

## Preparation and Characterization of Activated Carbons prepared from Putrescible Vegetable Waste for the Removal of Colour form Textile Effluent

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**Abstract:** In the present study an attempt has been made to prepare activated carbons which are capable of adsorbing a wide range of organic substances, oxidizing materials and certain heavy metals from liquids and water. Various physical and chemical activations were done to prepare activated carbons from putrescible vegetable waste which were collected from kitchens of houses, hotels, marriage halls and farmer shandy. Characterization studies like pH,  $pH_{ZPC}$ , electrical conductivity, moisture content, ash content, volatile content, fixed carbon, bulk density, specific gravity, porosity, surface area, methylene blue number and iodine number were done. Surface area analysis was done using BET isotherm under  $N_2$  atmosphere. Surface morphology was studied using SEM and SEM EDX. Finally it was concluded that the carbon (PAC) prepared from  $H_3PO_4$  treatment to be the best activated carbon which could be employed for the adsorption of color from dyeing industry effluent water. Batch mode adsorption study was performed and the effluent analysis was done before and after treatment. It was obvious from the results that PAC could be employed effectively for the treatment of dyeing industry effluent water.

**Keywords:** Activated carbon, solid waste, putrescible vegetable waste, BET isotherm, Adsorption.

### Introduction

Due to massive Industrialization and urbanization the development in printing, dyeing and textile industries has increased multifold. The effluent released proves to be highly hazardous<sup>1</sup>. The untreated effluents cause drastic reduction in the quality of water in the receiving body since they possess strong colors, high pH and large amount of suspended solids, TDS, BOD and COD. Thousands of dyes weighing approximately 0.7 million tons are produced annually for various industrial processes<sup>2</sup>.

Various methods are employed for the removals of dyes from wastewaters include anaerobic decolourisation<sup>3</sup>, chemical oxidation<sup>4</sup>, reverse osmosis<sup>5</sup>, ion exchange<sup>6</sup> and biological treatment<sup>7</sup>. These physico chemical methods are less efficient, expensive and produce wastes which are difficult to dispose. Among these methods, adsorption<sup>8</sup> onto activated carbon has been proven to be the most effective method for the color removal in terms of initial cost, simplicity of design, easy operation and insensitivity to toxic substances<sup>9</sup>.

Vegetable wastes that have been generated from hostels, hotels, marriage halls, etc., were mostly thrown out into open spaces. Many research works has been carried out for the proper disposal of vegetable wastes into value added products<sup>10</sup>. Certain processes like vermicomposting<sup>11</sup>, incineration<sup>12</sup> and anaerobic

digestion<sup>13</sup> were carried out for the conversion of vegetable wastes into useful products. Also conversion of vegetable residues into bio-adsorbents for waste water treatment was also suggested<sup>10</sup>. Activated carbon is a micro porous inert carbon with a large internal surface (up to 1500 m<sup>2</sup>/g). Activated carbon is capable of adsorbing a wide range of organic substances, oxidizing materials (such as chlorine and ozone) and certain heavy metals from liquids and water.

In the present study an attempt has been made to convert Putrescible Vegetable Waste (PVW) into activated carbons. Among the carbons, the best activated carbon was selected and it was employed for the removal of colour from textile dye effluent water.

## Experimental

### Effluent Sample Collection:

Water samples were collected (5 samples) from the Common Effluent Treatment Plant (CETP) at Tiruppur, Tamil Nadu, India at five different timings. PAC prepared was used for the treatment of various effluent samples, collected by batch mode adsorption studies. Effect of adsorbent dosage and effect of time were studied using the best activated carbon.

### Adsorbent:

### Collection of Raw Material:

Activated carbons were prepared using putrescible vegetable waste (PVW) which was collected from market places, kitchens of college hostels and marriage halls in and around Coimbatore city, Tamilnadu, India throughout the year 2010. During the one year period all types of vegetable wastes used in the region and their peel off were collected.

### Carbonization Procedures:

#### Direct Pyrolysis:

The dried PVW was subjected to carbonization 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 and dried to obtain thermal activated carbon (TAC).

