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RESEARCH ARTICLE

PREPARATION AND CHARACTERIZATION OF ACTIVATED CARBON FROM USED *TEA DUST* IN COMPARISON WITH COMMERCIAL ACTIVATED CARBON

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ABSTRACT

In the present study an attempt was made to prepare, characterise and test Activated Carbon using H_3PO_4 as activating agent and its efficiency in chromium removal from tannery effluent. Characteristics viz. pH(7.06), Bulk Density (0.8 g/ml), Ash (12.4%), Conductivity (0.025µs), BET Surface Area (280.39m^2/g), Moisture (7.2%) and percentage of carbon(73%) and oxygen (20%) content are compared with commercially available activated carbon. Activated carbon prepared from used tea dust and commercial, are characterised by XRD, SEM and EDX. The activated carbon both prepared and purchased, are evaluated for their chromium removal efficiency and found that activated carbon prepared from used tea dust is about 70% efficient when compared to commercially available activated carbon and thus present study adds one more bio-waste source as useful raw material for preparation of activated carbon.

INTRODUCTION

The useful properties of activated carbon have been known since ancient times. Today, activated carbons are useful in wide variety of applications such as decolourization, decontamination, respirators for protection against toxic gases, as desiccants and or medicinal agents in detoxification (Austin *et al*., 1984 and Sisca *et al*., 2009). Commercially available activated carbons are still expensive due to the use of non-renewable and relatively high-cost starting material such as coal (Sourja *et al*., 2005 and Martin *et al*., 2003). All attempts have been made to prepare activated carbon from organic precursors such as bamboo, coconut shell, sawdust, seeds and wood and similar agro wastes and biomaterials (Kumamoto *et al*., 1994 and Bhattacharya *et al*., 2004). Biomass waste including corn cob (Hiremath *et al*.,2012).,coconut shell (Hu, 1999), coconut tree sawdust (Kadirvelu *et al*.2000), palm shell (Adinata *et al*.,2007), apple pulp(Suairez-Garcıia, 2002), chickpea husks (Hayashi, 2000), grain sorghum (Diao, 2002), pistachio nut shell(H.Dolas *et al*.,2011), jute fiber (Senthilkumaar, 2005), olive stones and walnut shell (Martinez, 2006), cherry stones and Apricot stones and Grape seeds (Nartsislav Petrov), coir pith (Namasivayam and Kadirvelu, 1997), rice bran (Suzuki, 2007), cassava peel (Sudaryanto *et al*., 2006), gopher plant (Ozgul, 2007), jackfruit shell waste (Prahas, 2008), oil palm shell(Azreen *et al*.,2012), rubber tree seed coat (Hameed, 2008), cotton stalk (Deng, 2009), flamboyant (Hu M. R., 2009),Palmyra tree leaves, inflorescence and fruit nutshell waste have been found to be suitable precursors owing to their high carbon and low ash contents (Banerjee, 1985 and Kadirvelu, 2000).Several activating agents have been tried such as phosphoric acid, zinc chloride and alkaline metal compounds (Srinivasakannan, 2004). Phosphoric acid activation has been applied on a wide variety of cellulosic precursors such as coconut shell (Laine *et al*.1989 and 1992 and Kirubakaran *et al*.1991), peach stones (Molina-Sabio M *et al*.1995 and 1996), cotton stalks(Girgis *et al*.1999), shells of nuts like almond, pecan, English walnut, black walnut and macadamia nut (Ruiz Bevia *et al*.1984, Toles *et al*.1998 and 1999 and Dastgheib *et al*.2001), acorns and olive seeds(Lafi *et al*.2001), Peanut hull(Girgis *et al*.2002) and sugar cane bagasse(Ahmedna *et al*.2000).The carbon derived from the *Delonix regia* pods used as an adsorbent for the removal of toxic Cr (VI) from aqueous solution In the present study an attempt was made to prepare and characterise Activated Carbon using H_3PO_4 as activating agent and compare with commercially available activated carbon.

