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PHYSICAL PROPERTIES ANALYSIS OF RUBBER SEED SHELL ACTIVATED CARBON AS ALTERNATIVE MEDIA FOR BIO-AIR AND WATER FILTER

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ABSTRACT
Rubber seed shells as bio-waste materials have many potential for bio-filtration media. However, it needs some processes for developing the shell into Activated Carbon (AC) as filter media. One of the most promising methods is the indirect carbonization process by pyrolysis reactor to achieve high physical properties. Therefore, the aim of this research is to develop Rubber Seed Shell Activated Carbon (RSSAC) and analyze its physical properties. Charcoal production was performed by pyrolysis reactor with an indirect carbonization process at 450 °C with a heating rate of 15 °C/min and a holding time of 2 hours. Activation is conducted by a KOH agent with a ratio of 1:1 and mixed with distilled water followed by 12-hour soaking time. The analysis to be carried out consists of physical properties, density, porosity, and microstructure analysis performed by Scanning Electron Microscope (SEM). The results showed that the hardness of the charcoal decreased by 19.48HRC from 63.08HRC, and the moisture and ash content of the charcoal reached 76.46%, and 30.78%, respectively. The lowest product yield of 28.92 MJ/kg is much higher compared with palm shell at 18.4 MJ/kg. The porosity of RSSAC increased to 11.43% from 2.54% and it shows the honeycomb form in microstructure analysis. Therefore, it has a high potential for pollutant absorption to improve the air and water quality.
Keywords: Rubber Seed Shell, Porosity, Activated Carbon, Pyrolysis, Air And Water Filter.
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INTRODUCTION

Indonesia is one of the largest natural rubber producers in the world with an area of 1.3 million hectares of rubber plantations.[1,2] The annual production of rubber seed varies from 300 - 2060 kg/hectare[3] and this is a particular difficulty in the process of collecting and processing it because it does not have high selling value.[4] Rubber seeds have a total weight of 3-6g (fresh weight) consisting of 42-51% shell and 49-58% kernel. Based on dry weight, rubber kernels contain 40-50% oil and 19-23% protein. Therefore, the process of oil production and protein extraction has the potential to be carried out and developed as an alternative fuel better known as biofuel, as well as for the shell potential for producing bio-filter media.[5]
Bio-filtration media become a need to create fresh and healthy air and water in order to improve human health and working performance.[6] Several pollutants contain air pollution such as Carbon Monoxide (CO), Particulate Matter ($PM_{2.5}$ and PM_{10}), Carbon Dioxide (CO_2), and Volatile Organic Compound (VOC). Meanwhile, water contaminants contain several heavy metals that need to be filtered. Pollution may affect human health related to the respiratory system (asthma, lung cancer, and throat irritation), eyes, nose, and skin irritation.[1-3] One of the common methods to improve the air and water quality is Activated Carbon (AC) due to its abundant waste bio-material, cheap and simple process compared to other technologies.[7,8] Abundant waste material is the biggest factor that leads to continuous AC production. There are many advantages of AC such as high carbon content, high density, low ash content, dissolving organic ability, high absorption capacity, and chlorine removal capability, which can be used as a specific application.[9] This agricultural waste such as rubber seed can also be suitable as raw material for producing high-value products and fuels through biochemical and thermo-chemical conversion methods.[9] The thermo-chemical method include processes such as combustion, gasification, carbonization, pyrolysis, etc.[10] The pyrolysis process offers certain advantages such as operating at low temperatures, higher yield and liquid product quality, better control and safe storage, etc.[11] In the

