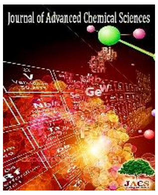
Journal of Advanced Chemical Sciences 2(3) (2016) 360–362

ISSN: 2394-5311

Contents List available at JACS Directory

Journal of Advanced Chemical Sciences

journal homepage: www.jacsdirectory.com/jacs



Preparation and Characterization of Activated Charcoal Derived from Orange Peel

S.D. Ashtaputrey*, P.D. Ashtaputrey

Department of Chemistry, Institute of Science, Nagpur – 440 001, Maharastra, India.

ARTICLE DETAILS

Article history:
Received 13 July 2016
Accepted 26 July 2016
Available online 28 August 2016

Keywords:
Carbonization
Activated Charcoal
Orange Peel

ABSTRACT

Powdered activated charcoal was prepared from orange peel. Carbonization was done at 300 °C, 350 °C, 400 °C, 450 °C and 500 °C for an hour and allowed to cool at room temperature. Chemical activation was achieved by impregnating the prepared charcoal with activating reagent 1 N HCl and heated to 300 °C, 350 °C, 400 °C, 450 °C and 500 °C temperature respectively for an hour. Characteristics of the activated carbon were determined using standard methods given by AWWA and CEFIC. A significant difference in the properties of pH, conductance, ash content, moisture, methylene blue value, iodine value and calorific value were observed on the activated carbons.

1. Introduction

Adsorption is a widely used as an effective physical method for elimination or lowering the concentration of wide range of dissolved pollutants (organic and inorganic) in an effluent. Activated carbon (AC) is the best adsorbent that can be used effectively for removal of broad spectrum of pollutants from air, soil and liquids. Activated carbon is prepared by carbonization and activation of a large number of raw materials of organic origin such as wood, coal, coconut shell and lignite [1]. Characteristics of the ACs depend on the physical and chemical properties of the raw materials as well as the methods of activation [2]. The carbonization process enriches the carbon content and introduces the porosity in the char while activation further develops the porosity and creates some ordering in the structure.

Orange is the important citrus fruit with a global production. In India Nagpur city is known as the Orange city for the major production of quality Oranges. The fruit is a hesperidium with carpels or segments filled with juicy arils and seeds. The peel includes the epidermis covering the exocarp consisting of irregular parenchymatous cells which completely enclose numerous glands or oil sacks. It is rich source of cellulose, proteins and lignin. Therefore an attempt was made to prepare the low cost adsorbent from Orange peels. The tons of Orange peels were discarded and send garbage as useless material and it is very essential to find applications and uses for these peels as a management of waste now days is becoming very serious environment issue. The waste peels are low cost, non-hazardous and environment friendly biomaterials which can be used as adsorbent in various applications.

The literature work revealed the suitability of variety of agro based materials like groundnut husk, rice husk [3, 4], coconut coir pith [5], and other biomaterials to treat the industrial waste water. The present study focused to prepare the activated carbon by the carbonization of Orange peel and then chemical activation of carbon.

2. Experimental Methods

2.1 Sample Collection and Preparation

For the present study Orange peels was collected from houses and local market and were cut into smaller sizes and washed with water. Washed Orange peel then sundried for fifteen days. The pieces were then dried in an oven at 110 °C for an hour.

