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PRODUCTION AND CHARACTERIZATION OF ACTIVATED CARBON FROM GROUNDNUT SHELL SOURCED IN MAIDUGURI

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ABSTRACT

Groundnut shell that has been considered as agricultural waste, which sometimes poses environmental threat can now be considered as a precursor for the production of activated carbon (A.C) as an adsorbent to adsorb metals in raw water, thereby we can convert waste to wealth. The physiochemical parameters (characteristics) of the AC produced are very well comparable with those prepared from similar agricultural wastes reported. The pH of the produced activated carbon was found to be 6.8, which falls within the range of pH of most agricultural by products, Volatile matter 11%, Carbon content 68%.and 15% Moisture content was recorded. The surface area from the results obtained was found to be 975m²/g. Scanning Electron Microscopy (SEM) technique was employed to observe the surface physical morphology of the groundnut shell-derived activated carbon. According to the SEM micrographs the raw material before chemical activation does not have porous surface, while the external surface of the chemically activated carbon is rich with cavities. The FT-IR analysis results indicate that only minor differences between the samples could be established. However, shift in bands and changes in wavelength numbers and absorbance between the untreated and treated samples indicate that chemical transformation would have taken place during chemical treatment.

Keywords: Activated Carbon, Adsorbent, Agricultural waste, metals, raw water

INTRODUCTION

World demand for virgin activated carbon is forecast to expand by 9 % per annum through 2014 to 1.7 million metric tons (Ramakrishna, 2012). Due to the increasing demand of A.C, there is a strong need for the sorting out new precursors for the preparation of A.C which should be cost effective. Although, a variety of raw materials were explored for the preparation of A.C in earlier studies, agricultural wastes such as coconut shells (Azevedo et al., 2007; Hu and Srinivasan, 2001), wood (Gomez-Serrano et al., 2005; Klijanienko et al., 2008; Zuo et al., 2009), cotton stalk (Deng et al., 2010), almond shells (Bansode et al., 2003), rice husk (Fierro et al., 2010; Guo and Rockstraw, 2007), date pits (Girgis and El-Hendawy, 2002) were used for the production of activated carbon. Scientists are still trying to explore new materials depending on their availability and suitability for A.C production. However, the utilization of agricultural wastes as raw material for

the preparation of activated carbon has increased notably in recent years.

Activated carbon has been explained in different ways by several authors. Marsh (1989) defined activated carbon as porous carbon, which have been treated by oxidizing gases during or after carbonization in order to increase porosity. While according to Bansode (2003) stated that activated carbon is predominantly an adsorbent with a large internal pore volume and surface area. Activated carbon can be prepared from many carbonaceous materials including municipal and industrial wastes and forest and agricultural by-products (Hu *et al.*, 2003; Hu and Srinivasan, 2001; Tseng, 2007; Tseng *et al.*, 2006; Wu and Tseng, 2006). Activated carbon can be produced in two different processes from a variety of carbonaceous source materials. It can be produced using one of the following processes either physical or chemical activation. Chemical activation

of activated carbon has been reported as more advantageous over physical activation due to higher yields, more surface area and better development of porous structures in carbon (Lua and Gua, 2001). The high adsorptive capacities of activated carbons are highly related to porous characteristics such as surface area, pore volume, and pore size distribution. All activated carbons have a porous structure, containing up to 15 % of mineral matter in the form of ash content (Bansal et al., 1988).

Characteristics of activated carbon depend on the physical and chemical properties of the raw materials as well as method of activation (Lua and Gua, 2001). Generally, the raw materials for the production of A.C are those with high carbon but low inorganic contents such as wood, lignite, peat and coal (Lua and Guo, 2001). Besides that, lots of agricultural waste and by product have successfully converted to A.C for examples macadamia nutshell (Ahmadpour and Do, 1997). In Nigeria there are potential raw materials resources for the production of the activated carbon. In this research, a local agricultural waste which is groundnut shells is used to produce an activated carbon using chemical activation and subsequently applied to remove iron and manganese in raw water.

MATERIALS AND METHODS

Raw Material

The carbonaceous precursor used for the preparation of activated carbon (AC) is Groundnut shell and was collected from the premises of Tashan Bama (Bama Motor Park), Maiduguri Borno state. Prior to use, precursor was washed gently with water to remove mud and other impurities present on the surface and then sundried. The dried sample was grounded followed by sieving with a 2mm mesh size sieve.

