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Effects of various Carbonization Processes in the Preparation of Nanoporous Carbon Materials using *Ipomoea Carnea* Stem Waste for the Removal of Dyes from Textile Industrial Effluents

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Abstract

Nanoporous activated carbon materials were prepared from abundantly available Ipomoea Carnea stem waste. A series of five Nanoporous carbon samples were prepared by subjecting the carbon material to various chemical processes. The physico-chemical properties and adsorption capacities were compared. The adsorption capacity of selected derived carbon sample using Ipomoea Carnea stem waste prepared by Dolomite process was much greater than the capacities of the other carbons prepared by various processes viz., Acid process, $ZnCl_2$ process, Sulphate process and Carbonate process. The influence of surface area and porosity of the carbon samples on dye removal was analysed. The effects and influence of pKa values of dyes and isoelectric point (pH $_{ppc}$) of Nanoporous carbon materials were broadly studied and interpreted with dye adsorption onto Nanoporous carbon material. The results reveal that the prepared activated carbon could be employed as a lowcost alternative for removing toxic and carcinogenic dyes from the textile industrial waste water.

Keywords: Adsorption; Isoelectric point (pH_{zpc}); Nanoporous activated carbon; pKa values; Surface area; Surface chemical groups.

1. INTRODUCTION

Activated carbons having high specific porosity, high surface areas are extremely versatile adsorbents of major industrial significance. Activated carbon has been extensively used for the purpose of water purification. In particular, it has been commonly used for the removal of organic dyes from textile waste water. In order to improve the adsorption potential of

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***I. Arockiaraj** Tel. No.: +919842687930 E-mail: arockiaraj 5000@ rediffmail.com the raw carbon, it is subjected to various suitable chemical treatments. Carbon based sorbents show excellent adsorption properties for a considerable number of synthetic dyes. The presence of very small amounts of dyes in water even less than 1ppm for some dyes is highly visible and undesirable (Robinson *et al.* 2002). The removal of colour from textile waste water is a major environmental problem and they are the first contaminant to be recognized in water (Banat *et al.* 1996). Coloured water is also objectionable on aesthetic grounds for drinking and agricultural purposes (Renugadevi *et al.* 2011). Nanoporous carbon materials

with larger surface area, micro porous character and chemical nature of their surface have made them potential adsorbents for the removal of dyes from industrial waste water. The adsorptive properties of active carbon for removal of pollutants are well documented (Macias *et al.* 1993).

Many industries such as dyestuff, paper, textile and plastics use dyes in order to colour their products and also consume substantial amount of water (Elinge et al. 2011). Activated carbon adsorption has been cited by the US Environmental Protection Agency as one of the best available control technologies (Sivakumar et al. 2010). However, the preparation of carbon sorbents is generally energy consuming. Consequently, the commercially available products are fairly expensive. Since a large amount of carbon sorbent is needed for the removal of dyes from a large volume of effluent, the expenses involved hamper their application (Forgacs et al. 2004). Synthetic dyes and pigments are extensively used in dyeing and printing industries. Over ten thousand tons per year and approximately 10000 dyes are produced annually worldwide of which 10 % is lost in the industrial effluents (Kishore K Das et al. 2011). As synthetic dyes in wastewater cannot be efficiently decolorized by traditional methods, the adsorption of synthetic dyes on inexpensive and efficient solid supports was considered as a simple and economical method for their removal from wastewater. Adsorption is operative in most natural, physical, biological, and chemical systems and is widely used in industrial applications such as activated charcoal and synthetic resins. Carbons with excellent surface properties and specific functionalities should be developed to create a high affinity for adsorbate adsorption.

The use of carbon based on relatively expensive starting materials is also unjustified for most pollution control applications (Streat *et al.* 1995). Hence, widely available plant sources are utilized to reduce the concentration of dyes from wastewater and industrial effluents. In the present study, the physico-chemical and adsorption characteristics of nanoporous activated

carbon derived from *Ipomoea Carnea* stem waste have been measured and their capacity to remove commonly used synthetic dyes such as Direct Red 28 (Congo Red), Basic Violet 10 (Rhodamine B), Reactive Blue 4 and Acid Red 2 (Methyl Red) has been evaluated.

