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Production of activated carbon from tea waste and its application in water treatment

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Abstract

Activated carbon is known as one of the most environment friendly adsorbents for its consumption in industries as well as in water treatment. The purpose of the study was to utilize tea waste as a low cost and readily available natural precursor for production of this valuable adsorbent by simple methods and to apply the product for water treatment. Dried tea wastes of two local brands treated with 0.5M and 1M sulfuric acid as an activating agent were subjected to direct combustion, carbonization at 500°C and sieving to produce activated carbon. The product was characterized for its adsorption activity through determination of iodine number, moisture and ash content, pH and percentage yield. Percentage yield of the activated carbons was more with 0.5M activating agent (24.6% and26.4%) compared to the yield with 1M activating agent (23.4% and 25%). Higher iodine number (672.3mg/g and 597.6mg/g) with 1M activating agent indicates better adsorption activity of the activated carbons compared to 0.5M concentration (224.1mg/g and 373.5mg/g). Higher molarity of sulfuric acid reduced total dissolved solids and turbidity more while increasing the dissolved oxygen both in canal and lake water samples compared to the untreated ones. The color removing ability was also checked. A change towards lighter color intensity for activated carbon treated red, green and yellow food color aqueous solutions was confirmed by spectrophotometric analysis. It is concluded that 1M sulfuric acid is a better option for producing activated carbon from tea waste and that the product is an effective adsorbent for water purification.

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Introduction

Activated Carbon (AC), also termed as activated charcoal or activated coal, is defined as a wide range of microcrystalline, non-graphitic carbonaceous materials that exhibit highly developed pore structure (Ekpete and Horsfall, 2011). The raw carbonaceous material undergoes the activation process in the presence of an appropriate activating agent and results in the production of activated carbon. Activated carbon is one of the most environment friendly adsorbent that has numerous applications worldwide. Its composition depends on the processing method and the raw materials or precursors, from which it is obtained. Its pore size ranges from 0.20 to 0.60 cm^3/g, and has been found to be as large as 1 cm^3/g. it exhibits a surface area ranging from 800 to 1500 m^2/g but has been found to be in excess of 3,000 m^2/g (Park *et al.*, 1997). Most of the raw carbon containing materials do not exhibit a high degree of porosity and their internal surface area is up to 10-15m^2/g. but after the activation process, the internal surface area ranges to 1500m^2/g (Jankowska *et al.*, 1991).

Most commonly used activating agents of carbonaceous material arephosphoric acid, hydrogen peroxide, zinc chloride, potassium permanganate, ammonium per-sulfate, potassium hydroxide, sulfuric acid and many others. Sulfuric acid is one of those agents that impart such characteristics and modifications in the graphitic structure that yields high adsorption capacity of the activated carbon (Kumar *et al.*, 2013).

Due to excessive urbanization and industrialization, the global trends have shifted to more rigorous and stringent environmental conservation standards, cost effectiveness and technical applicability in the utilization and selection of adsorbents throughout the world, especially for water treatments. To date, many raw carbonaceous materials have been used and are being continuously tested for the cost effective production of activated carbonincluding olive stones, coconut husk, banana empty fruit sticks, egg shells, pecan shells, sugar cane bagasse, molasses and many other sources as well. But somehow availability of such materials hinders the cost effective production due to the chemical activation treatment required to produce activated carbon from them (Jambulingam *et al.,* 2007; Manase, 2012).

