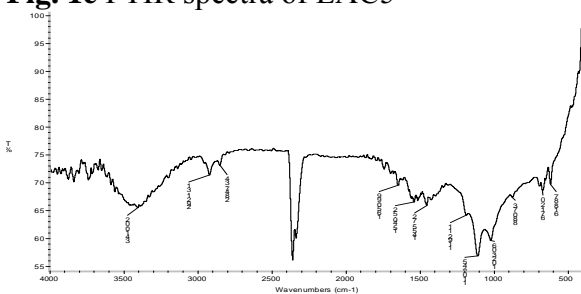


Fig. 1g FTIR spectra of EAC7.

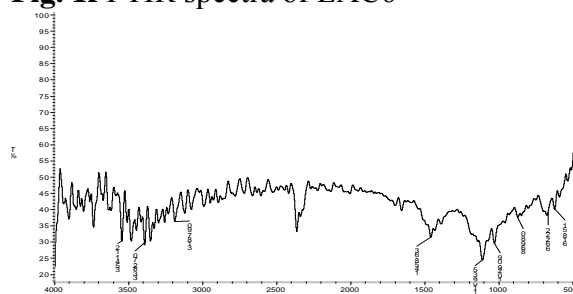


Fig. 1h FTIR spectra of EAC8.

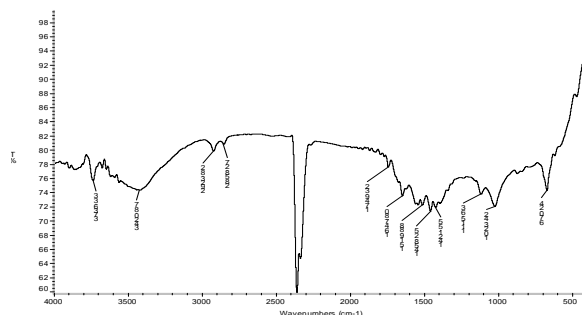


Fig. 1i FTIR spectra of EAC9.

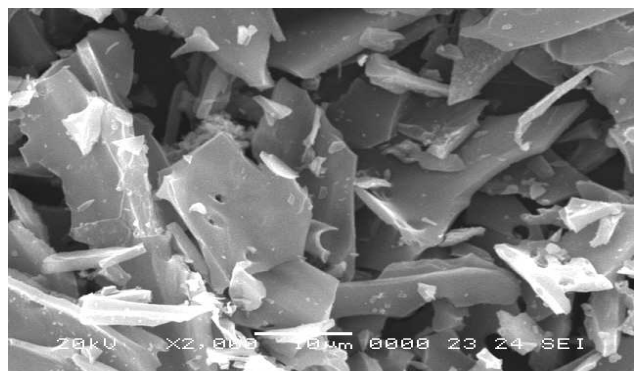


Fig. 2a SEM photograph of EAC1 (2000 X)

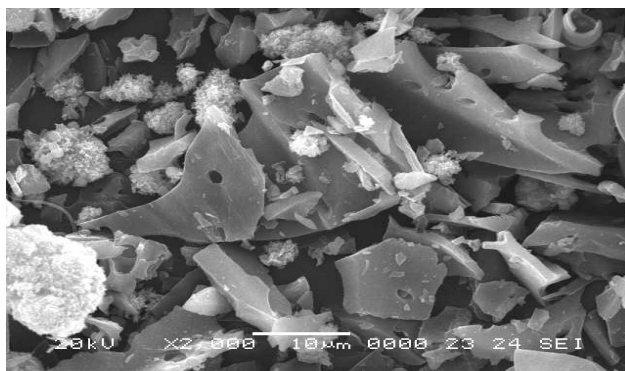


Fig. 2b SEM photograph of EAC2 (2000 X)

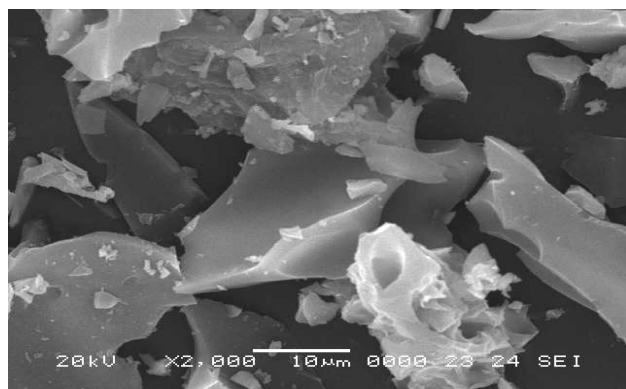


Fig. 2c SEM photograph of EAC3 (2000 X)