pyrolysis process, solids (biomass) are converted into liquid, gas, and charcoal products at a temperature range of 300-700 ^{0}C in the absence of oxygen.[12] About 25-70% of the liquid product yield can be obtained from the oil-containing seeds.[13] Based on previous research by Reshad *et al.,*[13] rubber seed shells as biomass raw material can offer more benefits and potential to reduce current environmental challenges.[14] The shell contains very little oil compared to the kernel. Ghani *et al.,* and Shaaban *et al.,*[15,16] have utilized oil from rubber seeds and husks through pyrolysis for product synthesis. Sun and Jiang, Leman *et al.,* and Feriyanto *et al.,*[17-19] studied the synthesis of activated carbon from rubber seed shells by physical and chemical activation. Studies show that rubber seed shells are a good precursor source of raw materials for the production of high-quality activated carbon. In addition, pyrolysis of pomegranate seeds was carried out by Ucar and Karagoz[20] resulting in the liquid yield (~ 54% of the maximum weight) obtained at two pyrolysis temperatures of 500 and 600 ^{0}C. AC production involves three major processes such as carbonization, crushing, and activation process. The carbonization called pyrolysis was operated at temperatures from 300-900 °C.[21] This process aimed to eliminate the non-carbon elements (Hydrogen (H_2), Oxygen (O_2), Nitrogen (N), and Sulphur (S)). There are many previous researchers that used the chemicals agent for the activation process such as H_3PO_4, $ZnCl_2$, NaOH, KOH, HNO_3, K_2CO_3, H_2SO_4, $NaHCO_3$, and NaCl.[21,22] Activation is divided into two activation processes which are physical and chemical activation.[23,25] The material that will be activated should be in granular form to create a very high surface area and porosity adequate for specific applications such as gasoline vapor control and gas storage.[26,27] These ACs use lignocellulosic precursors which are chemically activated with phosphoric acid and zinc chloride and activated under a flow of CO_2. Uniform, medium-size microporosity and surface areas above 3600 m^2/g are obtained with this mixed procedure.[28] Furthermore, significant weight loss is present during the CO_2 activation process and it is justified owing to the continuous release of volatile matter and carbon burn-off.[28-30] This AC production produces a yield of 19.25% and bulk density of 0.62 g/cm^3, achieving the highest adsorption capacity with a surface area of 217 gm^{-2}, meso-pore volume of 1 $cm^{-3}g^{-1} \times 10^{-2}$ and micro-pore volume (Vμ) is 11 $cm^{-3}\ g^{-1} \times 10^{-2}$.[31,32] Significant weight loss becomes the main concern to minimize due to it will reduce AC production.[33,34] Therefore, this research proposed to synthesize the activated carbon from a rubber seed shell with a simple indirect carbonization process and base activation.

EXPERIMENTAL

A lab-scale pyrolysis reactor, capable of generating temperatures up to 700 °C, was developed based on previous studies, as shown in Fig.-1. The reactor comprises a reactor tube, a heater, a temperature sensor, a condenser with a circular pipe, and water as a cooling medium. The experiment began with the collection of rubber seed kernels (Fig.-2a), which were soaked in distilled water for an hour, followed by pyrolysis at 600 °C, with a heating rate of 15 °C/min and a holding time of 2 hours. Additionally, the reactor was used to produce rubber seed shell charcoal at 450 °C for 2 hours. The system employed an indirect carbonization process to maximize the fixed carbon content in the samples and minimize the ash content. The reactor has a capacity of 2 kg for each carbonization process.

Fig.-1: Lab-Scale Pyrolysis Reactor

After carbonization, the rubber seed shells were crushed to create a granular shape (Fig.-2 (b)). The granular activated carbon (GAC) variety was chosen to serve as an effective filter for both air and water.

For air and water filtration, the size of granules is < 500 and > 500 µm, respectively. The process of activating charcoal involves both physical and chemical methods. First, the charcoal is ground and filtered using a size of 25 µm – 1 mm. The best sample obtained from the optimization process, which includes parameters such as fixed carbon content, porosity, and product yield, is then subjected to the activation process. Physical activation is achieved by reheating the charcoal using a tube burner at 900 °C for 1 h. Chemical activation involves using potassium hydroxide (KOH) with a charcoal-to-solution ratio of 1:1. The mixture is heated at 60 °C and stirred, followed by a 12-hour activation period and rinsing with water until the pH reaches 7. The chemically activated charcoal is then heated at 120 °C for 1 h to remove any remaining water content.

