

Novel Non-conventional Activated Carbon for the Remediation of Dyeing Industry Effluent

P.Sivakumar, P.N.Palanisamy, B. H. Hameed and K. Radha

Abstract—Activated carbon prepared from waste plants for the treatment of dyeing industry effluents has high significance in environmental sustainability and economic viability. The applicability of activated carbon prepared from *Euphorbia antiquorum* L wood impregnated with H_3PO_4 was analyzed for the removal of colouring matter present in the dyeing industry effluent. The mesoporous nature of the adsorbent was proved by the pore size distribution studies. Majority of the treated effluent samples were completely colourless and non-hazardous in nature. The optimum dose of adsorbent required varies from sample to sample depending upon the nature and concentration of pollutants present in the effluent. On the basis of the optimum dose of adsorbent required to treat 100 M³ of dye house effluent, the approximate cost of effluent was computed and compared with the commercial charcoal.

Keywords—Activated carbon, Adsorption, Charcoal, *Euphorbia antiquorum* L.

I. INTRODUCTION

TEXTILE wet processing is a highly water intensive operation. Various chemicals like starch, dye-stuff, auxiliaries, alkalis, acids, detergents etc., that are used in the processing contaminate the water and cause pollution in terms of BOD, COD, pH, TDS, color, hardness etc., in the discharged effluent [1]. The dye bearing wastewater is highly coloured and non-biodegradable which makes the water unfit for use. Recently many technologies like electrochemical coagulation, reverse osmosis, nano filtration, adsorption using activated materials etc., are used for the removal of dye from wastewater. Adsorption has been proposed as a feasible and economical process for the removal of dyes from wastewater. Adsorption was proved to be an efficient method for the removal of pollutants from the effluents into stable form [2].

In recent years, there has been growing interest in finding inexpensive and effective alternatives to the existing commercial activated carbon [3]. Exploration of good low cost and non-conventional adsorbent may contribute to the sustainability of the environment and offer promising benefits for commercial purpose in future. The costs of the activated carbon prepared from biomaterials are negligible when compared to the cost of commercial activated carbon.

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Some of the waste materials which are successfully employed for the manufacture of activated carbon in the recent past are, waste apricot [4], rubber seed coat [5], plum kernels [6], apricot shell [7], rice straw [8,9], bamboo [10], sunflower seed hull [11], agricultural waste [12] and rubber wood sawdust [13].

An attempt is made to prepare an activated carbon from *Euphorbia antiquorum* L wood by various physical and chemical activation processes. The physico-chemical characteristics of nine varieties of prepared activated carbon were published in our earlier publication [14,15]. The present work analyses the applicability of the *Euphorbia antiquorum* L activated carbon prepared by orthophosphoric acid impregnation method (EAAC) for the treatment of dyeing industry effluents.

II. MATERIALS AND METHODS

A. Preparation of Adsorbent

Euphorbia antiquorum L wood was used as precursor for the preparation of activated carbon. The wood was cut into pieces of 2 to 3 cm in size, dried in sunlight for 10 days. The dried material was then soaked in a boiling solution of 10 % H_3PO_4 for one h and kept at room temperature for 24 h. Thereafter, the wood material was separated, air dried at room temperature and carbonized in muffle furnace at 400°C for 20 min. The carbon was ground to powder and activated in a muffle furnace at 800°C for a period of 10 min. Then the activated carbon was washed with plenty of distilled water to remove residual acid, dried, sieved into 300 to 850 µm (20-50 ASTM mesh) size and stored in a tight lid container for further adsorption studies. The commercial activated charcoal was procured from Qualigens Fine Chemicals, Mumbai, India.

B. Characterization of Adsorbent

Physico-chemical characters of the activated carbon samples were analyzed as per the standard testing methods [16-18]. The morphological characteristics of the samples were studied using JSM-5610LV (JEOL - JAPAN) Scanning Electron Microscope (SEM). The pore characteristics of the activated carbon and N₂ adsorption-desorption isotherms were studied using ASAP-2020 Gas sorption analyzer.