#### Carbonization with H<sub>3</sub>PO<sub>4</sub>:

The dried PVW to be carbonized is impregnated with a boiling solution of 30 % H<sub>3</sub>PO<sub>4</sub> for 2 hours and soaked in the same solution for 24 hours<sup>14</sup>. At the end of 24 hours, the excess solution decanted off and air dried and was carbonized in muffle furnace at 400°C. The dried material was powdered and activated in a muffle furnace at 800°C for a period of 10 minutes. Finally the activated carbon (PAC) was washed with plenty of water to remove residual acid and then it was dried and powdered.

#### Carbonization with H<sub>2</sub>SO<sub>4</sub>:

The dried material was treated with 1:1 sulphuric acid solution<sup>15</sup>. Charring of the material occurred 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° C and was carbonized in muffle furnace at 400°C. It was finally activated at 800°C in muffle furnace for 10 minutes. Thus sulphuric acid treated activated carbon (SAC) was prepared.

#### Carbonization with ZnCl<sub>2</sub>:

The material to be carbonized is impregnated with a boiling solution of 10 % ZnCl<sub>2</sub> for 2 hours and soaked in the same solution for 24 hours<sup>16</sup>. At the end of 24 hours, the excess solution decanted off and air dried. The material was carbonized in muffle furnace at 400°C. The dried material was powdered and activated in a muffle furnace at 800°C for a period of 10 minutes. The activated carbon (ZAC) thus obtained was washed with plenty of water, dried and powdered.

### Carbonization with NaOH and KOH:

The material to be carbonized is impregnated with NaOH/KOH employing several conditions. Impregnated with various concentrations (2%, 5%, 7.5% and 10%) boiling NaOH/KOH for 2 hours and soaked in the same solution for 24 hours. At the end of 24 hours, the excess solution decanted off and air dried. The material was carbonized in muffle furnace at 400°C. It was observed that the particle size of the carbon was less than 10µm which could not be employed for adsorption process. On further activation it turned into a gum-like substance and hence this could not be employed as an activated carbon.

### Batch Mode Adsorption Studies:

Batch mode adsorption study was carried out by taking 50 ml of dye solution and required amount of activated carbon in 250 ml conical flask. Dye solutions were agitated at 120 rpm in mechanical shaker for different time intervals. The effluent and adsorbent were separated by centrifugation at 1000 rpm for 5 minutes. Effect of adsorbent dosage was carried out and the effluent samples were collected and stored for analysis.

## Results and Discussion:

### Physico-Chemical Characteristics Of Activated Carbons:

The characteristics of the activated carbons prepared from putrescible vegetable waste were listed in Table 1. Moisture content of the carbon ZAC was found to be 10% which is higher than other carbons. Even though higher 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 process<sup>17</sup>. Moisture content of the carbons PAC, SAC and TAC were comparable with the results of earlier researchers<sup>18,19</sup>.

**Table 1 Physico – Chemical parameters of PAC, ZAC, SAC and TAC**

S.No.	Parameters	PAC	ZAC	SAC	TAC
1	Moisture Content (%)	4.5	10	3.5	5
2	Ash Content (%)	8.37	8.89	12.43	7.58
3	Water soluble content (%)	1.926	7.1	4.5	4.2
4	Acid soluble content (%)	1.63	2.67	4.15	2.95
5	Volatile matter (%)	20.5	16.4	26.2	18.9
6	Fixed carbon (%)	66.63	64.71	57.87	68.52
7	Iodine number (mg/g)	1101.9	954.3	763.9	692.7
8	Methylene blue number	525	354	150	178.5
9	Bulk density (g/ml)	0.388	0.346	0.682	0.362
10	Specific gravity	0.98	0.86	0.892	0.75
11	Porosity	60.4	59.76	23.5	51.7
12	pH	6.65	6.75	6.9	7.81
13	pH <sub>ZPC</sub>	6.97	7.5	6.4	7.96
14	Electrical conductivity	0.151	0.260	0.315	0.274
15	Yield (%)	76.48	53.8	50.8	38.4
16	BET Surface area (m <sup>2</sup> /g)	603.7	401.9	105.5	23.58
17	Total pore volume (cm <sup>3</sup> /g)	0.7053	0.3141	0.0137	0.0014
18	Average pore diameter (A <sup>0</sup> )	2396	522.7	46.74	31.26
19	Langmuir surface area (m <sup>2</sup> /g)	2469	840.6	130.95	100.6