In recent years, tea waste has been found to be a highly effective precursor to obtain the activated carbon due to its high carbon content, and is used as the major adsorbent in water treatments (Wankhadeand Ganvir, 2013). This is due to the chemical nature of the cell wall of tea which consists of lignin, tannins, structural proteins, cellulose and hemicellulose. All these components are considered excellent metal scavengers and adsorbing other organic and inorganic solutes from water which include pesticides, acidic and basic residues. Impurities such as heavy metals, artificial dyes, food colors are also readily adsorbed from wastewater from food and beverage industries by activated carbon. Moreover, they are widely used for deodorization and purification of chlorinated waters for drinking purpose. The adsorption ability of activated carbon depends on different factors such as the pore size, concentration and composition of contaminate, temperature, pH of water, the chemical composition of activated carbon itself (Abdul Khalil *et al.*, 2013).According to Betzy and Soney, 2015, activated carbon can be produced from natural sources such as waste tea, bamboo waste, cherry stones, and Paulownia flower and it is good in removing hazardous compounds and dyes from waste water. The activating agents commonly usedare $ZnCl_2$, K_2CO_3, NaOH and H_3PO_4. Adsorption on activated carbons from lignocellulosic biomass is a cost-effective technique for elimination of environmental pollution as shown in a study by Muthanna 2016 on date palm stones used for waste water treatment.

Activated carbon consumption in different industries as well as in water treatment, make it noteworthy that activated carbon is produced in larger amounts using low cost precursors and procedures.

Tea waste is one of those precursors which is most readily available carbonaceous source throughout the world and produces highly adsorptive activated carbon. The objective of this study was to produce activated carbon from tea waste by using simple and economical method, to analyze its adsorption activity through application in water treatment on lab scale.

Materials and methods

Sample collection

Samples from used tea bags and open tea waste of the two brands were collected separately from some houses in green produce bags that are used to store food for refrigeration. These produce bags contain a mineral 'Oya', a zeolite. The zeolites have the ability to absorb ethylene gas produced by plants which increases the rate of ripening and decaying. These bags were used to prevent tea waste from decaying or producing foul odor.

Production of activated carbon

Production of activated carbon has been achieved by using the basic method (Abdul Khalil *et al*., 2013).

Washing of tea waste

The samples were washed with tap water several times with sieves and strainer to get rid of the traces of milk and any other flavors added to milk by the consumers. The wet weight of 700g of brand 1 and 680g of brand 2 was obtained.

Drying of tea waste

The tea waste samples were first sun dried for 20 consecutive days in steel troughs and trays. 107g of brand 1 and 101g of brand 2 were obtained. These were further oven dried at 50^{o}C for 24 hours and their dried weights were measured.

Digestion with sulfuric acid

Sample from each brand was digested with 0.5M and 1M sulfuric acid in 1:1 ratio. 50g of dried tea waste and 50ml of sulfuric acid were mixed separately in beakers in a fume hood, and shifted to steel troughs covered with aluminium foil at 40^{o}C for 12 hours in an oven.

Washing of digested sample

Each of the four digested samples were washed 10-15 times with distilled water to remove acid. pH was maintained at 7 between washings. Wet weights of all the samples were measured.

Direct combustion

The washed samples were directly combusted on Bunsen burner in fume hood using evaporating dishes to remove all moisture content from the sample. Ash was formed which was further carbonized.

Carbonization

Ash was transferred into pre-weighed crucibles and was kept in the muffle furnace at500^{o}C for 15 minutes. The activated carbon formed was collected after cooling, stored in air tight polyethylene (PET) bags.

Sieving

Sieving of the product was done in order to separate the oversize and undersize particles. The first sieving was done at 180 mesh, which was 40µm and the second was done at300 mesh, which was 80µm. Products with two different sizes were obtained after sieving.

Characterization of activated carbon

The activated carbon formed from two brands of tea waste treated with 0.5M and 1Msulfuric acid was characterized for adsorption activity and other properties. The iodine number, moisture, ash content and percentage yield were determined by using the formulae (Kirbaslar *et al*., 2001).

Iodine number

It is a direct measure of the activity level of the activated carbon. The higher the iodine number, higher is its degree of activation. The SI unit of iodine number is mg/g. This Iodine number was calculated following an iodometric titration in which alkaline iodide (0.1N) was titrated against sodium thiosulfate(0.05N) using starch solution (1%) as an indicator. Blank reading was taken.