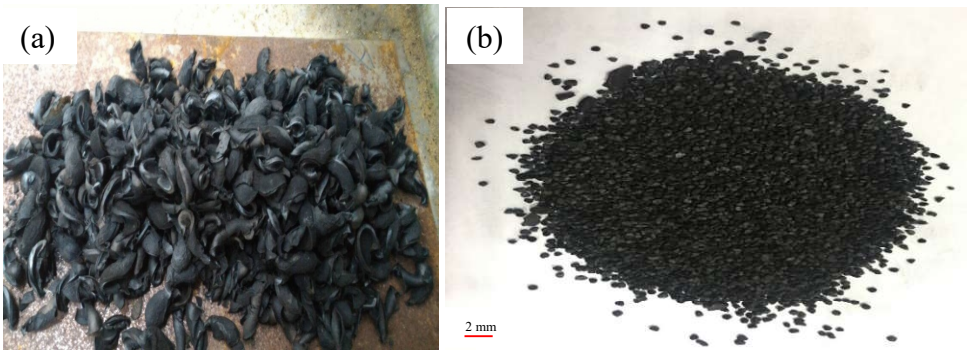

Fig.-2: Image of (a) Rubber Seed Shell and (b) Granular Rubber Seed Shell

This research involved examining various physical properties, including moisture content, ash content, volatile matter, fixed carbon, and product yield. Additionally, the ASTM D2240 standard (ISO standard, 1990) was used to perform hardness testing. The test was conducted using a hardness tester, with a minimum thickness requirement of 6.4 mm for the test object. The porosity and density tests were carried out by following the ASTM B328-96 standard and Archimedes' law. The tests were conducted at 26.6 °C, which was the room temperature, in a closed room to prevent any interference from the outside environment. Rubber seed was used for the test to determine the degree of porosity after the carbonization process that resulted in the formation of charcoal. Microstructure and chemical composition were examined by scanning electron microscopy (SEM, Hitachi, Japan) at various magnifications to observe pores.

RESULTS AND DISCUSSION

Upon analyzing rubber seed that underwent pyrolysis and carbonization, it was found that its average moisture content was 68.24 ± 2.53% and ash content was 21.74 ± 3.42%. It's worth noting that the high standard error is attributed to a significant variance in moisture content, which ranged from 30.78% to 76.46%. The high moisture content is likely proportional to the number of pores present in the material. Moreover, the ash content significantly influences the carbon amount in the material and its potential for combustion. Materials with low porosity tend to yield poor combustion results.

Table-1: Physical Properties of Rubber Seed Before and After

	Before	After
Density (g cm^{-3})	0.81 ± 0.05	0.42 ± 0.07
Porosity (%)	2.54 ± 0.25	11.43 ± 1.41
Hardness (HRC)	63.08 ± 0.27	43.60 ± 1.21

The physical characteristics of rubber seed before and after carbonization are shown in Table-1. The density of the rubber seed significantly decreased from 0.81 ± 0.05 to 0.42 ± 0.07 g cm^{-3} after carbonization, which was expected due to the evaporating of the volatile materials and carbonization process. On the other hand, the porosity of the rubber seed increased significantly from 2.54 ± 0.25 to 11.43 ± 1.41 % after carbonization. This high porosity indicates a high moisture content, as mentioned earlier. The mechanical properties of the samples are greatly influenced by their density and number of pores. A denser structure typically results in higher mechanical properties compared to a porous one. Consequently, the hardness of the samples decreased as the number of pores increased from 63.08 ± 0.27

to 43.60 ± 1.21 HRC before and after carbonization, respectively, as outlined in Table-1. These findings suggest that carbonization has a significant impact on the physical and mechanical properties of rubber seeds, which should be considered for various industrial applications.

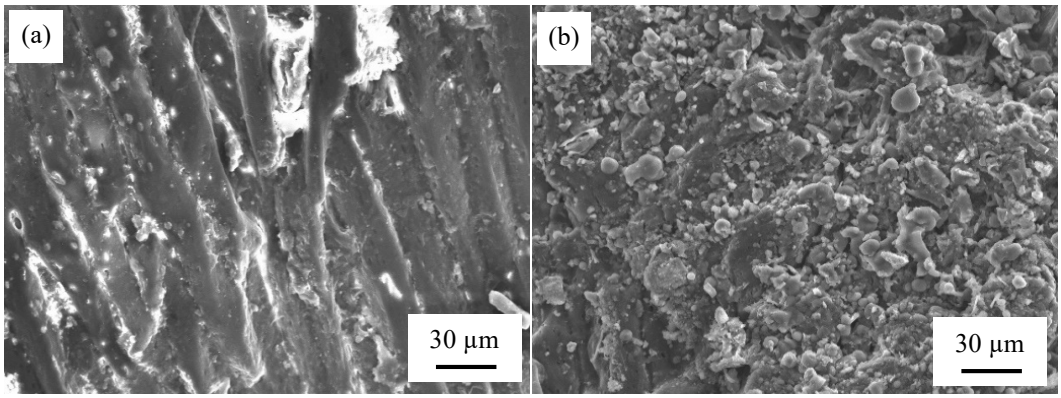

Fig.-3: SEM Images of Rubber Seed (a) Before Carbonization and (b) After Carbonization at 450°C for 1h