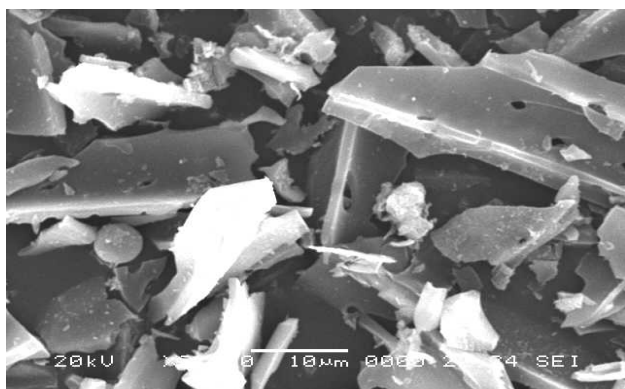


Fig. 2d SEM photograph of EAC4 (2000 X)

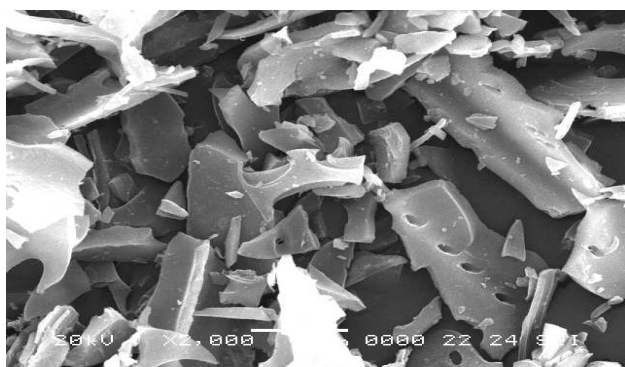
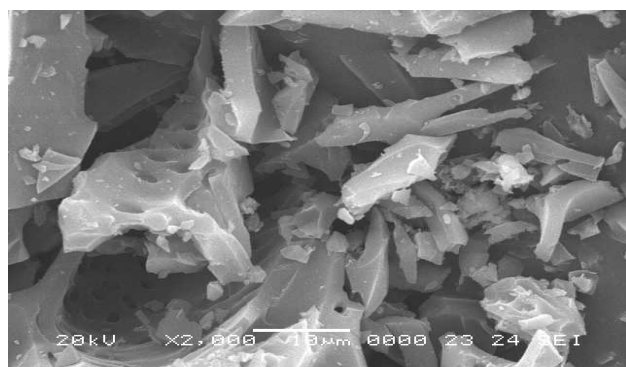


Fig. 2e SEM photograph of EAC5 (2000 X)

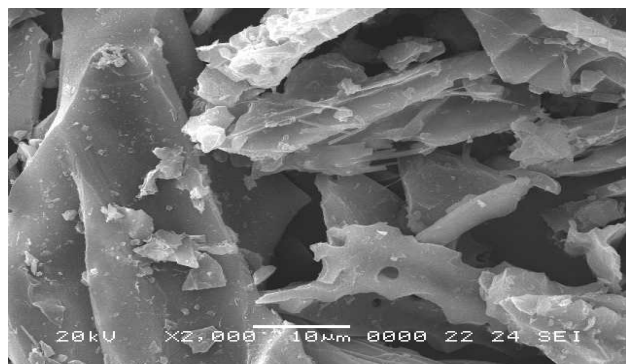


Fig. 2f SEM photograph of EAC6 (2000 X)

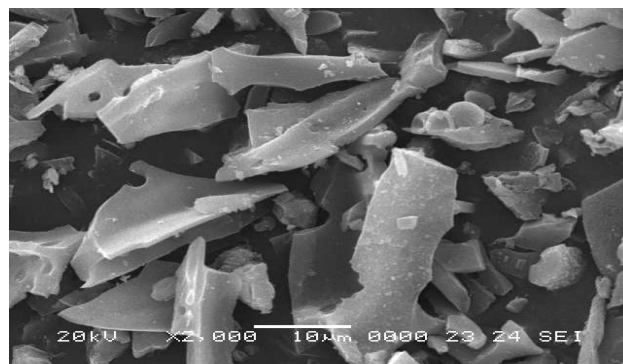


Fig. 2g SEM photograph of EAC7 (2000 X)

Fig. 2h SEM photograph of EAC8 (2000 X)