MATERIALS AND METHODS

Raw material used for preparation of activated carbon in the present study is *used tea dust,* a waste biomass. Used tea dust was obtained locally from the tea stall, which were otherwise disposed as solid municipal waste. Tea dust as a precursor material was washed till the colour disappears. The washed materials were dried at room temperature and the dried material

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was transferred in to a 500 ml beaker for boiling. The material was boiled at 60ºc for 15 minutes twice or thrice till the pH of the material shows neutral and become colourless. The boiled material was dried and kept in the hot air oven at 100ºC for 6 hours. The dried material was soaked in concentrated orthophosphoric acid for 24 hours (1:1 ratio). At the end of 24^{th} hour, the product was washed with excess of water to remove free acids till the pH become 7.0.The washed material was dried at room temperature. Then the dried material was kept in a muffle furnace for thermal activation at 600°C for a period of half an hour. Then the material is cooled at room temperature, powdered and stored in an airtight container for further testing (Ahamed, 2008).

Analysis of physico-chemical characteristics

pH determination, Electric Conductivity, bulk density, Ash content, Moisture content of both prepared and commercially obtained activated carbon were done by themethod described by Sugumaran (2012). Scanning electron microscopy (SEM) analysis was carried out on the activated carbon prepared under optimum conditions, to study its surface texture and the development of porosity. SEM samples of the aqueous suspension of AC were fabricated by dropping the suspension on to clean electric stubs and allowing water to completely evaporate. SEM observations were carried out on a Hitachi, model: S-3400N Electron microscope at 15.0kV (CIF, Pondicherry University). The surface areas measurements (m^2/g) of the activated carbon samples were made by low temperature nitrogen adsorption, by BET equation, using Micrometrics (ASAP 2010) (CIF, Pondicherry University).

RESULTS AND DISCUSSION

Activated carbons have been used for a long time as adsorbents in many applications in which impurities and contaminations in low concentration are removed. The adsorption capacities of activated carbons (ACs) strongly depend on their porosity and surface area. Since the textural properties of ACs depend on the starting material and the preparation method, many natural and synthetic materials have been used as precursors. ACs with high surface area and pore volumes can be prepared from a variety of carbonaceous materials such as coal, coconut shell, wood, agricultural wastes or industrial wastes. In industrial practice coal and coconut shell are two main sources for the production of ACs (Duran-Valle *et al.*2005; Sudaryanto *et al.* 2006)

The pH value (7.06) suggests that the substance is neutral in nature. When comparing with other results almost all the carbon samples recorded alkaline or acidic in nature. The untreated Banana Empty Fruit Bunch carbon (BEFP) recorded the highest pH (10.23±0.01) followed by H_3PO_4 and KOH treated carbon samples (9.78±0.0 and 9.04±0.01, respectively). Interestingly the KOH treated *Delonix Regia* Fruit Pod (DRFP) sample recorded a neutral pH (7.3±0.03) when compared to untreated carbon (8.72±0.08) (Sugumaran, 2012). H_3PO_4 treated carbon prepared from coconut saw dust also showed a highest pH of 9.31. The pH values of carbon samples prepared from coir pith differs in terms of activator used. H_2SO_4+H_2O_2 treated carbon showed the highest pH than other samples (H_2S0_4<H_3PO_4<HCl-<H_2SO_4+$NH_4S_2O_8$<H_2SO_4+H_20_2). The pH value (6.8) of Carbon sample from *Prosopis juliflora* plant treated by H_2SO_4 showed that the substance is acidic in nature. Similar results are reported in the case of Jatropha husk carbons treated by different activators (H_2SO_4-6.98, HCl-6.33, H_3PO_4-6.74, $ZnCl_2$-6.01, H_2SO_4+ $(NH_4)2S_20_8$-6.93 and HNO_3-5.53).In view of such varied pH values of ACs prepared from organic wastes, is influenced not only by the activators used but also the nature of precursors.