Fig.-3 displays the SEM images of rubber seeds before and after carbonization. Prior to carbonization, the microstructure of rubber seed was found to be quite dense, as seen in Fig.-3(a), confirming its low porosity and high density as indicated in Table-1. However, a significant change occurred after carbonization, as shown in Fig.-3(b), where the rubber seed underwent pulverization and resulted in a loose structure with small grain. This resulting loose structure and small grain have many pores and low density, as shown in Table-1. The pulverization through heating has been previously reported by Noviyanto *et al.*,[35] in their study on $La_2Ti_2O_7$ crushed by heating at 1000 °C for 20 hours in an ammonia-containing atmosphere. The carbonization process was performed to achieve high porosity, which significantly contributes to the absorption capacity of pollutants from both air and water due to its high specific surface area. It is essential for charcoal to possess specific physical properties, including volatile matter and fixed carbon. To examine these properties, we analyzed five samples of carbonized rubber seed. The results of our thorough investigation are displayed in Fig.-4, which demonstrates an inverse relationship between volatile matter and fixed carbon. For instance, sample 1 displayed the highest fixed carbon of 81.36% and the lowest volatile matter of 16.73%. In practical terms, a high fixed carbon percentage is desirable as it corresponds to a higher caloric value. Therefore, it is imperative to decrease the moisture content, which leads to volatile matter. The average caloric value of rubber seed was 30.87 ± 1.01 MJ/kg, with the highest and lowest values being 34.50 and 28.92 MJ/kg, respectively. For comparison, the caloric value of charcoal from palm shell was 18.4 MJ/kg, which is 10 points lower than the caloric value of rubber seed in this study.

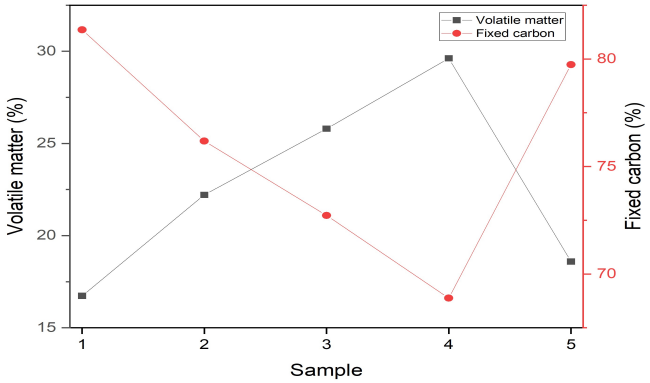

Fig.-4: Volatile Matter and Fixed Carbon of Rubber Seed After Carbonization

Moisture and ash content were analyzed after the carbonization process and it was examined from five samples as shown in Fig.-5. It is in line with fixed carbon and volatile matter that samples four has the highest moisture content of 76.65% and the lowest ash content of 10.28%. High moisture content leads to

porosity development for the absorption area. Meanwhile, the ash content affects the amount of carbon as a result of the carbonization process.

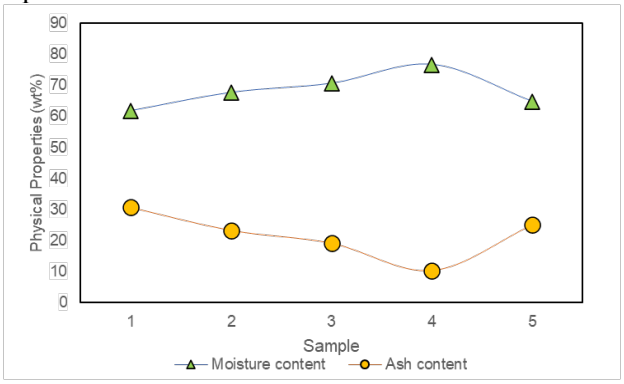

Fig.-5: Moisture and Ash Content of Rubber Sheet After Carbonization Process

The activation process was carried out using KOH with a soaking time of 12 hours. Figure-6 shows SEM images of the activated rubber seed. The microstructure experiences drastic change compared to carbonized rubber seed (Fig.-3 (b)), showing a hollow structure from a honeycomb-like structure, as shown in Fig.-6 (a). Hence, it implies the activation process using KOH for 12 hours succeeds. High magnification of activated rubber seed is shown in Fig.-6 (b), revealing the formation of small pores inside the hollow structure. In terms of absorption, the honeycomb structure, as shown in Fig.-5, generally has a high capacity to absorb pollutants, both in air and water. Thus, the activated rubber seed in this study is an alternative to the existing absorbent materials, as media to absorb pollutants in air or water.