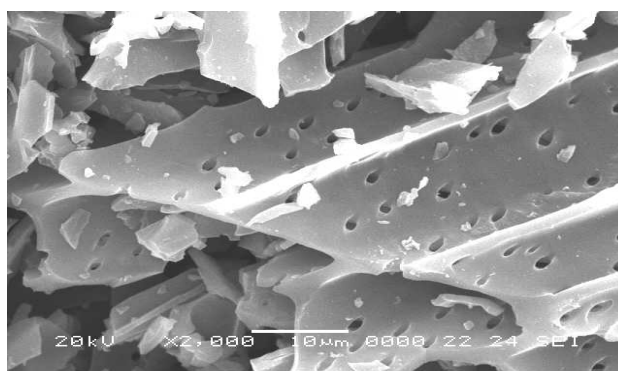


Fig. 2i SEM photograph of EAC9 (2000 X)

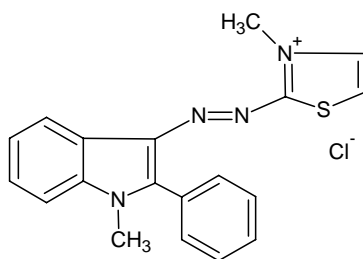


Fig. 3a Basic Red 29
(C.I No. 11460, F.W 368.89, λ max 511 nm)

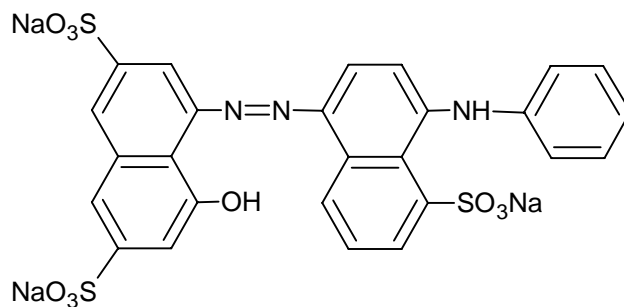


Fig. 3b Acid Blue 92(C.I No. 13390, F.W 695.59, λ max 571 nm)

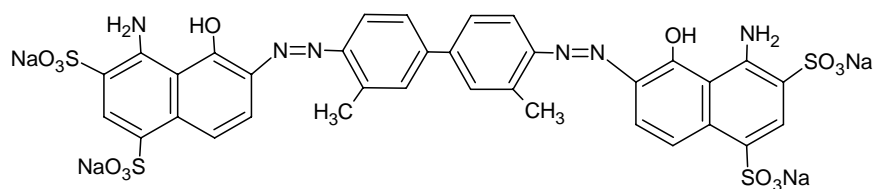


Fig. 3c Direct Blue 53
(C.I No. 23860, F.W 960.82, λ max 611 nm)

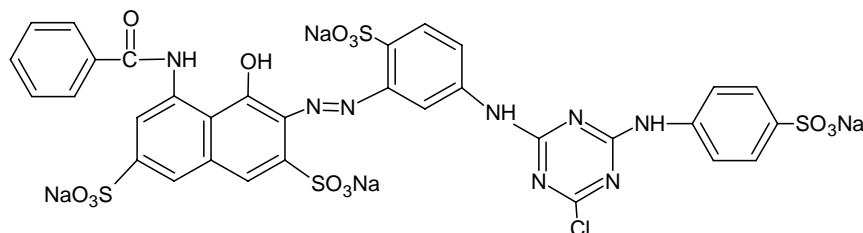


Fig. 3d Structure of Reactive Red 4
(C.I No. 18105, F.W 995.23, λ max 517 nm)

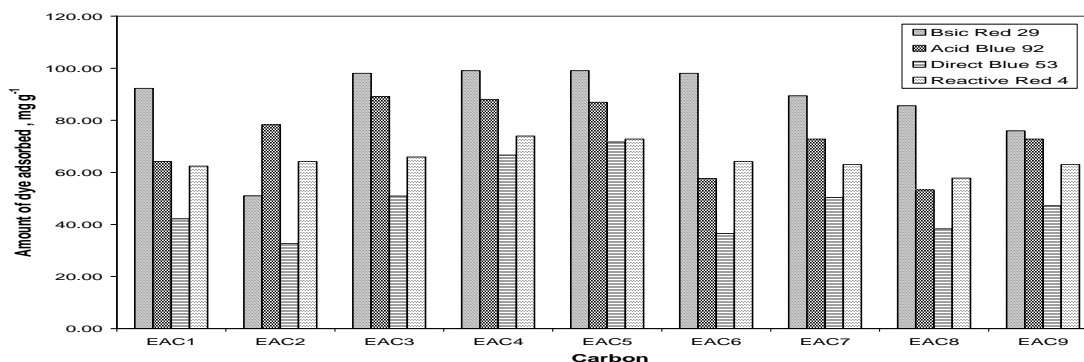


Fig.4 Amount of various dyes adsorbed onto various activated carbon prepared from *Euphorbia antiquorum L.*