Bulk density is an important characteristic of the carbon and is invariably related to the starting material. It is an important parameter of powdered solids. The American Water Work Association has set a lower limit on bulk density at 0.25 g/ml for activated carbon to be of practical use (Veena Devi *et al.*2012). The bulk density value of the prepared AC (0.8 g/ml) satisfies this condition.H_3PO_4 treated DRFP showed a bulk density (0.45 g/ml) followed by KOH treated *Delonix regia* Fruit Pod (DRFP) (0.46 g/ml). The H_3PO_4 treated Banana Empty Fruit Bunch (BEFP), however, showed lowest bulk density (0.33 g/ml) when compared to other experimental carbons (Sugumaran, 2012).AC prepared from coir pith using different activating agents showed the bulk density ranging between 0.22-0.25 g/ml and that of Jatropha husk samples treated by different activating agents also showed more or less the same range of 0.2-0.4 g/ml. The bulk density of activated carbon prepared from saw dust of coconut tree using H_3PO_4 and that prepared from *Prosopis juliflora* using H_2SO_4 is 0.76 g/ml and 0.60 g/ml respectively; which is nearest to our value (Ahamed A. A., 2008). However PAC that was prepared using H_3PO_4 showed a higher bulk density of 0.8 g/ml.

In prepared AC derived from waste tea dust had 1.9% moisture which is significantly lower than moisture content of ACs prepared from Jatropha husk using(15%), coir pith(11%),saw dust of coconut tree and jatropha husk using NaOH is 3% and 2% respectively. Further, it is very clear from **Table 1** that the ash content in prepared AC is high (12.4%). The low ash content makes activated carbon an attractive candidate for adsorption studies. The ash content of other samples is 3-13%, however AC prepared from jatropha husk by steam activation and from coir pith using H_2SO_4+H_2O_2 showed 31.8% and 18.04% respectively. Thus, interms of ash content, the AC prepared from tea dust is relatively low. Conductivity of PAC is 0.025µs.There are some similar results of 0.02µs and 0.07µs reported in $ZnCl_2$ treated and H_2SO_4+ $(NH_4)2S_2O_8$ jatropha husk carbons. When comparing to AC prepared from coir pith using different activators the conductivity of our sample is very less (Ramakrishna, and Namasivayam., 2009).

Table 1 Characteristics of prepared AC

Characteristics of Prepared AC	
pH	7.06
Bulk Density (g/ml)	0.8
Ash (%)	12.4
Conductivity (µs)	0.025
Surface Area (m^2/g)	280.3927
Moisture (%)	1.9
Carbon ((%)	73.09
Oxygen (%)	20.06
Magnesium (%)	0.43
Aluminium (%)	0.38
Phosphorous (%)	3.36
Calcium (%)	2.68

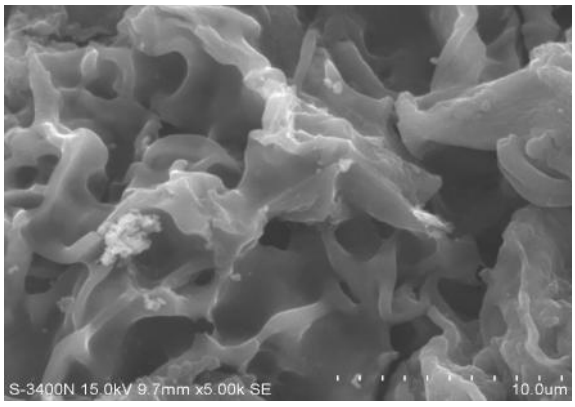
The results on BET surface area (m^2/g) are presented in the above tables. The morphological analysis of Activated Carbon from used tea dust (Present Study) and CAC (Commercial AC) was performed by scanning electron microscopy. Figures 1 and 2 show the structure of Prepared Activated Carbon (PAC) and