Chemicals and Impregnating Agent

All the chemicals used were AR-grade chemicals and were used without further purification. The impregnating agent used for the chemical activation of groundnut shells was H_3PO_4 .

Carbonization and Chemical Activation

The sample materials were carbonized in the absence of air in a muffle furnace at a temperature of 550-1000°C for 60 minutes and the 200g of carbonized sample was mixed with an aqueous solution of phosphoric acid (activating agent). The mixture was then subjected to heat at a temperature of 120°C for 3 hours to vaporize the water. The dried mixture was subjected to heat at a temperature of 650°C in a

muffle furnace to enable activation of the pores of the carbon sample.

Characterization of A.C

Various properties of the prepared activated carbon were characterized by following the standard procedures. The pH was measured using a pH meter, moisture content was found by oven-drying test method (ASTM D2867 – 09), The percentage of volatile matter of the AC sample was determined by the standard method (ASTM D5832 – 98) for the ash content (ASTM D2866 – 94) method was followed. The bulk density of activated carbon was determined by following the method described by Ahmedna et al., (2000) using a measuring cylinder. Electrical conductivity was measured using Ecoscan conductivity meter (Eutech, Singapore), and values were presented in micro Siemens $\mu S/cm$ as described by (Ahmedna et al., 2000).

Determination of the adsorption capacity:

2g of activated carbon was interacted with 100ml of raw water sample in a flask and this was allowed to stand for an equilibrium adsorption time of 60 minutes. It was then filtered using What-man filter paper. The concentration of manganese and iron in the water was determined before and after interaction with the activated carbon by a smart absorption spectrophotometer. The adsorption capacity was calculated using the equation below.

$$Q_t = (C_i - C_t) V/W \text{ -----(1)}$$

Q_t = adsorption capacity at time t (mg/g).

C_i = concentration of metal before interaction with the activated carbon (mg/L).

C_t = concentration of metal after interaction with the activated carbon (mg/L).

V = volume of the effluent (L).

W = weight of the activated carbon (g). (Hameed, 2009).

Particle Size Distribution

20g of the activated carbon was sieved using sieves of different sizes (2mm, 1mm, 850 μm , 710 μm , 500 μm , 425 μm , 300 μm , 250 μm , 150 μm , 63 μm , and pan) arranged in descending order on an electric vibrator, to vibrate the sample for a period of 5minute. Thereafter, the sieve fraction was collected and weighed for each size.

Surface Area

The specific surface area of activated carbon was estimated using sear method by agitating 1.5g of activated carbon samples in 100ml of dilute hydrochloric acid at pH=3. Then a 30g of sodium chloride was added while stirring the suspension and then the volume was made up to 150ml with deionised water. The solution was titrated with 0.1N NaOH to rise the pH from 4 to 9 and volume V recorded.

The surface area according to this method was calculated as:

$$S = 32V - 25 \text{-----} (2)$$

Where S = surface area of activated carbon, V = volume of sodium hydroxide required to raise the pH sample from 4 to 9.

Scanning Electron Microscope (SEM)

The surface morphological changes of activated carbon samples were investigated using a Scanning

Electron Microscope (SEM, Make: JEOL, Japan) operated at 25kV.

Fourier Transform Infrared Spectroscopy (FTIR) analysis

The samples of about 1 mg were grinded and milled with 100 mg KBr to form a fine powder. This powder was then compressed into a thin pellet under 7 tons for 5 minutes. The sample was then analyzed using Shimadzu 8300 spectrometer and the spectrum was recorded in a spectral range.

RESULTS AND DISCUSSION

Results

Adsorbent was characterized for its proximate and ultimate analysis (Table: 1). from the proximate analysis, it was observed that moisture, ash and volatile matter % was high which may be due to its plant origin. Surface area is very high for groundnut shell based adsorbent which shows its high adsorption capacity..

Table: 1. Characterization of the groundnut shell AC

S/No	Parameters	Results
1	Ph	6.8
2	Moisture content (%)	15
3	Volatile matter (%)	11
4	Ash content (%)	6
5	Carbon content (%)	68
6	Bulk density(g/L)	420
7	Electrical conductivity(μS/cm)	824
8	Surface area(m ² /g)	975
9	Adsorption capacity of Mn and Fe(mg/g)	55.5 and 130.5
10	% adsorbed of Mn and Fe	84. and 90

Table: 2. Shows the Result Of Particle Size Distribution

Mesh sizes(mm or μm)	Particle size distribution
2mm	0.08
1mm	2.03
850	2.25
710	2.46
500	6.44
425	2.52
300	2.34
250	0.86
150	0.19
63	0.00
Pan	0.00

SEM Analysis of the AC

To study the effect of activation on porosity Scanning Electron Microscopy (SEM) technique was employed to observe the surface physical morphology of the groundnut shell-derived activated carbon. Figure

below shows the SEM micrographs of the raw material before chemical activation after carbonization and the chemically prepared activated carbon.