Readings with the activated carbon were also taken. The iodine number was calculated by the formula

$$\text{Iodine number} = C \times \text{Conversion factor (mg/g)}$$

$$C = B - A$$

$$\text{Conversion factor} = \frac{\text{Molecular weight of iodine} \times \text{Normality of iodine} \times 40}{\text{Weight of carbon} \times \text{Blank reading}}$$

Moisture content

1g of each type of activated carbon was taken in a pre-weighed crucible. These crucibles were placed in an oven at 105°C for 4 hours. The moisture content of each type was calculated using formula:

$$\text{Moisture content (\%)} = \frac{\text{Loss in weight on drying (g)}}{\text{Initial sample weight (g)}} \times 100$$

Ash content

1g of each type of activated carbon was weighed accurately and transferred to the pre weighed crucibles. These crucibles were then placed in the muffle furnace at 500°C. After the furnace cooled down, the crucibles were removed and reweighed. The ash content was calculated by the formula:

$$\text{Ash content (\%)} = \frac{\text{Ash weight (g)}}{\text{Oven dry weight (g)}} \times 100$$

Percentage yield

The percentage yield of the activated carbons produced from both brands of tea waste and with both the molarities, was calculated by the formula

$$\text{Yield \%} = \frac{\text{Weight of activated carbon produced (g)}}{\text{Weight of dried precursor used (g)}} \times 100$$

pH determination

1g of each activated carbon was weighed accurately and stirred with 100ml of distilled water in a beaker for 1 hour and was left to stabilize for 5 minutes. pH of each solution with respective activated carbon was noted.

Application of activated carbon in treatment of canal and lake water**.** The activated carbon produced from tea waste was used to treat water from Lahore Canal, water from a man-made lake in Iqbal Park, Lahore, and water with commercially available food colors dissolved in it.

This was done using simple low cost methods, using only the available lab ware and poly ethylene (PET) bottles. A combination of different laboratory oriented low cost experiments was performed to purify and check quality of water samples. Single bed filtration was carried out in PET bottle shown in Fig. 1. Both untreated and treated water samples were subjected to a number of tests, to check the quality of water with respect to purification.

Total dissolved solids

The amount of total dissolved solids was checked by taking 50ml of each of the four samples in 100ml of pre-weighed evaporating dishes, cooling them down and weighing again. Total dissolved solids in all samples were calculated by the formula:

$$\text{TDS } (mgl^{-1}) = \frac{W_2 - W_1}{\text{volume of sample taken}} \times 10^6$$

Nitrate test

The presence of nitrates in the untreated and treated water samples was tested by adding Diphenylamine reagent to 1ml of each sample and color change was noted.

Turbidity test

Turbidity was checked using the Turbidity meter. This was done for 10ml of each of the four samples separately and the readings were taken.

Dissolved oxygen (DO)

The Dissolved Oxygen (DO) content of the untreated and treated water samples was measured for three consecutive days with a DO meter.

Treatment of water with food colors

Green, red and yellow commercially available food colors were used to check the adsorption activity of the activated carbon produced. 1% solutions were prepared by dissolving 1g of the powdered color in 100ml of distilled water. This colored water was then filtered through the activated carbon in a lab scale setup shown in Fig. 2.

The visible color difference before and after the filtration of colored water was observed to detect the activity of activated carbon. For further confirmation, the absorbance of each colored water were taken using a UV– VIS Spectrophotometer at 700 nm for red,530 nm for green and 577.5 nm for yellow.

Results and discussion

Four separate activated carbons were obtained from tea waste of two brands, each treated with 0.5M and 1.0M sulfuric acid as an activating agent. Fig. 3 shows the activated carbon produced with particle size of 40µm and 80µm after sieving.

Characterization of activated carbon

All the four types of carbons were characterized as given in Table 1.

Table 1. Characterization of activated carbon produced from tea wastes of the two brands treated with different molarities of sulfuric acid.

Tea waste	Molarity of H_2SO_4 (M)	Iodine number (mg/g)	Moisture content (%)	Ash content (%)	Percentage yield (%)	pH
Brand 1	0.5	224.1	12	25	24.6	6.8
	1	672.3	10	23.3	23.4	7.2
Brand 2	0.5	373.5	11	21.3	26.4	7.4
	1	597.6	12	22.7	25	7.2

The range of iodine number for an activated carbon to be effective adsorbent is 500-1200mg/g (Grassi, 2012). Activated carbon produced using 1M activating agent for both brands fall within this range as indicated by iodine number. 0.5M sulfuric acid treatment yielded the products with iodine number less than the normal range.