Table-1: List of Activated Carbons prepared from *Euphorbia antiquorum L* and their preparation methods

| S.No. | Activated carbon | Preparation method |
|-------|------------------|---|
| 1. | EAC1 | HCl process |
| 2. | EAC2 | H ₂ SO ₄ process |
| 3. | EAC3 | ZnCl ₂ impregnation |
| 4. | EAC4 | Na ₂ SO ₄ impregnation |
| 5. | EAC5 | H ₃ PO ₄ impregnation |
| 6. | EAC6 | Physical carbonization |
| 7. | EAC7 | Dolomite process |
| 8. | EAC8 | H ₂ SO ₄ +H ₂ O ₂ process |
| 9. | EAC9 | KOH impregnation |

Table-2: Physico-chemical Characteristics *Euphorbia antiquorum L* activated carbon

| S.No | Properties ↓ | Carbon → | EAC1 | EAC2 | EAC3 | EAC4 | EAC5 | EAC6 | EAC7 | EAC8 | EAC9 |
|------|--|----------|--------|-------|-------|-------|-------|-------|-------|--------|-------|
| 1 | pH | | 6.62 | 6.95 | 6.72 | 7.33 | 6.90 | 7.53 | 7.56 | 7.30 | 7.80 |
| 2 | Conductivity, mS cm ⁻² | | 0.417 | 0.225 | 0.512 | 0.605 | 0.181 | 0.299 | 0.721 | 0.756 | 0.456 |
| 3 | Moisture content, % | | 6.62 | 7.23 | 8.87 | 8.44 | 7.56 | 10.51 | 9.45 | 6.18 | 10.54 |
| 4 | Ash, % | | 8.9 | 10.8 | 21.6 | 15.2 | 13.4 | 6.4 | 16.4 | 18.3 | 22 |
| 5 | Volatile matter, % | | 23 | 20.2 | 18.3 | 21.6 | 21.1 | 24.8 | 19.4 | 20.5 | 21.4 |
| 6 | Matter soluble in water, % | | 0.35 | 0.32 | 0.40 | 0.28 | 0.40 | 0.20 | 0.82 | 0.33 | 0.84 |
| 7 | Matter soluble in 0.25 M HCl, % | | 1.41 | 1.35 | 1.44 | 1.16 | 1.22 | 1.45 | 1.9 | 1.8 | 1.94 |
| 8 | Bulk density, g mL ⁻¹ | | 0.41 | 0.43 | 0.45 | 0.47 | 0.48 | 0.41 | 0.52 | 0.4 | 0.39 |
| 9 | Specific Gravity | | 0.83 | 0.78 | 0.85 | 0.94 | 0.94 | 0.89 | 0.78 | 0.91 | 0.81 |
| 10 | Porosity, % | | 50.60 | 44.87 | 47.06 | 50.00 | 55.32 | 53.93 | 33.33 | 54.94 | 51.85 |
| 11 | Surface area (BET), m ² g ⁻¹ | | 116.28 | 73.44 | 673.2 | 612 | 918 | 428.2 | 112.6 | 110.16 | 210.5 |
| 12 | Methylene Blue Value, mg g ⁻¹ | | 47.5 | 30 | 275 | 250 | 375 | 175 | 46 | 45 | 85.78 |
| 13 | Iodine Number, mg g ⁻¹ | | 125 | 91 | 710 | 634 | 955 | 455 | 122 | 119 | 227 |
| 14 | Phenol number, mg g ⁻¹ | | 10.26 | 3.84 | 11.77 | 12.36 | 10.73 | 10.65 | 2.81 | 7.26 | 5.14 |
| 15 | Sodium, mg g ⁻¹ | | 0.92 | 0.84 | 0.29 | 5.7 | 0.24 | 0.85 | 5.5 | 3.1 | 1.8 |
| 16 | Potassium, mg g ⁻¹ | | 0.88 | 0.90 | 0.83 | 0.71 | 0.62 | 2.3 | 0.93 | 0.88 | 3.14 |
| 17 | Iron, mg g ⁻¹ | | 0.08 | 0.04 | 0.02 | 0.05 | 0.12 | 0.04 | 0.13 | 0.07 | 0.06 |
| 18 | Fixed Carbon, % | | 61.48 | 61.77 | 51.23 | 54.76 | 57.94 | 58.29 | 54.75 | 55.02 | 46.06 |
| 19 | Yield, % | | 39.5 | 44.4 | 55.8 | 54.8 | 56.5 | 55 | 58.8 | 41.5 | 52.2 |

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