C. Adsorption Studies of Effluent

Dyeing industrial effluent samples were collected from various dye processing industries in Perundurai SIPCOT industrial estate and industries around Tirupur (TN, India). The wastewater samples were collected immediately after the completion of dyeing process (at the outlet of dyeing machine). The samples were analysed for the physico-chemical characteristics as per the standard procedures [19-21]. Various methods employed for the analysis are given in Table 1.

Then 200 mL of the effluent was agitated with EAAC (depending upon the nature of effluent) for 180 min. The adsorbent dose is gradually increased until the maximum possible decolorization was achieved. After the attainment of maximum possible decolorization, the supernatant solution was separated using centrifuge and analyzed as before. The percentage of colour removal was calculated based on the absorbance (optical density) values at λ_{max} (maximum absorption wavelength) using Bio UV-Vis spectrometer (Elico make – BL 198). All the effluent samples were agitated with commercial charcoal by increasing the dosage gradually until the maximum possible decolorization was attained. The quantity of commercial charcoal is taken for cost calculation and the quality of the treated water was not analyzed.

TABLE I
METHOD OF ANALYSIS AND UNITS OF VARIOUS CHARACTERISTICS OF EFFLUENTS

S.No.	Parameter	Method of estimation	Unit of expression
1	pH	Using calibrated pH meter	--
2	Conductivity, $\mu\text{S}/\text{cm}$	Using calibrated conductivity meter	$\mu\text{S}/\text{cm}$
3	TDS	Gravimetric	mg/L
4	Total hardness	Volumetric (EDTA titration method)	mg/L in CaCO_3 equivalent
5	Alkalinity	Volumetric (Acid neutralization method)	mg/L in CaCO_3 equivalent
6	Chloride	Volumetric	mg/L
7	Sulphate	Gravimetric	mg/L
8	COD	Volumetric method with $\text{K}_2\text{Cr}_2\text{O}_7$	mg/L
9	BOD	Volumetric	mg/L

III. RESULTS AND DISCUSSION

A. Adsorbent Characteristics

The SEM image of EAAC (Fig.1) indicates that the activated carbon is highly branched and has many tubular pores in its structure. Various pore characteristics of the EAAC are presented in Table 2. The pore size distribution studies indicate that EAAC is mesoporous in nature. The mesopore volume was calculated by subtracting micropore volume from the total pore volume. Mesoporous carbon will adsorb more amounts of organic compounds (like dye molecules) through pore diffusion in its mesopores. Most of the commercial activated carbon used for the adsorption studies are mesoporous in nature. The Langmuir surface area is well in comparable limit with the surface area of the adsorbents reported in literature [22,23].

TABLE II
RESULTS OF N_2 ADSORPTION-DESORPTION ISOTHERM

S. No	Parameter	Value
1	Single point surface area ($P/P_0 = 0.21$), m^2/g	903.17
2	Langmuir Surface Area, m^2/g	1099.66
3	Total pore volume of ($P/P_0 = 0.99$), cm^3/g	0.4141
4	Micropore volume, cm^3/g	0.1778
5	Mesopore volume, cm^3/g	0.2363
6	Average pore width, \AA	20.554