CAC. The irregular surface, bigger holes and cave type of openings are seen on the surface of PAC. CAC showed higher surface area of 890 m^2/g than PAC, which showed the value of 280 m^2/g. The high surface area CAC was mainly due to high temperature carbonization adopted for CAC in preparation than PAC. Commercial carbons have a typical surface area range of about 400 to 1600 m^2/g. Among the BEFP and DRFP ACs, the H_3PO_4 treated DRFP carbon sample had the highest surface area (22.29 m^2/g) followed by untreated carbon (17.54 m^2/g) and H_3PO_4 treated BEFP carbon (15.37 m^2/g). The lowest surface area was observed in KOH treated DRFP (0.3242 m^2/g) followed by untreated BEFP carbon sample (1.04 m^2/g) Table 2.

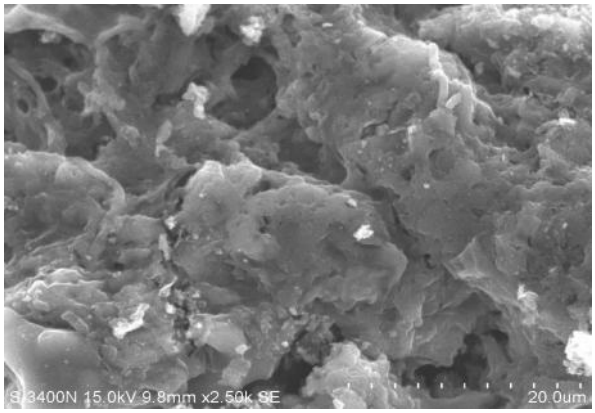
Table 2 BET surface area

Type of AC	Single point surface area at P/Po (m^2/g)	BET Surface Area (m^2/g)	Langmuir Surface Area (m^2/g)
CAC	0.250639080: 894.6307	890.0479	1261.6698
PAC	0.251059337: 281.8757	280.3927	401.0072

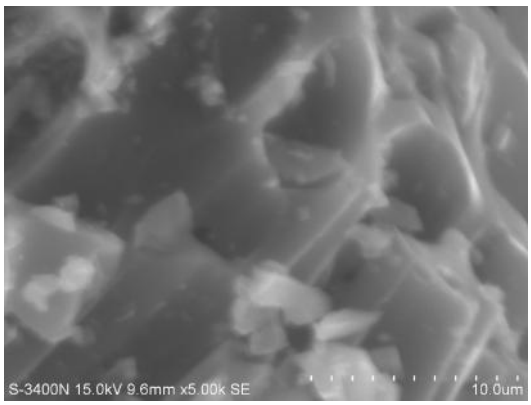
Considering the BEFP and DREP the SEM reports of the AC prepared from *Jatropha* husk showed that when the JH is treated with activates, the opening of the pores in the surface of the char occurs, due to extraction of some materials from the surface by the chemical activation (Kumar *et al*.2008). This may not be true for all activates, because not all activates have the same reactive properties. When comparing with commercial AC, carbon samples from used tea dust treated by H_3PO_4 showed clear open porous structures and it showed larger pore size also even when compared to commercially available ACs.