2. MATERIALS & METHODS

2.1 Adsorbent

Ipomoea carnea, the pink morning glory, is a species of morning glory. This flowering plant has stems and heart-shaped leaves that are a rich green and 6 to 9 inches long. It can be easily grown from seeds which are toxic and it can be hazardous to cattle. The stems are like hollow pipes. Another common name of this plant is "bush morning glory". Since this plant is abundantly available and has no specific significance, it can be utilized as an alternative low-cost adsorbent. The raw stems of Ipomoea Carnea were collected from in and around Trichy district, TamilNadu, India. They were cut into small pieces and dried in sunlight until the moisture was evaporated. The dried materials were used for the preparation of activated carbons using physical and chemical activation methods.



Fig. 1: Ipomoea Carnea

2.2 Adsorbates

The following four dyes were chosen for studying the adsorption capacities of the prepared nanoporous carbon materials.

2.3 Carbonization Procedures

Fundamentally there are two different activation methods for the preparation of activated carbon. They are physical and chemical activation methods respectively. The physical activation involves carbonization of precursor followed by activation of

the resulting char in the presence of activating agents such as steam or CO₂. But, the chemical activation involves the carbonization of the precursor in the presence of chemical agents.

In the chemical impregnation method, impregnation of precursor is done first with the particular chemical followed by carbonization and activation. Chemical activation is more advantageous than physical activation with respect to higher yield, more surface area and better development of porous structure (Rengaraj *et al.* 1998).

Table 1. List of dyes used for Adsorption study

S. No	Class	C.I. Name		рКа
1	Reactive dye	61205	Reactive Blue 4	5.2
2	Acid dye	13020	13020 Acid Red 2	
3	Direct dye	22120	Direct Red 28	4.2
4	Basic dye	45170	Basic violet 10	4.1

Table 2. List of various activation processes and abbreviation of activated carbon samples

S. No	Activated Carbon Sample	Treatment	
1	ICAC1	Acid process using H ₂ SO ₄	
2	ICAC2	Carbonate process using Na ₂ CO ₃ impregnation	
3	ICAC3	Chloride process using ZnCl ₂ impregnation	
4	ICAC4	Sulphate process using Na ₂ SO ₄ impregnation	
5	ICAC5	Dolomite process using CaCO ₃ impregnation	

2.3.1 Acid process with Sulphuric acid

The dried material was treated with excess of Sulphuric acid. Charring of the material occurred immediately with the evolution of fumes and heat. After the reaction subsided, the materials was left in air oven at 140-160 °C for a period of 24 hours. The dried masses were washed with excess of water to remove the free acid residues. They were dried at 110 °C sieved and finally activated at 800 °C (Karthikeyan *et al.* 2008).

2.3.2 Carbonate process with Sodium Carbonate

The materials to be carbonized were soaked with 10% solution of Na₂CO₃ for a period of 24 hours. After impregnation the liquid portion was decanted off and the material was dried. The dried mass was subjected to carbonization process at 400 °C, powdered well and thermally activated at 800 °C for a period of 10 minutes (Karthikeyan *et al.* 2008).

2.3.3 Carbonization with Zinc chloride

The materials to be carbonized were soaked with 10% solution of ZnCl_2 for a period of 24 hours. After impregnation the liquid portions decanted off and dried at 120 °C. The dried and neutralized mass was subjected to carbonization process at 400 °C, powdered well and thermally activated at 800 °C for a period of 10 mins (Jambulingam *et al.* 2007).