This shows that molarity directly affects the adsorption activity. Brand 1 is exhibiting higher adsorption activity than brand 2 and 1M concentration of sulfuric acid in both cases is more effective than 0.5M. However, the percentage yield of activated carbon obtained from tea waste treated with 1M activating agent was lesser than that of 0.5M concentration.

Table 2. Quality check of canal water and lake water samples after treatment with activated carbon produced from tea waste of the two local brands.

Tea waste	Water sample	Treated with AC of	TDS (mg/l)	Nitrate test	Turbidity (NTU)	DO (mg/l)
	Untreated canal water	-	260	+	90	4.97
	Untreated lake water	-	420	-	24.63	4.91
Brand 1	Treated canal water	0.5M	27	+	3.49	7.05
		1M	23	+	3.01	7.97
	Treated lake water	0.5M	55	-	4.04	6.23
		1M	49	-	3.69	7.00
Brand 2	Treated canal water	0.5M	28	+	5.09	6.98
		1M	24	+	3.27	7.58
	Treated lake water	0.5M	57	-	5.21	5.30
		1M	41	-	4.21	6.23

Moisture contents were also checked in all four products formed. The range of moisture already reported in activated carbon is not more than 3-6% (Yadavalli and Heggers, 2013).

Whereas, the moisture contents of activated carbons produced in this study were all above this typical range due to the lack of moisture free environment where these were produced and stored.

Neutral pH on average for all samples and less amount of ash for brand 2 was obtained. Lesser ash content could be obtained to achieve even better adsorption of the activated carbons, because the lesser the amount of ash content, higher is the adsorption ability of the activated carbon as reported by Yadavalli and Heggers, 2013.

Treatment of canal water and lake water samples

The four activated carbons were used as adsorbents to purify and check the quality of canal and lake water samples. Both untreated and carbon treated samples were tested for the parameters given in Table 2. Variations in adsorption activity of activated carbons from the two brands were observed.

Table 3. Treatment of food color solutions with activated carbon produced with different molarities of the activating agent.

Food Color aqueous solutions	Activated carbon from tea waste of	Molarities of the activating agent (M)	Wavelength (nm)	Absorbance	Color after filtration through AC
Untreated (control)	-	-	700	0.026	-
Red	Brand 1	0.5	700	0.018	Light pink
		1		0.002	
	Brand 2	0.5	700	0.016	
		1		0.002	
Untreated (control)	-	-	530	0.613	-
Green	Brand 1	0.5	530	0.234	Very light green
		1		0.001	
	Brand 2	0.5	530	0.349	
		1		0.020	
Untreated (control)	-	-	577.5	0.046	-
Yellow	Brand 1	0.5	577.5	0.015	Pale yellow
		1		0.003	
	Brand 2	0.5	577.5	0.021	
		1		0.003	

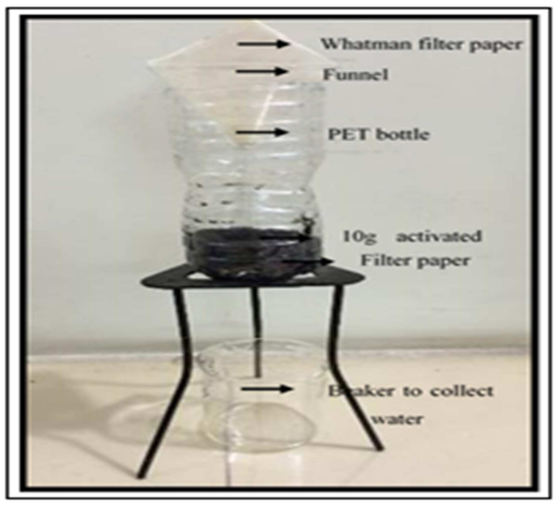

Fig. 1. Experimental set up for lab scale single-bed filtration using polyethylene (PET) bottle.