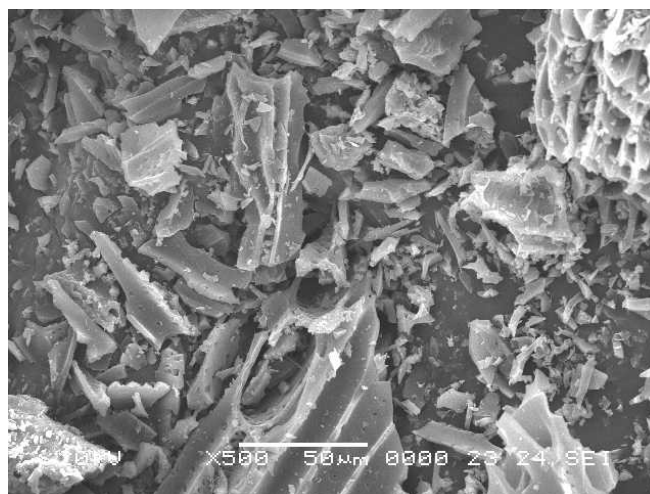


Fig. 1 SEM image of EAAC 500x

B. Characteristics of Effluent

All the effluent samples were brightly coloured and most of them are alkaline in nature. The applicability of EAAC for the treatment of dyeing industry effluent was evaluated by studying the colour removal capacity of the dye house effluent. As reported by Prakash et al.,[24] the percentage decrease in OD (optical density or percentage of light absorbance) was taken as the measure of reduction of the total dye concentration in the effluents and percentage decrease in COD as the measure of total organic constituents.

The results in the Table 3 to 7 show that 12 samples out of 16 samples analyzed were completely decolorized. The decrease in OD (Table 3) proves that more than 90% of the colouring matter is removed from the effluent.

TABLE III
PERCENTAGE OF COLOUR REMOVAL FROM DYE EFFLUENTS

Sample No.	Type of dye	λ_{\max} of the effluent (nm)	OD before treatment	OD after treatment	Percentage of colour removal
1	Reactive Red	520	0.239	0.003	98.74
2	Reactive Brown	416	0.948	0.033	96.52
3	Reactive Black	425	4.900	0.620	87.35
4	Reactive Violet	548	3.841	0.479	87.53
5	Direct Black	326	2.904	0.024	99.17
6	Direct Violet	526	3.676	0.092	97.50
7	Direct Brown	452	1.153	0.099	91.41
8	Direct Yellow	393	0.922	0.080	91.32
9	Basic Red	544	2.480	0.445	82.06
10	Basic Violet	556	2.141	0.310	85.52
11	Basic Green	427	0.359	0.024	93.31
12	Basic Blue	592	1.330	0.009	99.32
13	Acid Blue	593	0.249	0.015	93.98
14	Acid Yellow	394	2.110	0.088	95.83
15	Acid Red	494	1.560	0.088	94.36
16	Acid Orange	455	1.420	0.110	92.25

The properties of effluent samples before and after adsorption treatment are presented in Table 4 to 7. The samples 3, 4, 9 and 10 are reluctant for the colour removal using EAAC through adsorption. There are many factors responsible for the adsorption of colouring matter by an activated carbon [25]. In the samples 3, 4, 9 and 10 the TDS (Total Dissolved Solids) level is also very high. The reason for the poor adsorption may be the bulkiness of the dye molecule. Microporous and mesoporous adsorbents show poor adsorption of bulky dye molecules of $> 20\text{\AA}$ size, this bulkiness might be a reason for poor adsorption capacity. Other factors responsible for the poor adsorption may be high TDS (at high TDS more adsorbate will compete for the sorption sites) and poor affinity of dye molecule.

In general, the reactive dyes are reluctant for the adsorption with activated materials. Particularly violet, pink and black dyes are very hesitant for the adsorptive removal. The relationship between total dissolved solids and conductivity is a function of the type and nature of the dissolved cations and anions in the water and possible the nature of any suspended materials. In particular, the dyeing industry effluent contains organic dyes and some other non-ionic materials like micro textile fibres, urea, reducing agents, oxidizing agents, acetic acid, detergents and wetting agents [26].

Even though the samples 3, 4, 9 and 10 are not completely decolourised, the actual percentage of dye removal is more than 80%. The amount of activated carbon required for the treatment of effluent samples are given in Table 8. The samples 3, 4, 9 and 10 require slightly higher adsorbent dosage for the maximum decolouration. The amount of EAAC required was compared with commercial charcoal in order to carry out cost comparison analysis. For samples 1, 8, 9, 13, 15 and 16 the quantity of adsorbent required was same for EAAC and commercial charcoal. For other samples the EAAC requirement is higher than commercial charcoal.