2.3.4 Carbonization with Sodium Sulphate

The materials to be carbonized were soaked with 10% solution of $\mathrm{Na_2SO_4}$ for a period of 24 hours. After impregnation the liquid portion was decanted off and dried at 120 °C. The dried and neutralized mass was subjected to carbonization process at 400 °C, powdered well and thermally activated at 800 °C for a period of 10 mins (Jambulingam *et al.* 2007).

2.3.5 Carbonization with Dolomite

The sufficient quantities of dried raw materials were taken over a Calcium Carbonate bed and the upper

layer of the waste was also covered with a layer of Calcium Carbonate. The whole material was carbonized at 400 °C and powdered well followed by the thermal activation at 800 °C . After the activation, the material was repeatedly washed with excess of water to remove Calcium Carbonate and dried at 110 °C (Karthikeyan et al. 2008).

2.4 Characterization of the Activated Carbon

The materials treated with Concentrated H₂SO₄, 10% Na₂CO₃, 10% ZnCl₂, 10% Na₂SO₄ and dolomite have been named as ICAC1, ICAC2, ICAC3, ICAC4 and ICAC5 respectively. The particle size of the well powdered carbon materials ranges from 75 to 180 μm. The physico-chemical properties of treated activated carbon were measured by suitable standard methods (Karthikeyan et al. 2008). The pH and conductivity were analyzed using Elico make pH meter (LI-120) and conductivity meter (M-180) respectively. Moisture Content (%) by mass, Ash Content (%) by mass, volatile matter (%), fixed carbon (%), Bulk Density (g/L), Specific gravity, Water soluble matter, Acid soluble matter, Iodine number (mg/g), Surface Area (m²/g), Phenol adsorption capacity and porosity were analyzed as per standard procedures (ISI, 1989). The Isoelectric point or Zero point charge (pH_{max}) of the carbon samples were measured by using the pH drift method (Jia et al. 2002). A known quantity of a series of ten NaNO, solutions having the initial pH values ranging from 1 to 10 were prepared using dilute HCl and dilute NaOH. All the ten NaNO₂ Solutions taken in ten different bottles were mixed with 0.5 g of Ipomoea Carnea Activated Carbon (ICAC) for a specified period of time. Then the solutions were filtered off and the activated carbon was separated. The final pH values of the ten solutions were measured and thereby calculation of pH was made by subtracting the initial pH values from final pH values. The graph was drawn by plotting the final pH values against pH. From the graphs plotted, the pH_{zpc} (point of zero charge) of the Nanoporous activated carbon was determined. Thus, the pH_{max} values of ICAC1, ICAC2, ICAC3, ICAC4 and ICAC5 were obtained and shown in table 3. The surface area

Parameters	ICAC1	ICAC2	ICAC3	ICAC4	ICAC5
рН	5.12	7.32	6.94	7.76	7.80
Conductivity, Ms/cm	0.26	0.39	0.33	0.41	0.10
Moisture Content, %	8.40	8.42	8.80	6.60	9.60
Ash Content, %	10.74	10.64	12.06	7.78	18.81
Bulk Density, g/L	0.40	0.39	0.32	0.37	0.39
Specific Gravity, S	0.83	0.83	0.63	0.92	0.93
Water Soluble Matter, %	0.35	0.87	0.44	0.59	0.84
Acid Soluble Matter, %	0.49	1.53	1.65	1.07	0.88
Zero Point Charge in pH Units	5.6	5.8	5.7	5.8	5.9
Porosity, %	51.81	53.01	49.21	59.78	58.06
Iodine Number, mg/g	521	571	199	655	728
Surface Area, m ² /g	544	607	200	698	746
Phenol Adsorption Capacity, mg/g	3.00	2.60	1.20	3.00	3.80
Volatile matter, %	9.20	11.2	18.8	14.8	17.0
Fixed carbon, %	82.40	80.2	72.4	78.6	73.4

Table 3. Physico-Chemical parameters of Activated Carbon from Ipomoea Carnea

of activated carbon was measured using BET equations at liquid N_2 temperature using Nova 1000, Quanta chrome Analyser.