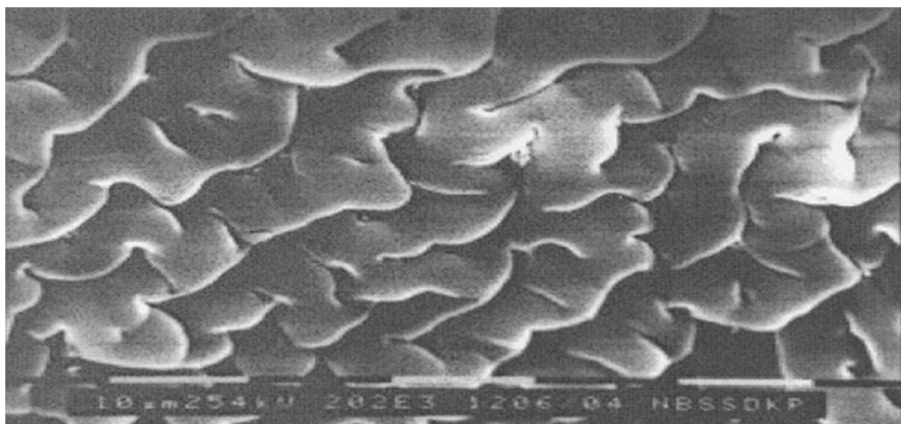


Fig 1. SEM Micrograph of Groundnut Shell Based Char before Activation

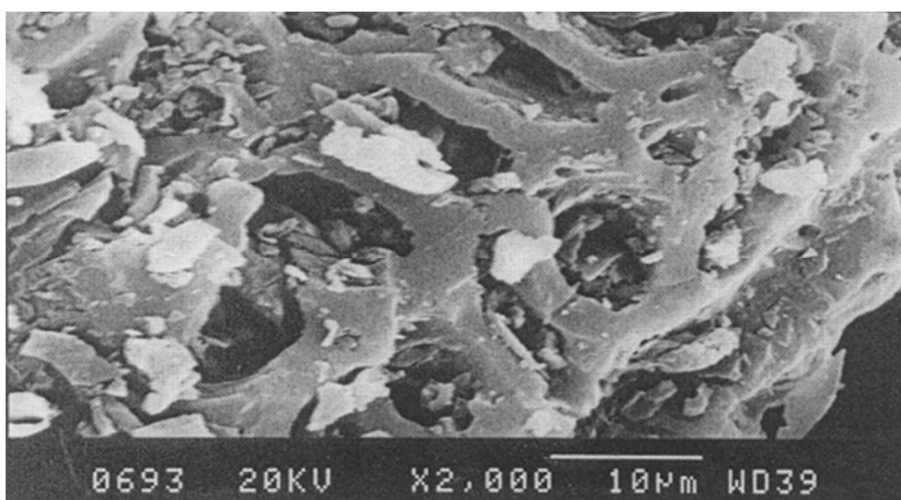


Fig 2. SEM Micrograph of Groundnut Shell Based Activated Carbon after Chemical Activation.

FTIR Analysis of Activated Carbon The FT-IR analysis results indicate that only minor differences between the samples could be established.