2.2 Carbonization

Carbonization of well dried sample was carried out in a muffle furnace (Bio-technics India, Model BTI 40) by placing a sample in a silica crucible at different temperatures of 300 °C, 350 °C, 400 °C, 450 °C, 500 °C for one hour each. It was ensure that little or no oxygen was present during carbonization. The charcoal thus produced was withdrawn from the furnace, cooled, Washed with tap water and dried in an oven at 110 °C and ground in a mortar by means of pestle applying moderable pressure. They were sieved through 100-200 mesh sieves. Then chemical activation was carried out according to the methods of Girgis [6] with slight modification. 1N aqueous solution of hydrochloric acid (HCl) was mixed with 25 g sample in the ratio 1:5. The mixture was left to soak for 12 hours and later heated to form a paste. The paste was placed in a preprogrammed furnace and carbonized at the same temperature of initial carbonization (300 °C, 350 °C, 400 °C, 450 °C and 500 °C) for one hour. The sample was allowed to cool to room temperature overnight. It was then removed, neutralized with NaOH and washed with distilled water till the pH was constant. Then it was dried at 105 °C in an oven and later removed to cool at room temperature. The carbon produced was sieved with 106 µm, put in an air tied bottle and labelled as OPAC300, OPAC350, OPAC400, OPAC450 and OPAC500 respectively and collectively called orange peel activated carbons.

2.3 *Determination of pH and Conductivity*

The pH and conductivity were determined according to the standard test methods of ASTMD3838-80. 1 g of sample was weighted and transferred into a beaker and 100 mL of distilled water was added and stirred for one hour. Samples were allowed to stabilize and then pH measures using a Toshniwal pH meter Model CL 54. The conductivity of supernatant solution was also recorded every time using Systronics conductometer Model 304.

2.4 Ash Content Determination

Ash content determination was done according to ASTMD2866-94 method. 1 g Dry sample (W_o) was placed into a porcelain crucible and transferred into a preheated muffle furnace set at 1000 °C temperature. The furnace was left on for one hour after which the crucible along with content was transferred to desiccator and allowed to cool. The crucible along with the content was reweighted (W_{ash}) and the weight loss was recorded as the ash content of the AC sample. Then the percentage ash content was determined from the equation,

$$\text{Ash }\% = \frac{100 \text{ X Wash}}{\text{Wo}}\ \%$$

---

*Corresponding Author
Email Address: santoshashtaputrey@gmail.com (S.D. Ashtaputrey)

Cite this Article as: S.D. Ashtaputrey, P.D. Ashtaputrey, Preparation and characterization of activated charcoal derived from orange peel, J. Adv. Chem. Sci. 2(3) (2016) 360–362.

2.5 Volatile Matter Content

A known quantity of sample was taken in cylindrical crucible closed with a lid. It was then heated to 925 °C for exactly 7 minutes in a muffle furnace. Then the crucible was cooled in a desiccator and weighted. Volatile matter content was determined from the equation,

$$\text{VM}\ \% = \frac{100[100(\text{B}-\text{F})-\text{M}(\text{B}-\text{G})]}{[(\text{B}-\text{G})(100-\text{M})]}$$

where B=Mass of crucible, lid and sample before heating
F=Mass of crucible, lid and contents after heating
G=Mass of empty crucible and lid
M=% of moistures determined

2.6 Moisture Content

Small amount of activated carbon sample weight was measured and then taken in a petri dish. It was spread nicely on the dish. It was then heated in an oven at a temperature of (105-110) °C for 1.5 hr. The petri dish was left open or not covered during heating process. After heating petri dish was removed and cooled in a desiccator. After cooling the weight of dried sample was measured. Moisture content was determined from the equation,

$$\text{M}\ \% = \frac{100(\text{B}-\text{F})}{(\text{B}-\text{G})}$$

where B=weight of Petri dish +original sample
F=weight of Petri dish+ dried sample
G=weight of Petri dish

2.7 Iodine Value

This was done according to the ASTMD4607-94 method as modified by Okuo and Ozioko [7]. 0.1 M sodium thiosulphate solution was titrated against 20 mL carbon sample free aliquot solution (prepared by centrifuging 0.5 g of the AC sample in 25 mL of 0.1 M iodine solution). Freshly prepared 1 % starch solution (5 mL) was used as indicator.

Similarly the quantity of thiosulphate needed to titrate 20 mL of blank solution was determined. Each titration was carried out in triplicate and the average titer volume used in calculating the iodine value (IV) using equation,

$$\text{IV} = \frac{(\text{v}-\text{x})}{\text{y}} X \frac{\text{v}}{\text{w}} X\ M(126.9)\ mg\ of\ Iodine/g\ of\ carbon\ sample$$

where x=volume of thiosulphate used for carbon free aliquot
y=volume of thiosulphate used for blank solution
w=weight of sample
M=Molarity of Iodine solution used for titration
V=Volume of Iodine solution used for titration

2.8 Methylene Blue Value/ Decolourisation Power

Methylene blue test solution was prepared by dissolving 1500 mg of pure dye in 1000 mL of water using volumetric flask. The solution was then allowed to stand overnight. The solution was then checked by diluting 5 mL with 0.25 % acetic acid in 1 L. Volumetric flask and measuring the absorbance at 620 nm for 1 cm. (The absorbance must be 0.840 $\pm$0.01).

0.1 g of activated carbon was added to 10 mL of methylene blue test solution in a 50 mL glass stoppard flask which was then shaken till gets decolorized. The addition of 1 mL of methylene blue test solution was continued as long as decolourisation occurs within 5 minutes. The total volume of test solution added till decolourisation is expressed in terms of mg of methylene blue adsorbed by 1 g of activated carbon as methylene blue value of the sample by using the formula,

$$\text{Methylene blue value} = \frac{15\ X\ V}{10\ X\ M}$$

where V=Volume of methylene blue consumed
M=Mass in gm of activated carbon taken for the test

2.9 Calorific Value

The energy values were determined by bomb calorimeter following the standard method ASTMD1989-95.

2.10 Fixed Carbon Content

Fixed carbon content was determined by using the following equation,

Fixed carbon FC = 100–(% moisture content+ % volatile matter + % ash content)

3. Results and Discussion

Table 1 shows the various activated carbons prepared from the carbonization of orange peels followed by chemical activation using HCl at the same temperature of carbonization. All carbons characterized were of particle size of 106 µm. It was noticed that the change in characteristics of various activated samples were due to activation temperature.

Table 1 List of activated carbons prepared by chemical activation method

Sr. No.	Sample code	Activation Method
1	OPAC300	Chemical activation using HCl at 300 °C
2	OPAC350	Chemical activation using HCl at 350 °C
3	OPAC400	Chemical activation using HCl at 400 °C
4	OPAC450	Chemical activation using HCl at 450 °C
5	OPAC500	Chemical activation using HCl at 500 °C

The pH of all the ACs was generally slightly acidic in the range 6.04 to 6.74. The carbons of pH range 6 to 8 are useful for most applications [8, 9]. Hence the studied ACs could be acceptable for most applications involving adsorption from aqueous solutions.

The conductivity study shows the presence of leachable ash which is considered as an impurity and undesirable in AC. Good conductivity in ACs ranges from 51.85 µS to 70.75 µS [9]. In comparison with commercial carbons most carbons prepared in this study exhibited high conductivity values which suggested that acid and water wash may not have been able to reduce leachable ash to level observed in commercial carbons. This could be due to presence of substantial amount of water soluble minerals remained in the ACs.

The ash content shows the amount of inorganic substituent present in the carbon. From Table 2 it was found that all the ACs has less ash content which can increase the fixed carbon value. High ash content is undesirable for activated carbon since it reduces the mechanical strength of carbon and affects adsorptive capacity.

Table 2 Analysis of OPAC samples

Sr. No.	Parameters/ Characteristics	OPAC300	OPAC350	OPAC400	OPAC450	OPAC500
1	pH	6.74	6.04	6.52	6.2	6.37
2	Conductivity (µS)	990.4	1015.6	1142.4	1178.5	1105.6
3	Ash content (%)	7.10	7.89	8.35	9.07	9.12
4	Volatile content (%)	29.64	31.27	32.38	33.04	33.84
5	Moisture content (%)	4.75	4.31	3.87	3.13	2.79
6	Iodine Value (mg/g)	983	853	829	911	827
7	Methylene blue value (mg/g)	405	360	320	375	315
8	Calorific value (Kcal/kg)	6719.7	6494.71	6321.5	6124.3	5873.3
9	Fixed carbon (%)	58.51	56.53	55.4	54.79	54.25

Volatile matter is due to presence of organic compounds present in the raw material. From the data it was clear that all the carbons have a good percentage of fixed carbon.