Ash content of an activated carbon gives an indication about the amount of inorganic constituents present in the carbon. Among the four carbons ash content percentage was higher in SAC and lower in TAC. More ash content may be incorporated by the activating agents and it ultimately reduces the fixed carbon percentage. It was evident from the Table 1 that water soluble and acid soluble matters were low in PAC than other carbons<sup>20</sup>.

Volatile matter in all the carbons was found to be above 16%. It is evident that activated carbon derived from most of the naturally occurring plants and agro products have high percentage of volatile matter. Fixed carbon was found to higher in TAC and PAC compared to other two carbons.

From Table 1, it was clear that PAC has higher Iodine number, indicating that it is best suitable for dye removal. Methylene blue number indicates the adsorption power of carbon to adsorb high molecular weight substances. If it is greater than 400 then it indicates that the carbon is good for dye adsorption. Among the carbons PAC has got higher methylene blue number.

Porosity is the main factor for increasing the adsorptive power of an activated carbon<sup>21</sup>. Porosity is related to the bulk density and specific gravity of activated carbon. Bulk density indicates the fiber content of the carbon. Porosity was greater for PAC compared to other carbons.

pH values are near neutral which will be helpful for the treatment of all classes of dye waste water and the carbon can also be used for drinking water purification. Conductivity values do not show much variation, this means that cations in acidic carbons and anions in basic carbons may be responsible for the conductivity.

Point of zero charge ( $pH_{ZPC}$ ) is of fundamental importance in surface science. The  $pH_{ZPC}$  in physical chemistry is a concept relating to the phenomenon of adsorption, and it describes the condition when the electrical charge density on a surface is zero<sup>22</sup>.  $pH_{ZPC}$  is near to neutral for PAC, acidic for SAC and basic for ZAC and TAC. The value for  $pH_{ZPC}$  of PAC confirmed that its surface is neutral which could attract both anionic and cationic dyes<sup>23, 24</sup> whereas SAC could attract only anionic dyes and other two carbons attract only cationic dyes.

### Scanning Electron Microscope and Elemental Analysis:

Examination of the SEM and SEM EDX analysis of the activated carbon particles show smooth areas having long ridges and rough areas with mesopores and more number of edges. An electron microscope study reveals highly porous and branched particles particularly in PAC and TAC. Small cavities, pores and more rough surfaces on the carbon indicate the presence of interconnected porous network. Tubular pores and cavities will increase the surface area of the adsorbent. SEM images of all the activated carbons were shown in Figure 1.

SEM EDX was also carried out to find out the elements that are present in the carbons. The results were presented in Table 2. It was found that the carbon percentage was higher in TAC compared to other carbons. In SAC and TAC elements like calcium, magnesium and potassium were present in few percentages.