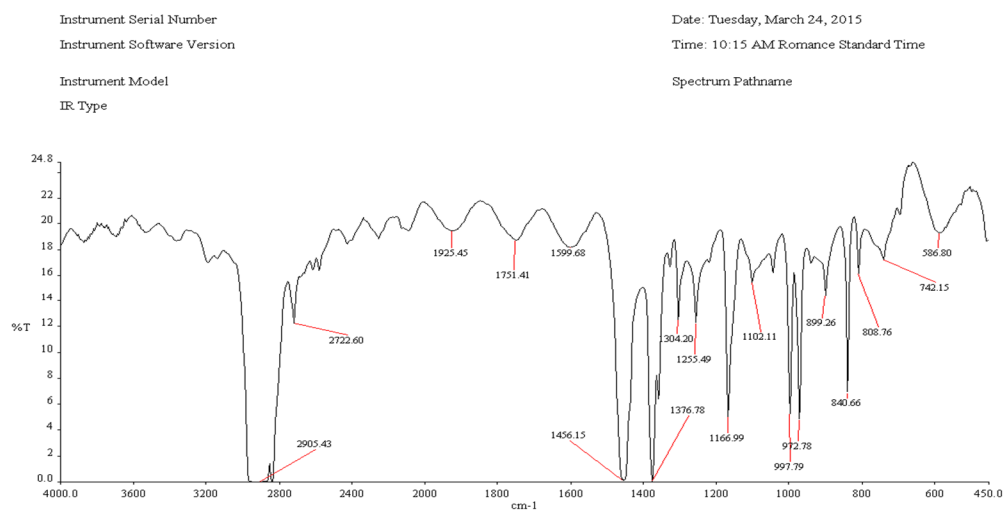


Fig 3. Result of the FTIR Analysis of untreated A.C

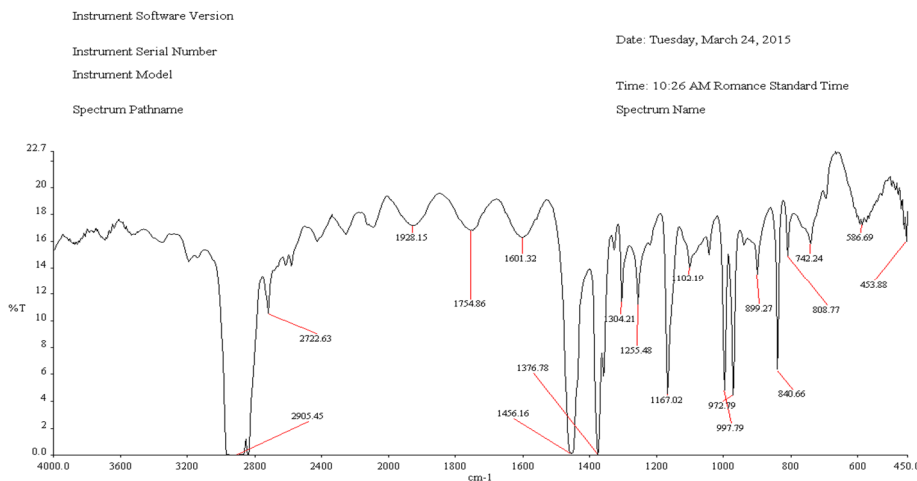


Fig 4. Result of the FTIR Analysis of Chemical A.C

DISCUSSIONS

The pH of the produced activated carbon was found to be 6.8, which falls within the range of pH of most agricultural by products. Generally, adsorbents with pH of 6-8 are acceptable in most applications (Martinez *et al* 2003).

The moisture content in this work was found to be 15%. Moisture content, according to Aziza *et al.*, (2008) has a relationship with porosity (α) of a given carbon. Adsorbent with high moisture content is expected to swell less, thus retarding pore size expansion for adsorbate uptake.

Volatile matter of 11% was recorded in this study. This is responsible for decomposition of the organic materials to release volatiles and development of microporous structures (Malik *et al.*, 2006). Report has shown that low volatile matter content implies the high porosity of the adsorbent since volatile matter remains clogging in the carbon pores (Olowoyo and Orere 2012).

Ash is a measure of inorganic impurities in the carbons (Bansode *et al.*, 2003). The ash content in this research was found to be 6% which is in range of most ash content of agricultural waste.

Carbon content is the residual amount of carbon present in the sample. The result obtained shows that carbon content is 68%. This is in concord with the findings of Malik *et al.*, (2006) who reported that most of the carbon composition of A.C falls within 50-90%.

The density of a generated activated carbon plays a great role on adsorbate uptake. Generally, higher

density carbons hold more adsorbate per unit volume (Jibril, *et al.*, 2007). According to Itodo *et al.*, (2008) bulk density has impact on adsorbate-retention level by any adsorbent.

The role played by electrical conductivity has a direct link to the nature of activating agent (Itodo, 2008). Generally, high conductivity of carbon adsorbents is undesirable in the adsorption process of nonionic pollutants (Bansode *et al.*, 2003). This is because it interferes with the leachable minerals associated with the carbon surface.

The surface area from the results obtained was found to be 975m²/g. Sometimes there is variation of surface area of a material if a different mode of activation is followed. The variation in surface area is as a result of chemical added.