The moisture content of the carbons should be less than 5 %. All the activated carbons studied had the moisture content less than 5 %.

Iodine adsorption is a simple and quick technique to determine the adsorptive capacity of AC, also known as iodine number often reported in mg/g (typical range 500-1200 mg/g). It has been established that the iodine number gives an estimate of the surface area in m^2/g [10], and is related to the porosity characteristics of the AC. A lower iodine number can be ascribed to the presence of pores narrower than 1.0 nm [9]. It should be noted that OPAC300 AC showed the highest value of iodine number implying higher surface area where as sample OPAC500 has the lowest iodine value implying the lower surface area. AC recommended for water treatment expected to have iodine value in the range 600 mg/g to

Cite this Article as: S.D. Ashtaputrey, P.D. Ashtaputrey, Preparation and characterization of activated charcoal derived from orange peel, J. Adv. Chem. Sci. 2(3) (2016) 360–362.

1100 mg/g [11]. Thus the ACs produced in this study is having more efficiency for use in water treatment.

Similarly methylene blue value is an indication of adsorptive capacity of AC for molecules with dimensions similar to methylene blue and other high molecular weight substances like dye molecules. It also helps to determine the surface area which results from presence of pore sizes greater than 1.5 nm. OPAC300 shows the methylene blue value greater than 400 indicates that the carbon is good for dye adsorption.

The calorific values indicate how the activated carbon is close to allotrope graphite carbon. Higher values indicate good carbonization and good activation process resulting in good quality AC. The calorific values of ACs were found in the order OPAC300 > OPAC350 > OPAC400 > OPAC450 > OPAC500. The fixed carbon values are in the range 54.25 to 58.51.

4. Conclusion

The result of the study could provide activated carbon consumers with cost effective and eco-friendly alternative sources. The ACs prepared at lower temperature of carbonization was exhibiting better Characteristics than at the higher temperature of carbonization. The ACs was better adsorbents for water treatment.

Acknowledgement

The authors gratefully acknowledge the Director, Institute of Science, Nagpur and Head, Department of Chemistry, Institute of Science, Nagpur for providing the facilities and support.

References

[1] H. Zhonghuo, M.P. Sriniwasan, N. Yaming, Novel activation process for preparing highly mesoporous activated carbons, Carbon 39 (2001) 877-886.

[2] A.C. Lua, J. Guo, Microporous oil-palm-shell activated carbon prepared by physical activation for gas phase adsorption, Lagmuir 17 (2001) 7112-7117.

[3] D. Shivakumar, J. Nouri, Removal of contaminants in apaper mill effluent by *Azolla caroliniana*, Pollution Res. 1(4) (2015) 297-304.

[4] D. Shivakumar, N. Murugan, R. Rajeshwaran, T. Shobana, C. Soundarya, et al, Role of rice husk silica powder for removing Cr(VI) in a tannery industry waste water, Inter. J. Chem. Tech. Res. 6 (2014) 4373-4378.

[5] D. Shankar, D. Shivakumar, M. Thiruvengadam, M. Manojkumar, Colour removal in textile industry waste water using coconut coir pith, Pollution Res. 33 (2014) 490-503.

[6] B.S. Grigis, M.F. Ishak, Activated Carbon from Cotton stalks by impregnated with phosphoric acid, Mat. Lett. 39 (1999) 107-114.

[7] A.C. Ozioko, J.M. Okuo, Adsorption of lead and mercury ions on chemically treated periwingkle shells, J. Chem. Soc. Nigeria 26(1) (2001) 60-65.

[8] F. Okiemmen, C. Okiemen, A. Wuana, Preparation and characterization of activated carbon from rice husk, J. Chem. Soc. Nigeria 32 (2007) 126-136.