2.5 Dye Adsorption studies

All reagents used were of AR –grade (E-merk). Different concentrations of individual dye solutions such as Acid Red 2, Direct Red 28, Reactive Blue 4 and Basic Violet 10 were prepared with distilled water. 50 ml of each chosen dye solution of known initial concentration (C_o)and initial pH was taken in a 100 ml screw-cap conical flask with a required amount of adsorbent and was agitated at a speed of 200 rpm in a temperature controlled thermodynamic shaker bath at 30 °C for a specified period of time. Then the solution was filtered through a 0.45 μ m membrane filter. The concentrations of dyes in solutions were determined

before and after adsorption using Elico UV-visible spectrophotometer. The amount of dye adsorbed and adsorption efficiency was calculated as follows:

Adsorption efficiency (%) =
$$((C_0-C)/C_0) \times 100$$

Where,

 C_0 = initial dye concentration (mg/l), C = residual dye concentration (mg/l).

3. RESULTS & DISCUSSION

3.1 Physico-chemical characteristics of Activated Carbon prepared from *Ipomoea Carnea* Stem Waste

The Physico-chemical parameters of chemically treated nanoporous activated carbon obtained from

Ipomoea Carnea stem waste were illustrated in table 3. The pH of the carbon derived from acid treatment was lower than other carbons impregnated with dolomite, Chloride, Sulphate and Carbonate salts. The higher pH values of ICAC5, ICAC4 and ICAC2 can be related to the introduction of some basic functional groups like carbonate ions and removal of acidic functional groups when compared with the lower pH value of acid treated carbon ICAC1. The results reveal that the dolomite treated and acid treated carbon samples have effectively adsorbed the basic dye. The Conductivity values indicated in table 3 do not show much variation which implies that the carbon samples were almost free from dissolved impurities. The cations in acidic carbon and anions in basic carbon are responsible for these similar conductivity values. It has been found that the pores of the carbon were completely available for the accumulation of adsorbate (Bansal et al. 1988; Bansal et al. 2002; Tripathi et al. 1996). Low moisture content of carbon indicates that the carbon samples have poor affinity on water. Even though moisture content of the carbon does not affect the adsorptive power, it dilutes the carbon which demands the use of additional weight of carbon during treatment processes (Bansal et al. 1988). The higher moisture content of dolomite treated carbon may be due to the moisture adsorptive nature of carbonate ions present in the activated pores of carbon particle.

He fixed carbon values of the prepared carbon samples obtained by using the values of moisture content, volatile matter and ash content indicate the presence of higher amount of fixed carbon though the ash content value of dolomite treated carbon was slightly higher than the carbons prepared by other activation processes. The ash content of the carbon samples substantiate that dolomite treated carbon has higher inorganic constituent than acid treated carbon. These values of ash content can be attributed to minimum amount of inorganic content and high fixed carbon. The carbon having little amount of ash content may remove the inorganic contaminants effectively (Bansal *et al.* 2002; Pereira *et al.* 2003). The Carbon of higher bulk densities are suitable for treatment

processes as the low density carbon suspends on water and the treatment processes cannot be effective. The lower bulk density values of carbons demonstrate that the carbon samples are highly branched and porous with more void space (Summers et al. 1988). Similarly, the lower bulk density values of carbons derived from *ipomoea carnea* also demonstrate that the carbon samples are highly branched and porous. Carbon prepared from the treatments with dolomite and Na₂SO₄ gives higher water soluble constituents which implies that the presence of water soluble impurities may have impact on the treated water. The values of water soluble matter and acid soluble matter are low in carbon prepared from acid treatment. This depicts that the acid treatment lowers the leachable constituents.