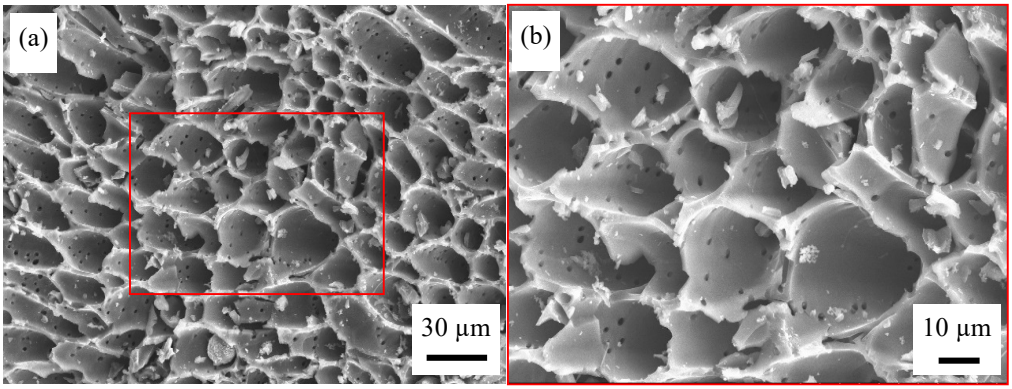

Fig.-6: SEM Images of (a) Activated Rubber Seed and its Magnification in Red Line are Shown in (b)

CONCLUSION

Charcoal was successfully synthesized using a rubber seed shell prepared by indirect carbonization in a pyrolysis reactor for 2 hours followed by base activation using KOH. The properties of carbonized rubber seed, such as density, hardness, and porosity, have decreased compared to raw material, indicating that carbonization has succeeded. The average caloric value of carbonized rubber seed was 30.87 ± 1.01 MJ/kg, which is almost two times the caloric value of a palm shell. Furthermore, the KOH activation resulted in a unique honeycomb-like hollow structure, making it a promising absorbent material with a high capacity to absorb pollutants in air or water. As a result, the activated rubber seed shells are an alternative to other absorbent materials, with potential applications as a medium for pollutant absorption. Further research will be needed to confirm the absorption properties of this activated rubber seed.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

All the authors contributed significantly to this manuscript, participated in reviewing/editing, and approved the final draft for publication. The research profile of the authors can be verified from their ORCID IDs, given below:

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REFERENCES

1. https://databoks.katadata.co.id/datapublish/2019/10/30/inilah-10-provinsi-penghasil-karet-di-indonesia
2. M. A. Suprayudi, C. Inara, J. Ekasari, N. Priyoutomo, Y. Haga, T. Takeuchi, S. Satoh, *Aquaculture. Research,* **46(12)**, 2972(2014), https://doi.org/10.1111/are.12452
3. Y. Zhu, J. Xu, Q. Li, P. E. Mortimer, *Energy*, **69**, 837(2014), https://doi.org/10.1016/j.energy.2014.03.079
4. T. T. X. Nguyen, V. A. T. Huynh, T. B. Y. Le, *Journal of Science and Technology*, **6(115)**, 45(2017), https://doi.org/10.1039/C6CY00358C
5. T. E. Odetoye, K. R. Onifade, M. S. Abu Bakar, J. O. Titiloye, *Industrial Crops and Products*, **44**, 62(2013), https://doi.org/10.1016/j.indcrop.2012.10.013
6. H. T. Mudassir, M. I. Rana, B. H. Muhammad, A. Hesham, T. Yusuf, *ACS Omega*, **6(49)**, 33694(2021), https://doi.org/10.1021/acsomega.1c04717
7. I. B. Bankovic-Ilic, S. Ostamenkovic, V. B. Veljkovic, *Renewable and Sustainable Energy Reviews*, **16**, 3621(2012), https://doi.org/10.1016/j.rser.2012.03.002
8. J. Gimbun, S. Ali, C. C. S. C. Kanwal, L. A. Shah, N. H. M. Ghazali, C. K. Cheng, *MUCET 2012 Part 3-Civil and Chemical Engineering*, 13-19(2013), https://doi.org/10.1016/j.proeng.2013.02.003
9. T. H. Mohammad, R. Lakhmiri, A. