

Synthesis and Characterization of Activated Carbon Prepared from Jute Stick Charcoal for Industrial Uses

M. Nurul Islam^{1*}, Jahid Sarker², Ayesha Khatton², S. M. Mahruf Hossain², Helena Akhter Sikder³, Rashed Ahmed³, A. M. Sarwaruddin Chowdhury³

¹Chief Scientific Officer, Chemistry Division, Bangladesh Jute Research Institute, Manik Mia Avenue, and Dhaka-1207, Bangladesh

²Chemistry Division, Bangladesh Jute Research Institute, Dhaka-1207, Bangladesh

³Department of Applied Chemistry and Chemical Engineering, Dhaka University, Dhaka-1000, Bangladesh

DOI: [10.36348/sijcms.2022.v05i03.003](https://doi.org/10.36348/sijcms.2022.v05i03.003)

| Received: 17.02.2022 | Accepted: 21.03.2022 | Published: 30.03.2022

*Corresponding author: Dr. M. N. Islam

Chief Scientific Officer, Chemistry Division, Bangladesh Jute Research Institute, Manik Mia Avenue, and Dhaka-1207, Bangladesh

Abstract

Activated Carbons (ACs) were prepared from jute stick charcoal by chemical activation using H₂SO₄ and H₃PO₄ ranging temperature from 300^oC to 350^o. The activated carbons and charcoal prepared from jute sticks were characterized by evaluating the surface chemistry, structural features and surface morphology. The properties of the carbons were characterized by Scanning Electron Microscope (SEM), Brunauer – Emmett-Teller (BET), X-Ray Diffraction (XRD) and the FT-IR method. The jute sticks were converted to activated carbons with the highest surface area (135–245m²/g) and largest mesopores volume (0.14–0.16 cm³/g). The FT-IR spectra exhibited that the pyrolysis of jute stick resulted in the release of aliphatic and O - containing functional groups by thermal effect. However, the release of functional groups is effect of chemical reaction in the ZnCl₂, H₃PO₄, and H₂SO₄ activation process. A honeycomb carbon structure in activated carbon was formed as observed on SEM images. Although charcoal and activated carbon were prepared at 300^oC to 350^o, the activated carbon exhibited much lower Raman sensitivity due to the formation of condensed aromatic ring systems. Due to high surface area and high porous structure with abundance of functional groups, the activated carbon prepared from jute sticks charcoal absorbed molecules with much higher than those of other activated carbon.

Keywords: Jute Stick, Charcoal, Activated Carbon, Surface Area, Characterized.

Copyright © 2022 The Author(s): This is an open-access article distributed under the terms of the Creative Commons Attribution 4.0 International License (CC BY-NC 4.0) which permits unrestricted use, distribution, and reproduction in any medium for non-commercial use provided the original author and source are credited.

INTRODUCTION

Bangladesh is the second largest producer of jute in the world. Jute is one of the major cash crops in the country. The production of charcoal from jute stick has created new possibilities for multifaceted use of jute (Islam *et al.*, 2022). In Bangladesh, jute sticks are used as burning resource in the rural area (Martin *et al.*, 2003). There is very little commercial usage of jute sticks in Bangladesh. The jute stick can be used as raw material of charcoal. About 40 lakh MT of jute sticks are produced against 16 lakh MT of jute, which used to fall as agricultural waste. Most of the jute sticks produced in the country used for cooking fuel in the villages, tents for battle leaves and for household purposes. The abundance and availability of agricultural by-products make them good sources of raw materials for activated carbon production (Malik *et al.*, 2007).

Activated carbon production from agriculture waste materials has attracted researchers in recent years (Prahas *et al.*, 2008; Ioannidou *et al.*, 2007). Finding cheap raw materials for producing activated carbon is a difficult task. That's why different agricultural waste materials like corn cob, coconut shell, palm shell (Daud *et al.*, 2000), apple pulp (Sua' rez-Garci'a *et al.*, 2002) chickpea husks (Hayashi *et al.*, 2000), grain sorghum (Diao *et al.*, 2002), pistachio nutshell (Lua *et al.*, 2004), jute fiber (Senthilkumaar *et al.*, 2004), olive stones and walnut shell (Martinez *et al.*, 2006), cherry stones (Olivares *et al.*, 2003), coir pith (Ash *et al.*, 2006), wild rose seeds (Gurses *et al.*, 2006), rice bran (Suzuki *et al.*, 2007), gopher plant (Ozgul *et al.*, 2007), oil palm shell (Tan *et al.*, 2007) rubber tree seed coat (Hameed *et al.*, 2008), cotton stalk (Deng *et al.*, 2009) examined to produce AC. The production of AC from jute is one of the promising ways now a days for fulfilling the crisis

of raw material. But ACs is highly adsorbents due to their high surface area, controllable pore size distribution, and amendable surface activity. AC production is involved in two stages at first carbonization and then activation. Carbonization removes the non-carbon elements (Diao *et al.*, 2002). The porosity of the charcoal that find after activation is not then completely accessible. Lignocellulose-based fiber like jute can also be converted into carbon materials through pyrolysis then chemical activation by different chemicals. Generally jute stick converted into charcoal through pyrolysis and charcoal then activated to prepare carbon materials. Over the last several years, considerable research activities have been dedicated to the jute-derived carbon (JC), including nano-carbon production through direct pyrolysis to tune the characteristics of carbon materials, such as morphology, pore characteristics, surface functionality, and surface area, to improve its capability for practical applications. The purpose of the activation process is to increase the pore structure of the charcoal. Activation can be done in two ways. Physical or thermal activation completes through steam, carbon dioxide, mixture of these. Chemical activation involves activating the charcoal by chemical agents like H_3PO_4 , $ZnCl_2$, H_2SO_4 , KOH ,

$FeCl_3$, etc. AC with well-developed porosity (after appropriate washing) may be obtained in a single operation. This paper contains the preparation of Activated carbons from jute stick charcoal by using activating agents H_3PO_4 , H_2SO_4 for using in various industrial sectors.

MATERIALS AND METHODS

Materials

Jute sticks used in this study were provided by Bangladesh Jute Research Institute (BJRI). The whole experimental works had been carried out in the Industrial Chemistry Laboratory, Chemistry Division, Bangladesh Jute Research Institute (BJRI), Dhaka and Applied Chemistry & Chemical Engineering Department of Dhaka University.

Chemicals

Activated carbon preparation chemicals such as H_2SO_4 and H_3PO_4 were procured from local market in Bangladesh. All reagents were laboratory grade and were used without further purification. Table 1 shows the preparation of activated carbon from jute stick charcoal

Table 1: Preparation of activated carbon from jute sticks charcoal

Sample No.	Activated carbon with chemicals
S-1	Activated carbon preparation with 10% H_2SO_4
S-2	Activated carbon preparation with 10% H_3PO_4

Methods

Charcoal Preparation

The jute stick as received was first washed with distilled and deionized water and dried in an oven at $85^\circ C$ for one day. Then the washed jute stick was grounded in a coffee mill, and the fraction passing through 50 mesh US Standard sieve was used in the preparation. After that, it was kept in a muffle furnace at $300^\circ C \sim 350^\circ C$ for 3 hrs. After that, prepared charcoal powder as stored in a desiccator. When charcoal was at room temperature, it was taken from the desiccator, and weight was calculated. Then, Analysis of fixed carbon (SNI 13-3479-1994), ash content (SNI

3478: 2010), volatile matter: (SNI 13-3999-1995), moisture in the charcoal (SNI 13-3477-1994) was calculated and shown in Table 2.

Table 2: Composition of charcoal powder from jute stick

SI No	Name of the content	Result
1	Fixed Carbon	71.86%
2	Moisture	5.31%
3	Ash Content	4.73%
4	Volatile Matter	18.10%



Fig 1: Preparation of activated carbon from jute sticks charcoal

Preparation of Activated Carbon from jute stick charcoal with chemicals

At first, 10% sulfuric acid was prepared. Then by maintaining Charcoal: Acid = 1:10, charcoal powder soaked with 10% sulfuric acid solution. The charcoal soaked with the sulfuric acid sample was kept at room temperature and it was kept for 5 ~ 6 hrs. Soaked samples were washed with distilled water to avoid an

excessive amount of acid solution. Then pH was checked to ensure neutrality. After that sample was filtered by a vacuum dryer and then the sample was dried in an oven at a temperature of 70°C to 80°C. The dried sample was collected and weighed in the electric balance. In the same way, Activated Carbon was prepared from 10% phosphoric acid.



Fig 2: Activated carbon from charcoal with H₂SO₄



Fig 3: Activated carbon from charcoal with H₃PO₄

Surface Morphology

Surface morphology measurements were carried out on the samples using a Leo-JEOL Scanning Electron Microscope. The activated carbon samples were coated with gold by a gold sputtering device for clear visibility of the surface morphology.

X-ray Diffraction Technique

X-ray diffraction experiments were performed with a Philips X'pert diffractometer for 2θ values from 10 to 80° using Cu K α radiation at a wavelength of λ) 1.54060 Å. The other experimental conditions included $1/2^\circ$ divergence slits, a 5-s residence time at each step, and intensity measured in count.

Adsorption-Desorption

N₂ adsorption-desorption isotherms of the activated carbons were measured using an automatic adsorption instrument (Quantachrome Corp., Nova-1000 gas sorption analyzer) for the determination of the surface area and the total pore volumes. Prior to measurement, the carbon samples were degassed at 120°C overnight. The nitrogen adsorption-desorption data were recorded at liquid nitrogen temperature, 77 K. The total pore volume was estimated to be the liquid volume of adsorbate at a relative pressure of 0.99. All surface area measurements were calculated from the nitrogen adsorption isotherms by assuming the area of the nitrogen molecule to be 0.162 nm².

RESULTS AND DISCUSSION

Yield percentage of AC

In activated carbon preparation, the yield is usually defined as the final weight of activated carbon divided by the initial weight of raw material, both on a dry basis (Kim *et al.*, 2001). The activated carbons yield, R (%) was calculated using the following formula: $R (\%) = m/m_0 \times 100$. Where, m and m_0 are

the dry weight of final activated carbon and dry weight of precursor, respectively.

Different things were observed from the yield percentage of activated carbon stable. The yield percentage value was better if AC was prepared by activation with H_2SO_4 than H_3PO_4 two samples.

Table 3: Yield percentage of activated carbons

Sample Id	Time(hr)	Temperature (°C)	Activation Burn off (wt %)	Yield (wt %)
S-1	3	350	6	94
S-2	3	350	14	86

Surface Area and Pore Volume

The specific surface area was calculated using the BET model. The mesopore surface area was calculated by the *t*-plot method. The BET surface area increased considerably after impregnation and

activation at higher temperatures. The increase in surface area can be attributed to the release of certain volatile components as a result of the acid treatment on the precursor material containing organic and inorganic materials over the temperature range of 700-900 °C.

Table 4: BET analysis results of activated carbons

Sample No.	Average Pore Diameter (4V/S)	Porosity based on skeletal density of 1.0000 g/cc	BET Surface Area	Total Pore Volume
S-1	42.12 Angstroms	0.13 per gram of sample	135.99 m ² /g	0.14 cc/g
S-2	25.48 Angstroms	0.14 per gram of sample	245.91 m ² /g	0.16 cc/g

X-ray Studies

It is a general fact that the creation of porosity is considerably influenced by various factors such as active-site clustering or fusion, carbon structure, inorganic impurities, and gas diffusion. Gundogdu *et al.*, indicated that the internal structure of the carbon is considered to be the most important among these factors. The X-ray diffractograms of the two samples S-1 and S-2 are shown in Figure 4. Sample 1 indicates the presence of completely amorphous silica by the appearance of a broad peak centered at the 2θ angle of 22°. Sample S-2, in contrast, contains a mixture of amorphous and crystalline phases of silica. The

temperature employed for the activities of the precursor material can be cited as the reason for the formation of the small graphitic structures and for the crystallization of silica. The one interesting fact is that the peak at $2\theta \approx 44^\circ$ is very small, indicating that the pores are also created by the decomposition of carbon structures along the *a* direction of the graphitic structures. It also clearly suggests that precarbonization of the raw material before chemical activation produces relatively well organized aromatic carbons with sp^2 bonding character that are more stable than the amorphous-like carbons of sp^3 bonding character.

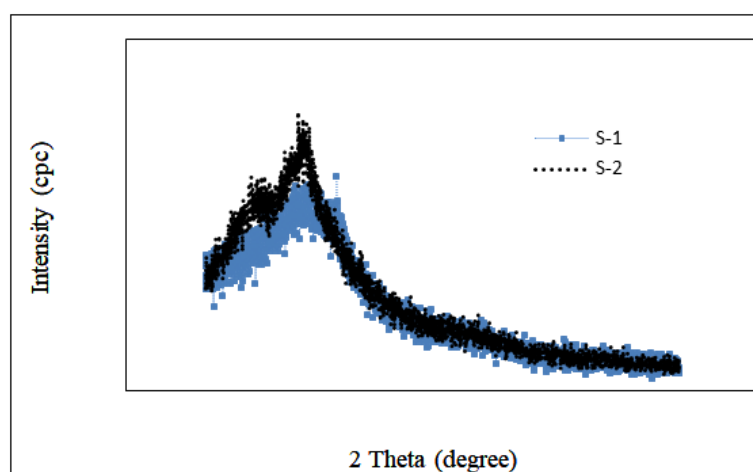


Fig 4: X-ray diffraction curves for S-1 (Activated carbon from charcoal with H_2SO_4) and S-2 (Activated carbon from charcoal with H_3PO_4)

FTIR analysis

FTIR is perhaps the most powerful tool for identifying types of chemical bonds (functional groups). The jute stick, impregnated sample with chemical agents and activated carbons (ACs) were analyzed by FTIR to clarify the structural changes after the activation and carbonization processes. The assignments of the absorption bands are listed in Table 5. The FTIR spectrum of S-1 and S-2 are shown in Fig 5. The broad and intense absorption peaks in the 3700–3100 cm^{-1} correspond to the O-H stretching vibrations of cellulose, pectin, absorbed water, hemicellulose, and lignin (Gundogdu *et al.*, 2013) The presence of the peak

at 1740-1700 cm^{-1} in the spectrum indicates the carbonyl (C=O) stretching vibration of the carboxyl groups of pectin, hemicellulose, and lignin in A, B and C, of activated carbon (Hameed *et al.*, 2008) The peaks around 1620 to 1580 cm^{-1} are due to the C=C stretching that can be ascribed to the presence of aromatic in lignin of jute stick. The bands in the extent of 1300–1000 cm^{-1} can be attributed to the C-O stretching vibration of carboxylic acids and alcohols (Angin *et al.*, 2014). The band in between 700 and 900 cm^{-1} contains different peaks assigned to aromatic out of plane C-H bending with varying degrees of substitution.

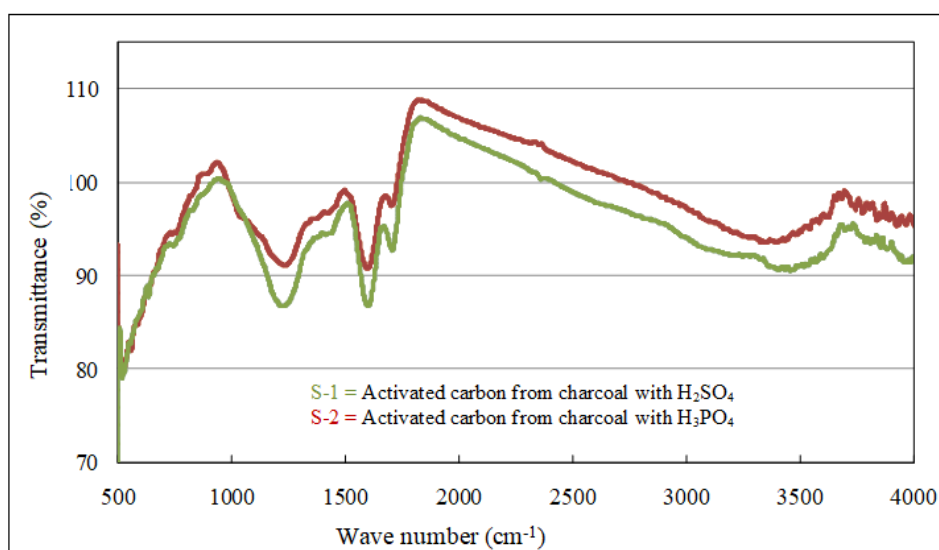


Fig 5: FT-IR spectra of activated carbon from jute stick charcoal

Table 5: Peak assignments of functional group of charcoal and AC from different chemical activation

Wave number (cm^{-1})	S-1	S-2
3600-3400 O-H stretching	3354	3456
1740-1700 C=O stretching	1701	1706
1620-1580 Aromatic C=C stretching	1594	1597
1300-1000 C-O stretching	1225	1217
900-700 stretching of C-H Group in alkane, alkene and aromatic group	744	630

Surface Morphology

Figure 6 shows the morphology of the samples before and after phosphoric acid activation. In the sample 1, there is no evidence of pore formation in the jute stick where only carbonization of the raw material takes place without creating pores. In contrast, sample 2 indicates the formation of pores due to chemical activation. It is made very clear that the opening of the pores in the surface of the rice husk should be due to the extraction is made very clear that the opening of the

pores in the surface of the rice husk should be due to the extraction of some materials, e.g., dissolution of lignin's and other mineral components from the husk during the impregnation process, so as to create, upon activation, micro- and mesopores in the carbon and silica components. As a result of the creation of pores, there is an increase in both the surface area and the pore volume, which are stably created in the carbon composite.