The isoelectricpoint or zero point charge implies that the prepared carbon is free from surface charges nearby neutral pH. Hence the carbon samples are suitable for the treatment of water even at the neutral medium. The higher level of iodine number reveals that the presence of high porosity in the carbon. The Carbon samples ICAC5 and ICAC4 are comparatively having higher porosity which highly influences the adsorption capacity of Nanoporous carbon samples. These results are supported by the surface area and phenol adsorption capacity of the carbon samples. Chemically modified activated carbon was found to be an excellent adsorbent for the basic and acidic dyes but direct dye showed less affinity for adsorption (Hirata et al. 2002). The usefulness of carbon is a result of their specific surface area. Activated Carbon ICAC5 having larger surface area removed all the four dyes effectively from their aqueous solutions.

3.1.1 Effect of surface area and surface chemical groups on the adsorption capacity: Removal of the dye Basic Violet 10 by prepared activated carbon samples

The effectiveness of removal of the dye Basic Violet 10 from aqueous solution by various chemically treated Nanoporous carbon samples is

ICAC5 > ICAC1 > ICAC4 > ICAC2 > ICAC3

The higher surface area of the dolomite treated carbon ICAC5 may perhaps be the reason for its greater affinity towards the adsorption of basic dye compared with Na₂CO₃ treated carbon ICAC2 which has comparatively lower surface area. The acid treated carbon ICAC1 also has greater adsorption towards the basic dye Rhodamine B, due to the presence of acidic functional groups on its surface. The similar results of basic dyes (Methylene Blue) carbonaceous material produced from coffee grounds depended upon the acidic polar groups on the adsorbent material (Hirata *et al.* 2002) also reported. Each low-cost adsorbent has its specific advantage in waste water treatment. For example clay minerals exhibit a strong affinity for basic dye (Bagane *et al.* 2000).

3.1.2 Removal of the dye Direct Red 28 by prepared activated carbon samples

The effectiveness of removal of the dye Direct Red 28 from aqueous solution by various chemically treated nanoporous carbon samples is

The Carbon ICAC5 having higher surface area removed the Direct Red 28 dye from aqueous solutions effectively. The adsorption of dyes is higher on activated carbon having higher value of surface area. The presence of surface polar groups on the adsorbent has greater influence on the adsorptive nature of the carbon (Amit Bhatnagar *et al.* 2006).

The Carbon ICAC4 having higher surface area but with bulky radicals like sulphate may decrease the adsorptive efficiency. The similar results were also reported by the removal of the dye Direct Congo Red by waste red mud (Namasivayam *et al.* 1998) a by-product of aluminium production with 90 minutes of equilibrium time, rice hull ash (Chou *et al.* 2001) and banana pith (Namasivayam *et al.* 1997).

3.1.3 Removal of the dye Reactive Blue 4 by prepared activated carbon samples

The effectiveness of removal of the dye Reactive Blue 4 from aqueous solution by the various chemically treated nanoporous carbon samples is

ICAC1 > ICAC5 > ICAC4 < ICAC2 < ICAC3

The carbon samples ICAC1 and ICAC5 demonstrate greater adsorption of the dye Reactive Blue 4. These two Carbon samples have greater affinity towards the removal of the anionic dye Reactive Blue 4. This is due to the presence of acidic functional groups in the voids of carbon ICAC1 and also due to the higher surface area and porosity of carbon ICAC5. Similar investigations reported the effect of modified carbon surface in the adsorption of three anionic reactive dyes remazol yellow, remazol black and remazol red from its aqueous solutions (Al – Degs et al. 2000). The recent reported adsorption capacity values 712.3, 278, 714 of Reactive red 2 (Chio et al. 2001), Reactive red 5 and Reactive orange 107 (Aksu et al. 2001) respectively with commercially activated carbon materials and adsorption of Reactive red 120, Reactive red 141 and Reactive red 2 (Netpradit et al. 2003) with low cost adsorbent yields the adsorption capacity values 48.31, 56.18, 62.5 respectively indicates the substantial removal of reactive dye with chemically modified surfaced carbon.

3.1.4 Removal of the dye Acid Red 2 by prepared activated carbon samples

The effectiveness of removal of the dye Acid red 2 from aqueous solution by the various chemically treated nanoporous carbon samples is

ICAC5 > ICAC4 > ICAC1 > ICAC2 > ICAC3

The carbon ICAC5 and ICAC4 exhibit the larger elimination of the dye acid red 2 from the aqueous solution due to their higher surface area and porosity.

This is also supported by their iodine number and phenol adsorption capacity.

Adsorption of acid dye by dolomite treated carbon ICAC5 having higher surface area reveals the presence of anionic surface groups (carbonate ions) on its surface. This is supported by its pH value. Due to the smaller surface area and lower iodine number of ZnCl₂ treated carbon ICAC3 shows lower adsorption capacity towards the adsorption of Acid dye. By the use of activated carbon, other components of the waste water can also be adsorbed by the carbon and the competition among the adsorbates can influence the dye binding capacity. The similar results were also reported that the percentage removal of the dye methyl red by the acid treated sugarcane baggase carbon was higher due to higher surface area and available adsorbent sites when compared to the formaldehyde treated sugarcane baggase carbon having comparatively lower surface area (Saiful Azhar et al. 2005).

3.2 Effect of pKa of dyes and pH_{mc} of adsorbent

Among the prepared carbon samples, ICAC5 (zpc value is 5.9) was chosen to study the adsorption

of various dyes. The adsorption of anionic dyes chosen increases when the solution pH is less than the isoelectric point (ZPC) of activated carbon as the surface of the carbon becomes more positively charged. Hence, the electrostatic attraction takes place between the anionic dyes and the positively charged sites on the carbon at lower solution pH. The adsorption of cationic dyes chosen increases when the solution pH is greater than the isoelectric point (ZPC) of activated carbon as the surface of the carbon becomes more negatively charged. Hence, the electrostatic attraction takes place between the cationic dyes and the negatively charged sites on the carbon at higher pH. The adsorption of anionic dyes such as Acid Red 2 (pKa = 5.1), Reactive Blue 4 (pKa = 5.2), Direct Red 2 (pKa = 4.1) decreases above their pKa values. The adsorption of cationic dye increases above its pKa value. The adsorption of the basic dye Rhodamine B (pKa = 4.2) starts increasing above its pKa value. At a pH of solution above the pH_{mc} of the adsorbent, the surface of sawdust is negatively charged and can attract cations from the solution and when solution $pH < pH_{mc}$, the surface of sawdust is positively charged and attractive to anions (Sharma et al. 2009; Sun et al. 2007).

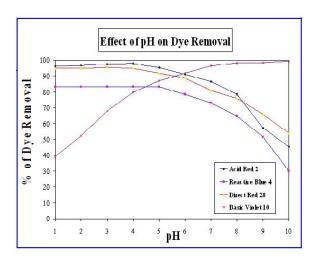


Fig. 2: Effect of pKa of dyes and pH_{zoc} of adsorbent on dye removal

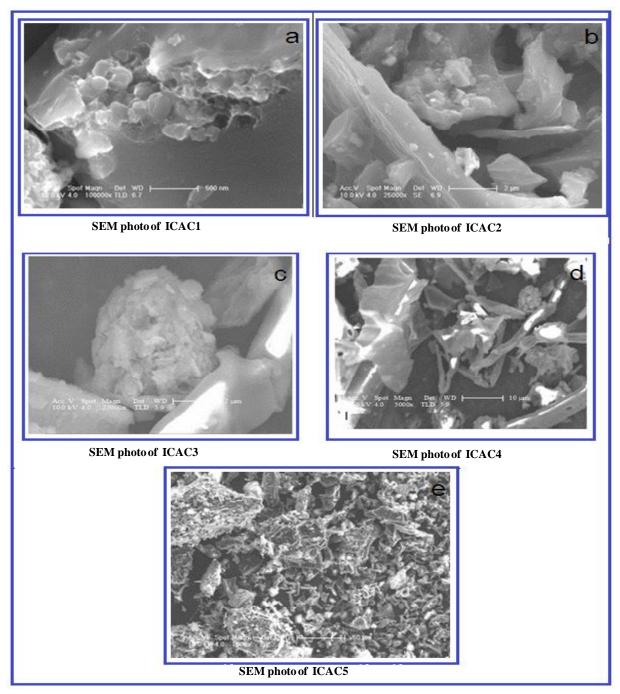


Fig. 3: SEM images of activated carbon prepared from Ipomoea Carnea waste materials