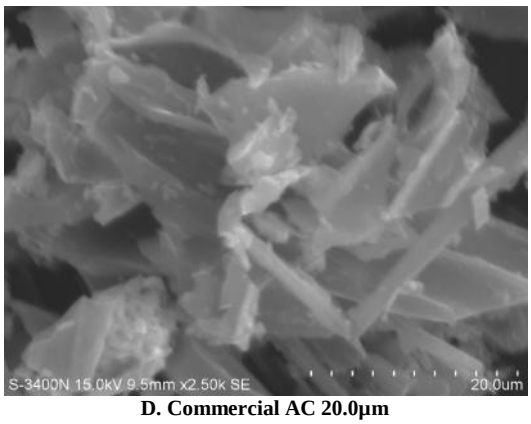
A. Prepared AC 10.0µm



B. Prepared AC 20.0µm



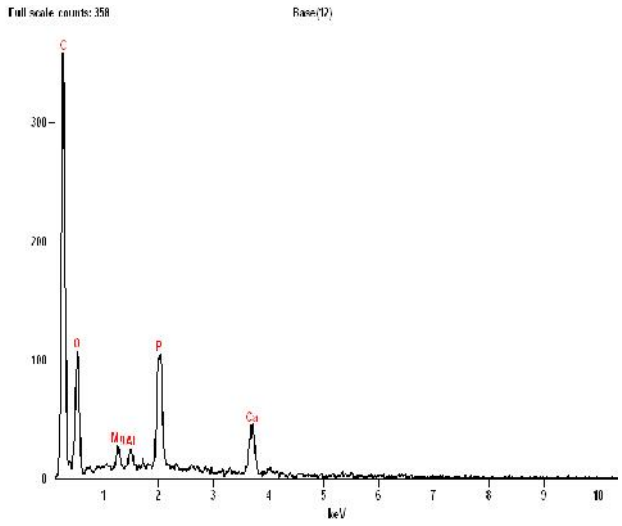
C. Commercial AC 10.0µm



D. Commercial AC 20.0µm

Fig 1 The SEM images of prepared AC (present study) and commercially available AC

Greater amount of minerals like potassium, calcium, phosphorus, and magnesium are naturally present in green tea leaves; however, phosphorus constitutes between15-20% of weights of all minerals in tea dust Another important mineral is located in the tea - phosphorus.



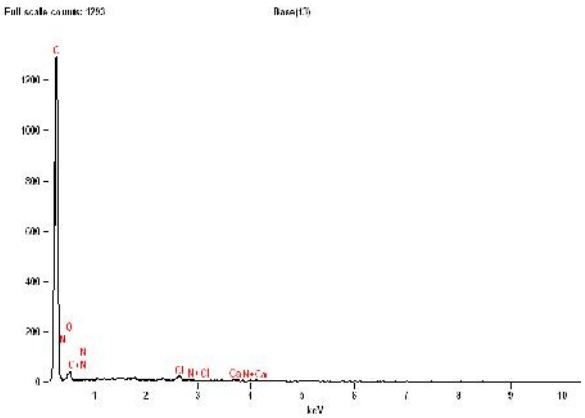
Live Time: 30.0 sec

Fig.3 EDX spectrum of commercially available

The EDX spectrum indicated that carbon content of the CAC is very high (91.75%) whereas Carbon content of the PAC prepared from used tea dust (73.09%); however, present values in tea dust is relatively higher when compared to carbon prepared from fresh tea waste (Yagmur *et al*., 2008).

Element Line	Net Counts	Net Counts Error	Weight %	Atom %	Formula
C K	2773	+/- 62	73.09	80.64	C
O K	780	+/- 47	20.06	16.62	O
Mg K	174	+/- 19	0.43	0.23	Mg
Al K	157	+/- 19	0.38	0.19	Al
P K	1207	+/- 57	3.36	1.44	P
Ca K	581	+/- 28	2.68	0.89	Ca
Ca L	0	+/- 32	---	---	
Total			100.00	100.00	

Element Line	Net Counts	Net Counts Error	Weight %	Atom %	Formula
C K	9407	+/- 90	89.89	91.75	C
N K	88	+/- 61	7.32	6.41	N
O K	112	+/- 33	2.10	1.61	O
Cl K	230	+/- 22	0.44	0.15	Cl
Cl L	4	+/- 24	---	---	
Ca K	95	+/- 15	0.25	0.08	Ca
Ca L	0	+/- 40	---	---	
Total			100.00	100.00	



Live Time: 30.0 sec

Higher values of phosphorus in PAC might be due to use of orthophosphoric acid as activating agent used to prepare activated carbon coupled with the naturally present phosphorus (Naturland-2001). Further, indication of aluminium and fluoride in the EDX spectrum might have been due to environmental pollution (Fung *et al*.1999) and normally present in the tea leaves.

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