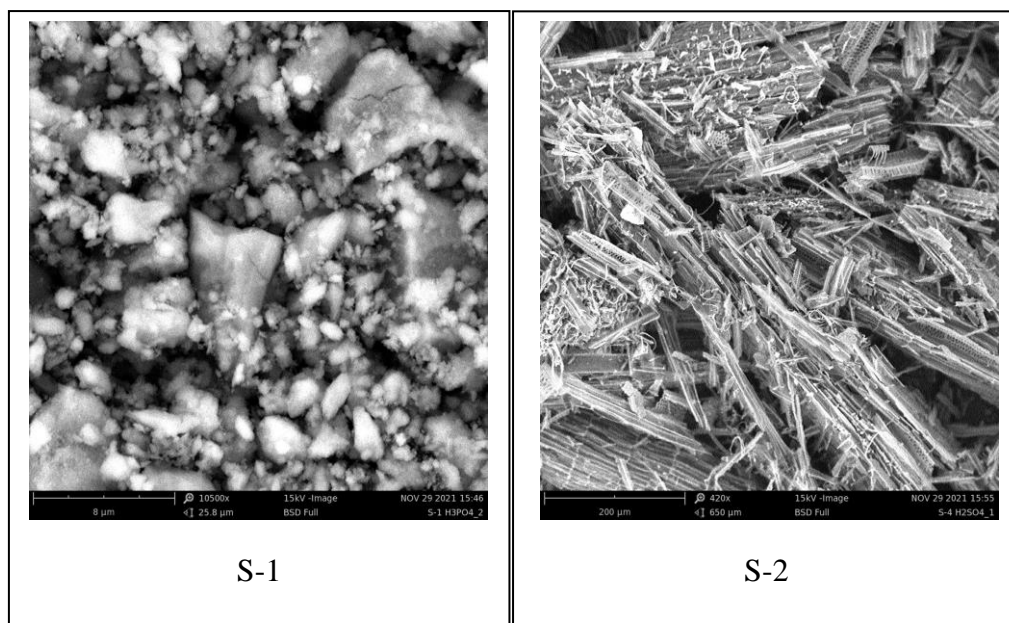


Fig 6: Microscopically view of S-1 and S-2

CONCLUSION

There is the difficulty in analyzing the chemical properties of the surface derived from the special nature of activated carbon itself, which is a black body and has complex components on the surface, so the different characterization methods were proposed and used. Further research activities need to be conducted from laboratory scale to industrial or commercial production and application for getting the highest utilization of jute fiber. We expect that charcoal and AC from jute will certainly improve the quality of life of people with the development of next-generation materials for advanced applications. The field is very promising because the availability of raw materials is very common and cheap.

REFERENCES

- Islam, M. N., Khatton, A., Sarker, J., Sikder, H. A., & Chowdhury, A. S. (2022). Preparation and Characterization of Activated Carbon from Jute Stick by Chemical Activation: Comparison of Different Activating Agents. *Saudi J Eng Technol*, 7(2), 112-117.
- Malik, R., Ramteke, D. S., & Wate, S. R. (2007). Adsorption of malachite green on groundnut shell waste based powdered activated carbon. *Waste management*, 27(9), 1129-1138.
- Martin, M. J., Artola, A., Balaguer, M. D., & Rigola, M. (2003). Activated carbons developed from surplus sewage sludge for the removal of dyes from dilute aqueous solutions. *Chemical Engineering Journal*, 94(3), 231-239.
- Prahas, D., Kartika, Y., Indraswati, N., & Ismadji, S. J. C. E. J. (2008). Activated carbon from jackfruit peel waste by H₃PO₄ chemical activation: Pore structure and surface chemistry characterization. *Chemical Engineering Journal*, 140(1-3), 32-42.
- Ioannidou, O., & Zabaniotou, A. (2007). Agricultural residues as precursors for activated carbon production—a review. *Renewable and sustainable energy reviews*, 11(9), 1966-2005.
- Daud, W. M. A. W., Ali, W. S. W., & Sulaiman, M. Z. (2000). The effects of carbonization temperature on pore development in palm-shell-based activated carbon. *Carbon*, 38(14), 1925-1932.
- Suárez-García, F., Martínez-Alonso, A., & Tascón, J. D. (2002). Pyrolysis of apple pulp: chemical activation with phosphoric acid. *Journal of Analytical and Applied Pyrolysis*, 63(2), 283-301.
- Hayashi, J. I., Kazehaya, A., Muroyama, K., & Watkinson, A. P. (2000). Preparation of activated carbon from lignin by chemical activation. *Carbon*, 38(13), 1873-1878.
- Diao, Y., Walawender, W. P., & Fan, L. T. (2002). Activated carbons prepared from phosphoric acid activation of grain sorghum. *Bioresource technology*, 81(1), 45-52.
- Lua, A. C., Yang, T., & Guo, J. (2004). Effects of pyrolysis conditions on the properties of activated carbons prepared from pistachio-nut shells. *Journal of analytical and applied pyrolysis*, 72(2), 279-287.
- Senthilkumar, S., Varadarajan, P. R., Porkodi, K., & Subbhuraam, C. V. (2005). Adsorption of methylene blue onto jute fiber carbon: kinetics and equilibrium studies. *Journal of colloid and interface science*, 284(1), 78-82.
- Martínez-Martínez, J., Norland, S., Thingstad, T. F., Schroeder, D. C., Bratbak, G., Wilson, W. H., & Larsen, A. (2006). Variability in microbial population dynamics between similarly perturbed mesocosms. *Journal of plankton research*, 28(8), 783-791.