$$H_3C$$
 $COOH$
 $COOH$

Acid Red 2 (C.I. No.: 13020, F.W: 269, λmax 526 nm)

Direct Red 28 (C.I. No.: 22120, F.W:696, λmax 497 nm)

Reactive Blue 4 (C.I. No.: 61205, F.W:637, \(\lambda \) max 600 nm) Basic Violet 10 (C.I. No.: 45170, F.W:479, \(\lambda \) max 554 nm)

Fig. 4: Structure and characterization of dyes used for adsorption study

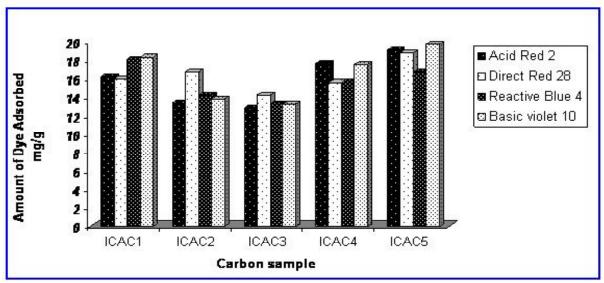


Fig. 5: Amount of various dyes adsorbed onto various activated carbon samples prepared from *Ipomoea Carnea* stem waste

3.3 Scanning Electron Micrograph studies

The morphology of the surface of the prepared Nanoporous activated carbon samples was examined using Scanning Electron Micrographs. These micrographs provide positive reception of the porosity of adsorbents and consequently a qualitative evaluation of their ability to adsorb the dye molecules in solution. ICAC5, ICAC4 and ICAC1 have many pores, small cavities and rough areas with micro pores which were clearly found on the surface. This shows that dolomite Na₂SO₄ and H₂SO₄ were effective to create well developed pores with uniform distribution on the surface of the precursor, therefore leading to the activated carbon with large surface area and porous structure.

4. CONCLUSION

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

1. Nanoporous activated carbons can be prepared conveniently and economically from *Ipomoea*

Carnea plant source which is abundantly present.

- 2. The prepared adsorbents have a substantial variation in the dye removal capacity. The adsorption capacity of carbon sample prepared by Dolomite process was much greater than the capacities of the other carbons prepared by various processes.
- 3. These carbon samples significantly remove the dyes from aqueous solutions based on their physico-chemical properties.
- 4. The extensive characterization studies of the different activated carbons prepared by various processes reveal that the carbons obtained from *Ipomoea Carnea* stem waste can be assessed as superior grade carbon for adsorption processes.
- 5. Activated carbon with good surface properties from various treatment procedures were identified. The surface chemical groups of the activated carbon play a vital role in removing variety of dyes from its aqueous solutions.

- 6. Dolomite and Na₂SO₄ treated activated carbons have very high surface area and porosity.
- 7. Porosity plays a vital role in determining the adsorption capacity of carbons.
- 8. The adsorption of anionic dyes increases when the solution pH is less than the isoelectric point (ZPC) of activated carbon as the surface of the carbon becomes more positively charged.
- The adsorption of cationic dyes increases when the solution pH is greater than the isoelectric point (ZPC) of activated carbon as the surface of the carbon becomes more negatively charged.
- 10. The adsorption of anionic dyes decreases above their pKa values.
- 11. The adsorption of cationic dyes increases above their pKa values.

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