[9] Q. Khadija, B. Inamullah, K. Rafique, K.A. Abdul, Physical chemical analysis of activated carbon prepared from sugarcane bagasse and use for sugar decolourisation, Inter. J. Chem. Biomolecular Engg. 1(3) (2008) 145-149.

[10] K. Gergova, V.N. Petrov, S. Eser, Adsorption properties and microstructure of activated carbon from agricultural by-products by stream pyrolysis, Carbon 32(4) (1994) 693-702.

[11] AWWA, Standards for granular activated carbons, American Water Works Association, 1991.

[12] ASTM, Standard test methods for activated carbons, American Society for Testing Materials, 1980.

[13] A.W. Verla, M. Horsfall (Jnr), E.N. Verla, A.J. Spiff, O.A. Ekpete, Preparation and characterization of activated carbon from fluted pumkin, Asian J. Nat. Appl. Sci. 1(3) (2012) 39-50.

[14] BIS, Activated carbons, powdered and granular- methods of sampling and tests, Bureau of Indian Standards, 1990.

[15] CEFIC, Test methods for activated carbons, European Council of chemical manufacturers federations, 1986.

[16] D.F.Aloko, G.A. Adebayo, Production and characterization of activated carbon from agricultural waste (rice-husk and corn-cob), J. Engg. Appl. Sci. 2(2) (2007) 440-444.

[17] I. Musa, C. Gimba, Preparation of activated from agricultural waste; Cyanide bindind with activated carbon matrix from coconut shell, J. Chem. Soc. 32 (2007) 167-170.

[18] J. Lain, A. Kalafat ,M. Labady, Preparation and characterization of activated carbons from coconut shells impregnated with phosphoric acid, Carbon 27(2) (1989) 191-195.

[19] J. Raffica Baseri, P.N. Palanisamy, P. Shivakumar, Preparation and characterisation of activated carbon from *Thevetia peruviane* for the removal dyed from textile waste water, Adv. Appl. Sci. Res. 3(1) (2012) 377-383.

[20] M.A. Rahman, M. Asadullah, M.M. Haque, M.A. Motin, M.D. Sultan, et al, Preparation and characterization of activated charcoal as an adsorbent, J.Surf. Sci. Technol. 22(3,4) (2006) 133-140.

[21] O.A. Ekpete, M.J. Horsfall, Preparation and Characterization of activated carbon derived from fluted pumkin stem waste, Res. J. Chem. Sci. 1(3) (2011) 10-15.

[22] O. Akaranta, T.O. Odozie, Activated carbon from kernael shell, Nigeria J. Appl. Sci. 4(1) (1986) 85-90.

[23] P. Sugumaran, V. Priya Susan, P. Ravichandran, S. Seshadri, Production and characterization of activated carbon from banana empty fruit bunch and *Delonix regia* fruit pod, J. Sustain. Ener. Environ. 3 (2012) 125-132.

[24] S. Karthikeyan, P. Sivakumar, P.N. Palanisamy, Novel activated carbon from agricultural waste and their characterization, E-Jour. Chem. 5(1) (2008) 409-426.

[25] T.M. Al Khusaibi, J. Jessica Dumaran, M. Geetha Devi, L. Nageswara Rao, S. Firoz, Treatment of dairy waste matter using orange and banana peels. J. Chem. Pharm. Res. 7(4) (2015) 1385-1391.

[26] W.K. Lafi, Production of activated carbon from acorns and olive seeds, Biomass Bioener. 20(1) (2001) 57-62.

[27] Y. Diao, W.P. Walawenber, L.T. Fan, Activated carbons prepared from phosphoric acid activation of grain sorghum, Biores. Technol. 81(1) (2002) 45-52.

Cite this Article as: S.D. Ashtaputrey, P.D. Ashtaputrey, Preparation and characterization of activated charcoal derived from orange peel, J. Adv. Chem. Sci. 2(3) (2016) 360–362.