According to the SEM micrographs the raw material before chemical activation does not have porous surface, while the external surface of the chemically activated carbon is rich with cavities SEM micrograph of char shows the presence of some flaky structure and rudimentary pores while SEM micrograph of activated carbon shows the presence of wide pores which result due to chemical activation with acid which is highly corrosive and dehydrating in nature.

Fourier Transform Infrared Spectroscopy (FTIR) analysis was carried out in order to identify the functional groups present in activated carbon. Functional groups of adsorbents not only affect the adsorption behaviour, but also dominate the adsorption mechanism (Zheng *et al.*, 2014). The

peaks appearing in the FTIR spectrum were assigned to various functional groups according to their respective wave numbers. Figure 3 and 4 shows the FTIR spectrum for the untreated and the chemically treated spectra peaks representing phenols, carboxyl and carbonyl were observed in 2905.45 cm⁻¹, 2722.63 – 1456.16 cm⁻¹, and 1376.78 – 997.79 cm⁻¹, for chemical A.C while for the untreated AC the peaks were observed in 2905.43cm⁻¹, 2722.60 – 1456.15 cm⁻¹, and 1376.78 – 997.79cm⁻¹, respectively.

CONCLUSION

Production and characterization of activated carbon prepared from groundnut shell, an abundantly available carbonaceous solid waste from Maiduguri by chemical activation were carried out. The production was done first by carbonization of the groundnut shell, impregnating the prepared raw material with activating reagent, and then activation in a muffle furnace. Characteristics of the activated carbons were determined using standard methods. The characterization of the adsorbents includes estimation of various parameters such as moisture content, ash content, and carbon content, electrical conductivity, bulk density, particle size, surface area, pH, adsorption capacity, SEM and FTIR analysis, where carried out. Changes in surface morphology of char before and after activation were examined by scanning electron microscopy which showed well developed pore structure in activated carbon demonstrating the effect of chemical activation with H₃PO₄. The surface area of produced A.C from groundnut shell was found to be **975m²/g**. The FT-IR analysis results indicate that only minor differences between the samples could be established. However, shift in bands and changes in wavelength numbers and absorbance between the untreated and treated samples indicate that chemical transformation would have taken place during chemical treatment or during pyrolysis. FTIR spectrum for the untreated and the chemically treated spectra peaks representing phenols, carboxyl and carbonyl were observed in 2905.45 cm⁻¹, 2722.63 – 1456.16 cm⁻¹, and 1376.78 – 997.79 cm⁻¹, for chemical A.C while for the physical or untreated AC the peaks were observed in 2905.43cm⁻¹, 2722.60 – 1456.15 cm⁻¹, and 1376.78 – 997.79cm⁻¹, respectively.