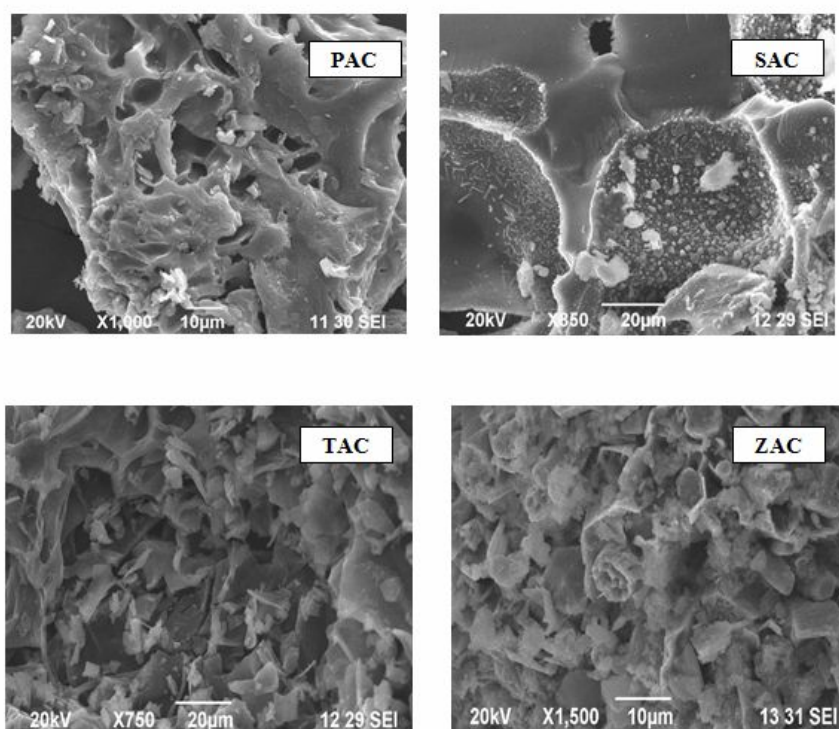


Figure 1 SEM images of Activated carbons

**Table 2 Elements and their weight percentage present in the activated carbons through SEM EDX analysis**

Elements	Weight %			
	PAC	ZAC	SAC	TAC
Carbon	49.76	59.46	47.25	68.6
Oxygen	37	31.67	36.9	24.34
Phosphorus	13.09	1.98	1.19	1.74
Sulphur	0.15	0.31	5.83	0.37
Magnesium	-	-	0.6	1.16
Potassium	-	-	7.19	2.28
Calcium	-	-	1.04	1.51
Zinc	-	6.06	-	-
Chlorine	-	0.52	-	-

**BET Analysis:**

For all the carbons the specific surfaces area determined by BET, total pore volume and average pore diameter were summarized in Table 3. From the table it could be seen that the BET surface area, micro pore surface area and micro pore volume of the PAC were all higher than those of all the carbons. Pore size distribution (PSD), a very important property of adsorbents, determines the fraction of the total pore volume accessible to molecules of a given size and shape<sup>25</sup>. It was possible to demonstrate that the porosity of activated carbons could be controlled by using different activation agents<sup>26, 27, 28</sup>.

**Table 3 BET surface area and porosity studies for the activated carbons**

Parameters	PAC	ZAC	SAC	TAC
BET Surface area (m <sup>2</sup> /g)	603.7	401.1	105.5	23.58
Langmuir Surface area (m <sup>2</sup> /g)	2469	840.6	139.5	10.06
Total Pore Volume (cm <sup>3</sup> /g)	0.7053	0.3140	0.01379	0.01412
Average Pore Diameter (A <sup>0</sup> )	46.74	31.26	52.27	21.396 X 10 <sup>3</sup>

**Treatment of Textile Dye Effluent:**

The main purpose of effluent analysis is to evaluate better – performing, advanced treatment methods with the aim of reuse, recovery of valuable products or safe disposal. On the other hand, this helps to evaluate the efficiency of the treatment system by analyzing the pollutant level remaining in the treated effluent. Above all, the quality of water should be known prior to its release into the environment<sup>29</sup>.

The effluent parameters before and after treatment are presented in Table 4. From the table it is clearly understood that PAC completely removes the colour from the samples 1, 4 and 5. pH of the samples determine whether the sample is acidic or alkaline in nature. It is clear from the table that before treatment they were highly alkaline in nature. After treatment with PAC it was seen that sample 1 and 4 are slightly alkaline, sample 3 and 5 are slightly acidic and sample 2 is acidic in nature.

Turbidity is measured using Nephelometric turbidity meter and in all the samples turbidity is reduced to a greater extent<sup>30</sup>. Biological oxygen demand (BOD) and Chemical oxygen demand (COD) are the indicators of excess organic matter and chemical compounds in water respectively. The maximum permissible limit of BOD is 30ppm. After treatment in the samples 1, 2 and 4 the BOD is not detectable and in samples 3 and 5 minimum BOD is observed<sup>31</sup>. Before treatment COD is higher in all the samples and after treatment there was a considerable decrease in COD.