The high levels of BOD indicate that there could be low oxygen available for living organisms in the wastewater when utilizing the organic matter present. High COD levels imply toxic condition and the presence of biologically resistant organic substances [27]. The COD and BOD decreased in accordance with colour removal. An excellent improvement was noticed in the treated effluent with

respect to the pH, COD and BOD. The low alkalinity value after treatment also substantiates the reduction of basicity of effluents.

Total Dissolved Solids are the important parameters for evaluating the suitability of effluent for irrigation purpose, since, these solids might clog both the soil pores and components of water distribution system [28,29]. The TDS value show a good improvement after the adsorption treatment, but it further requires some treatment to bring down the level of TDS within the WHO standard. This is true, as the samples 3, 6, 7, 8, 9, 10 and 11 TDS values are alarming ($> 10,000$ mg/L), even after the adsorption treatment.

Appreciable amount of chlorides and sulphates were removed by the EAAC. The samples 3, 6, 7, 8, 9, 10 and 11 had the chloride and sulphate levels are well above the permissible levels. High chloride contents are harmful for metallic pipes as well as for agricultural crops if such wastes containing high chlorides are used for irrigation purposes. Moreover, high chloride contents also kill some micro-organisms which are important in some food chains of aquatic life [30]. On the other hand the EAAC is an excellent adsorbent for the removal of majority of dyes in its aqueous solutions without any neutralization of the effluent. Still the effluent requires some other secondary treatment like RO (Reverse Osmosis) to remove the excess salts present in the treated effluent.

C. Cost Analysis

Table 8 shows the optimum dose of EAAC and commercial charcoal required for different effluents. The optimum dose of adsorbent required varies from sample to sample depending upon the nature and concentration of pollutants present in the effluent. On the basis of the optimum dose of EAAC required to treat 100 M^3 (0.1 million litre) of dye house effluent, the approximate cost of effluent was computed considering only the cost of the material used in the treatment process. For treating 100 M^3 of dyeing effluent, the optimum dose of EAAC, commercial charcoal and corresponding cost in US \$ are given in Table 8. The cost of EAAC preparation is 1.75 US \$ per kg and commercial charcoal is procured at 3.25 US \$ per kg.

All the effluent samples except 1, 8, 9, 13, 15 and 16 require more amount of EAAC than the commercial charcoal. Even though some samples require high amount of EAAC, on the basis of cost involved

for the treatment of 100 M³ of effluent, the adsorption using EAAC is more economical than commercial charcoal.

TABLE IV
PHYSICO-CHEMICAL CHARACTERISTICS OF REACTIVE DYE EFFLUENT SAMPLES

Colour & Appearance	Sample Number							
	1		2		3		4	
	BT	AT	BT	AT	BT	AT	BT	AT
	Purple Red	Colourless	Brown	Colourless	Black	Light Black	Violet	Light Violet
pH	7.35	7.29	7.98	7.5	9.93	7.92	10.2	7.97
Conductivity, $\mu\text{S}/\text{cm}$	6.94	4.48	8.39	5.72	117.5	56.5	14.7	6.11
TDS	5,000	3,000	6,200	4,200	98,400	40,200	10,000	5,600
Total hardness	1,155	320	1,077	445	1,136	540	1,096	290
Alkalinity	950	220	2250	275	4,600	455	7,450	330
Chloride	665	310	665	410	2,825	2,210	800	665
Sulphate	400	250	800	400	3,450	1,250	900	760
COD	1,560	80	2,080	90	8,400	600	1,050	290
BOD	410	10	610	15	2,850	80	335	55

TABLE V
PHYSICO-CHEMICAL CHARACTERISTICS OF DIRECT DYE EFFLUENT SAMPLES

Colour & Appearance	Sample Number							
	5		6		7		8	
	BT	AT	BT	AT	BT	AT	BT	AT
	Bluish Black	Colourless	Violet	Colourless	Brown	Colourless	Yellow	Colourless
pH	7.55	7.15	10.16	8.08	10.2	8.07	9.9	8.66
Conductivity, $\mu\text{S}/\text{cm}$	7.86	5.17	55.4	19.2	32.2	19.5	31.4	22.7
TDS	6,800	3,000	36,600	10,900	18,800	11,800	20,800	11,600
Total hardness	1,097	520	1201	440	1,154	355	1,070	660
Alkalinity	1,600	245	6,450	255	7,800	280	2,010	545
Chloride	550	355	2,120	1,540	1,420	945	1,910	775
Sulphate	950	350	2,550	1,100	1,900	1,200	2,010	1,110
COD	840	160	2,980	220	1,950	245	2,660	410
BOD	260	15	815	40	585	10	1,850	120

TABLE VI
PHYSICO-CHEMICAL CHARACTERISTICS OF BASIC DYE EFFLUENT SAMPLES

	Sample Number							
	9		10		11		12	
	BT	AT	BT	AT	BT	AT	BT	AT
Colour & Appearance	Purple	Light Purple	Violet Blue	Mild Violet	Green	Colourless	Blue	Colourless
pH	10.23	7.95	10.55	8.35	10.62	8.32	10.2	8.05
Conductivity, $\mu\text{S/cm}$	45.5	33.1	49.3	16.3	30.2	17.9	27.7	17.2
TDS	38,800	19,600	49,200	14,600	29,000	15,600	24,000	8,400
Total hardness	1,125	510	1,260	310	1240	360	995	540
Alkalinity	7,550	310	8,100	300	8,200	240	41,060	770
Chloride	2,025	1,800	2,345	1,350	1,110	545	2,210	1,440
Sulphate	2,850	1,000	3,150	1,540	1,450	750	1,270	790
COD	3,084	350	5,400	350	2,390	120	1,900	210
BOD	950	65	1450	75	780	20	985	45

TABLE VII
PHYSICO-CHEMICAL CHARACTERISTICS OF ACID DYE EFFLUENT SAMPLES

	Sample Number							
	13		14		15		16	
	BT	AT	BT	AT	BT	AT	BT	AT
Colour & Appearance	Blue	Colourless	Yellow	Colourless	Red	Colourless	Orange	Colourless
pH	7.59	7.35	7.54	7.22	8.04	7.71	8.33	7.57
Conductivity, $\mu\text{S/cm}$	7.74	4.92	5.41	3.36	8.32	7.4	11.2	6.15
TDS	5,400	4,400	3,700	1,800	9,200	4,000	9,700	3,100
Total hardness	1,058	426	612	400	728	600	1,055	395
Alkalinity	1,700	250	410	210	820	465	1,220	775
Chloride	580	290	665	300	970	545	1,050	665
Sulphate	650	420	600	290	1,200	815	900	370
COD	1,120	120	290	140	1,050	340	2,050	170
BOD	360	20	190	20	370	60	415	70

BT – Before Treatment & AT – After Treatment

IV. CONCLUSION

The pore distribution characteristics of EAAC substantiates that it is mesoporous in nature. The carbon (EAAC) is an excellent adsorbent for the removal of majority of dyes in its aqueous solutions. The treated effluents (12 out of 16 samples analyzed) are

completely colourless (more than 90 % of colour removal). The amount of COD and BOD of the treated effluent indicates that the treated water is fit for further use. On the basis of the optimum dose of EAAC required to treat 100 M³ of dye house effluent, the approximate cost of effluent was computed and the cost of treatment with EAAC is much more economical than commercial charcoal.

TABLE VIII
COST COMPARISON FOR THE TREATMENT OF 100 M³ OF EFFLUENT

Sample No.	EAAC		Commercial charcoal	
	Amount of carbon required (kg)	Cost of treatment in US \$	Amount of carbon required (kg)	Cost of treatment in US \$
1	50	87.5	50	162.5
2	100	175	50	162.5
3	350	612.5	250	812.5
4	250	437.5	150	487.5
5	100	175	50	162.5
6	250	437.5	150	487.5
7	150	262.5	100	325
8	150	262.5	150	487.5
9	200	350	200	650
10	400	700	300	975
11	150	262.5	100	325
12	250	437.5	200	650
13	50	87.5	50	162.5
14	100	175	50	162.5
15	50	87.5	50	162.5
16	50	87.5	50	162.5

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