The activated carbons produced with 1M sulfuric acid were more active in adsorbing impurities from water samples.

Nitrates present in canal water were not removed after treatment with activated carbon as indicated by blue coloration persisting in diphenylamine test. No nitrates were present in lake water.

The nitrates, calcium and magnesium present in wastewater or any water for filtration are not removed by the activated carbon as already reported by Singh *et al.*, 2011.

1M sulfuric acid treatment has reduced the turbidity (NTU) and total dissolved solids (TDS mg/l) in all four carbon treated water samples. Increase in dissolved oxygen (DO mg/l) throughout indicates that both canal and lake water have been purified effectively (Table 2).

Fig. 2. Experimental set up for treatment of food color solutions with activated carbon.

The activated carbon produced by 1M sulfuric acid from both brands exhibited a higher adsorption activity and removed a large number of impurities from canal water and lake water.

Treatment of water containing artificial food colors

The treatment of three food color aqueous solutions with activated carbon was done in order to relate it with the industrial effluents that contain food colors and dyes in them (Hameed, 2009).

The results in this treatment have also shown that higher molarity of the activating agent enhances the color removal ability of the activated carbon. Further confirmation was done by measuring the color intensity of untreated and treated solutions with UV-VIS spectrophotometer (Table 3).

Least absorbance values after treatment with activated carbon were seen for 1M concentration of sulfuric acid indicating the effective adsorption of colors for purification of water.

Therefore, the adsorption activity of activated carbon mainly depends on molarity of the activating agent used and not on the brand difference of tea waste in this study.

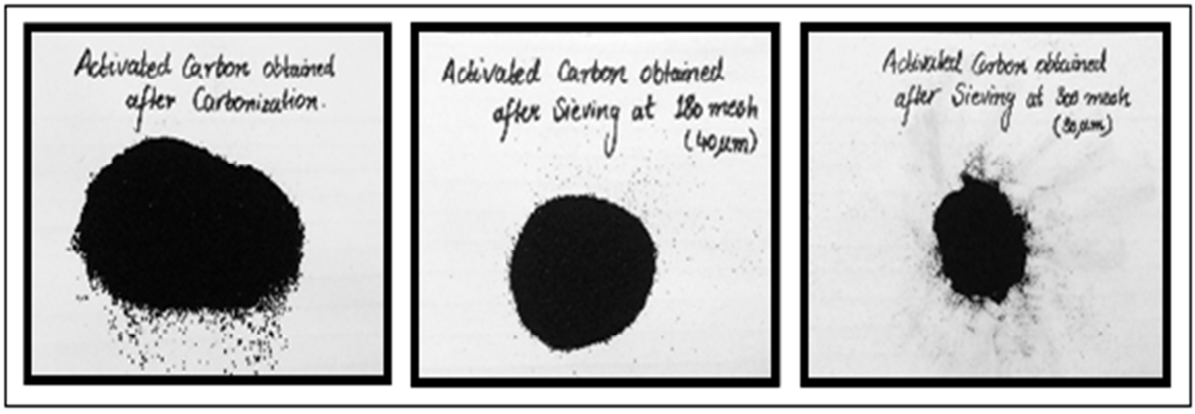

Fig. 3. Activated carbon produced after carbonization and sieving through 180 and 300 mesh sizes, released 40µm and 80µm particles.

Conclusion

Four activated carbon products were prepared from two brands of tea waste after treatment with 0.5M and 1M sulfuric acid as an activating agent. Higher percentage yield of activated carbons was obtained for brand 2than for brand 1 tea waste. The adsorption activity of all four activated carbons was much higher with 1M sulfuric acid as compared to 0.5M after characterization and quality tests. Hence, the adsorption activity of the product is dependent on the molarity of the activating agent and not on the type of the tea waste. Tea waste is a good organic source for production of activated carbon that can be utilized as an effective adsorbent for removal of impurities including colors or dyes for purification of water.

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