An appreciable decrease in the total dissolved solids in all the samples was observed. Total hardness in any water sample reflects the nature of all geological formations with which it has been in contact. The limit of hardness should not exceed 600 for any water sample<sup>32</sup>. In all the samples initially the total hardness level was higher and after treatment with PAC it decreased to a greater extent.

The presence of calcium in water sample supplies results in over deposits of limestone, dolomite and gypsum. It contributes to the total hardness. The calcium level should be within 75 – 200 mg/l. In all the

effluent samples, the calcium level was well below the lower acceptable limit. Similar analysis of calcium was done by Harinath<sup>33</sup> and was reported to have higher levels of calcium.

**Table 4 Characteristics of effluent samples before and after treatment**

Parameters	Sample 1		Sample 2		Sample 3		Sample 4		Sample 5	
	BT	AT	BT	AT	BT	AT	BT	AT	BT	AT
Colour	Blackish green	Clear	Dark blue	Pale grey	Reddish black	Pale red	Black	Clear	Dark green	Clear
pH (at 25 <sup>0</sup> C)	10.3	8.5	9.6	3.94	11.1	6.78	10.8	7.6	11.7	6.62
Turbidity (NTU)	34	7	45	4	38	4	51	3	28	ND
BOD (20 <sup>0</sup> )C, 3 days, ppm)	28	ND	45	ND	52	12	32	ND	41	5
COD (ppm)	860	72	580	55	430	45	400	50	345	42
TDS (mg/L)	4110	524	5274	410	5040	356	2727	186	2012	184
Total hardness (ppm)	1200	156	799	76	656	80	645	42	384	44
Ca <sup>2+</sup> (ppm)	240	44	348	40	251	44	300	30	224	30
Mg <sup>2+</sup> (ppm)	219	36	546	35	325	36	181	10	204	12
Total alkalinity(ppm)	950	242	1124	180	786	211	815	174	642	80
Chloride (mg/L)	622	181	220	60	408	60	392	80	361	60
Sulphate (mg/L)	1080	534	612	15	400	10	128	5	135	4

Magnesium is a common constituent of natural water and an important contributor to the hardness of water. It forms scales in boiler when water containing magnesium is heated. It should be within the limit of 30 – 150 mg/l. It was observed that all the samples exhibit lower magnesium level nearing the acceptable limit as per BIS<sup>34</sup> (1991).

Chloride content is lower even before the treatment and it became much lower after treatment with PAC<sup>35</sup>. Sulphate content considerably decreased after treatment except sample 1<sup>36</sup>. Alkalinity of all the samples was found to be within the limits after treating them with the activated carbon.

From all the above analysis, the effluent quality was found to satisfy the water quality parameters as per the BIS (1991) standard. Hence it could be proved that phosphoric acid activated carbon (PAC) prepared from putrescible vegetable waste (PVW) is an effective adsorbent which removes pollutants like dye content from dyeing industry effluents. Further some secondary treatment methods could be adopted to improve the quality of the effluent.

## Conclusion

PVW was converted into activated carbon using carbonization techniques like treatment with H<sub>3</sub>PO<sub>4</sub>, H<sub>2</sub>SO<sub>4</sub>, ZnCl<sub>2</sub> and pyrolysis. Iodine number, methylene blue number and fixed carbon were higher for phosphoric acid treated activated carbon. Low moisture content was observed for PAC. SEM images show pores on the surface of the carbons. SEM EDX concluded that the carbon percentage was higher in PAC. BET surface area analysis revealed that PAC possessed high surface area of 603.7m<sup>2</sup>/g. Pore volume and pore diameter were also higher in PAC than other carbons. The physico-chemical parameters thus revealed that PAC was the best activated carbon among the four carbons. Effluent samples were collected from an effluent treatment plant and through batch mode adsorption study it was treated. The effluent parameters before and after treatment were analyzed for their quality. It was found that there was a tremendous improvement in the properties like turbidity, colour, BOD, COD, total dissolved solids, total hardness and alkalinity. Hence PAC prepared from putrescible vegetable waste could be an effective adsorbent for the textile dye effluents.

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