REFERENCES

- Ahmadpour, A. and Do. D., (1997) Production and Characterization of Activated Carbon from Peanut Shell using steam activation, 35(12), 1723
- Ahmedna, M; Marshall, W and Rao, M. (2000). Production of Granular Activated Carbon from selected Agricultural by-products. *Bioresources technology* 7(2): p113-123
- Azevedo, D.C.S., Araujo, J.C.S., Bastos-Neto, M., Torres, A.E.B., Jaguaribe, E.F., and Cavalcante, C.L. (2007). Microporous Activated Carbon Prepared From Coconut Shells Using Chemical Activation With Zinc Chloride. *Microporous And Mesoporous Materials*, 100, 2007, 361-364.
- Aziza, A; Odiakosa, A, Nwajei, G. Orodu, V. (2008). Modification and Characterization of Activated Carbon Derived from Sawdust. Conference Proceeding, *CSN Delta Chem.* 2008: 235-243
- Bansal, R.C., Donnet, J.B. and Stoeckli, H.F. (1988). *Active Carbon*. Marcel Dekker, 1988, New York.
- Bansode, R.R., Losso, J.N., Marshall, W.E., Rao, R.M., and Portier, R.J. (2003). Adsorption Of Volatile Organic Compounds By Pecan Shell And Almond Shell Based Granular Activated Carbons. *Bioresource Technology*, 90, 2003, 175-184.
- Deng, H., Li, G., Yang, H., Tang, J. and Tang, J. (2010). Preparation of activated carbons from cotton stalk by microwave assisted KOH and K₂CO₃ activation. *Chemical Engineering Journal*, 163, 2010, 373-381.
- Fierro, V., Muniz, G., Basta, A.H., El-Saied, H. and Celzard, A. (2010). Rice Straw as Precursor of Activated Carbons: Activation with Ortho-Phosphoric Acid. *Journal of Hazardous Materials*, 181, 2010, 27-34.
- Girgis, B.S. and El-Hendawy, A.N.A. (2002). Porosity development in activated carbons obtained from date pits under chemical activation with phosphoric acid. *Microporous and Mesoporous Materials*, 52, 2002, 105-117.
- Gomez-Sarrano, V., Cuerda-Correa, E.M., Fernandez-Gonzalez, M.C., Alexandre-Franco, M.F. and Macias-Garcia, A. (2005). Preparation of activated carbons from chestnut wood by phosphoric acidchemical activation. Study of microporosity and fractal dimension. *Materials Letters*, 59, 846-853.
- Guo, Y. and Rockstraw, D.A., (2007). Activated carbons prepared from rice hull by one-step phosphoric acid activation. *Microporous and Mesoporous Materials*, 100, 2007, 12-19.
- Hu, Z., and Srinivasan, M.P. (2001). Mesoporous high-surface-area activated carbon. *Microporous and Mesoporous Materials*, 43, 2001, 267-275.
- Hu, Z., Lei, L., Li, Y., Ni, Y. (2003), Chromium adsorption on high-performance activated carbons from aqueous solution. *Separation and Purification Technology*, 31, 13-18.
- Itodo, A.U (2008). Comparative studies on the preparation, adsorption and evaluation of granular grades activated carbon from selected animal and plant coasts. A Ph.D proposal, *UDUSOK* p69-74
- Jibril, B; Maamari, R; Houache, O; Aamri, M. & Qalhati, M. 2007. Effect of H₃PO₄ and KOH on Pyrolysis of Bituminous Coal in Preparation of Activated Carbon. *Journal of applied sciences Research*. 3 (11), 2007: p1343 – 135.
- Klijanienko, A., Lorenc-Grabowska, E. and Gryglewicz, G.(2008). Development of Mesoporosity during Phosphoric Acid Activation of Wood in Steam Atmosphere. *Bioresource Technology*, 99 (15), 2008, 7208-7214.

- Lua A. C. ,and Gua J., (2001). Microporous oil-palm shell A.C prepared by physical activation for gas phase adsorption, *lagmuir* 17, 7112-7117
- Malik, R; Ramteke, D., Water, S (2006). Physico-chemical and surface characterization of adsorbent prepared from groundnut shell by ZnCl₂ activation and its ability to adsorb colour. *Indian journal of chemical Technology*. 13: p329-333
- Marsh H., D.S. Yan, T.M. O'Grady. A. Wennerberg. (1984). Formation of active carbons from cokes using potassium hydroxide. *Carbon*, 22, 603-611
- Martinez M. L., Moiraghi L., Agnese M and Guzman C. (2003). Making and Some Properties of Activated Carbon Produced From Agricultural And Industrial Residues From Argentina. *The Journal of the Argentine Chemical Society* Vol. 91, No. 416, Pp103-108.
- Olowoyo D. N and Orere E. E (2012). Preparation and characterization of A.C. made from Palm kernel shell, coconut shell, groundnut shell and Obechi wood. Investigation of apparent density, total ash content, moisture contents and particle size distribution. *International journal of Research in chem. And environment*. Vol. 2 issue 3, pp32-35. ISSN 2248-9649
- Ramakrishna Gottipati, and Susmita Mishra. (2012). Application of response surface methodology for optimization of Cr(III) and Cr(VI) adsorption on commercial activated carbons, *Research Journal of Chemical Sciences*, 2(2), 2012, 40-48.
- Wu, F.C., Tseng, R.L.(2006). Preparation of highly porous carbon from fir wood by KOH etching and CO₂ gasification for adsorption of dyes and phenols from water. *Journal of Colloid and Interface Science*, 294, 2006, 21–30.
- Zheng X., Jin X., Zhenzhen W and Weihao Li (2014). Removal of iron and manganese in steel industry drainage by biological activated carbon. *Desalination and water treatment*. www.destwater.com doi: 10.1080/19443994.2014-963682
- Zuo, S., Yang, J., Liu, J. and Cai, X. (2009). Significance of the carbonization of volatile pyrolytic products on the properties of activated carbons from phosphoric acid activation of lignocellulosic material. *Fuel Processing Technology*, 90, 2009, 994-1001.