- Olivares-Marín, M., Fernandez-Gonzalez, C., Macías-García, A., Gomez-Serrano, V., Lillo-Rodenas, M. A., Carzola-Ameros, D., & Linares-Solano, A. (2003). Chemical reactions between carbons and NaOH and KOH-an insight into chemical activation mechanisms. *Carbon*, *41*, 265-267.
- Ash, B., Satapathy, D., Mukherjee, P. S., Nanda, B., Gumaste, J. L., & Mishra, B. K. (2006). Characterization and application of activated carbon prepared from waste coir pith. *J Sci Ind Res*, *65*, 1008-1012.
- Gürses, A., Dođar, Ç., Karaca, S., Acikyildiz, M., & Bayrak, R. (2006). Production of granular activated carbon from waste Rosa canina sp. seeds and its adsorption characteristics for dye. *Journal of Hazardous Materials*, *131*(1-3), 254-259.
- Suzuki, R. M., Andrade, A. D., Sousa, J. C., & Rollemberg, M. C. (2007). Preparation and characterization of activated carbon from rice bran. *Bioresource technology*, *98*(10), 1985-1991.
- Gerçel, Ö., Özcan, A., Özcan, A. S., & Gerçel, H. F. (2007). Preparation of activated carbon from a renewable bio-plant of Euphorbia rigida by H₂SO₄ activation and its adsorption behavior in aqueous solutions. *Applied surface science*, *253*(11), 4843-4852.
- Tan, I. A. W., Hameed, B. H., & Ahmad, A. L. (2007). Equilibrium and kinetic studies on basic dye adsorption by oil palm fibre activated carbon. *Chemical engineering journal*, *127*(1-3), 111-119.
- Hameed, B. H., & Daud, F. B. M. (2008). Adsorption studies of basic dye on activated carbon derived from agricultural waste: Hevea brasiliensis seed coat. *Chemical Engineering Journal*, *139*(1), 48-55.
- H. Deng, L. Yangb, G. TAO, J. Daia, Preparation and characterization of activated carbon from cotton stalk by microwave assisted chemical activation application in methylene blue adsorption from aqueous solution, *J. Hazard.Mater.* *166* (2009) 1514–1521.
- Diao, Y., Walawender, W. P., & Fan, L. T. (2002). Activated carbons prepared from phosphoric acid activation of grain sorghum. *Bioresource technology*, *81*(1), 45-52.
- Kim, D. Y., Nishiyama, Y., Wada, M., & Kuga, S. (2001). High-yield carbonization of cellulose by sulfuric acid impregnation. *Cellulose*, *8*(1), 29-33.
- Mopoung, S., Moonsri, P., Palas, W., & Khumpai, S. (2015). Characterization and properties of activated carbon prepared from tamarind seeds by KOH activation for Fe (III) adsorption from aqueous solution. *The scientific world journal*, 2015.
- Gundogdu, A., Duran, C., Senturk, H. B., Soylok, M., Imamoglu, M., & Onal, Y. (2013). Physicochemical characteristics of a novel activated carbon produced from tea industry waste. *Journal of Analytical and Applied Pyrolysis*, *104*, 249-259.
- Hameed, B. H., & Daud, F. B. M. (2008). Adsorption studies of basic dye on activated carbon derived from agricultural waste: Hevea brasiliensis seed coat. *Chemical Engineering Journal*, *139*(1), 48-55.
- Angin, D. (2014). Production and characterization of activated carbon from sour cherry stones by zinc chloride. *Fuel*, *115*, 804-811.
- Mastalerz, M., & Bustin, R. M. (1995). Application of reflectance micro-Fourier transform infrared spectrometry in studying coal macerals: comparison with other Fourier transform infrared techniques. *Fuel*, *74*(4), 536-542.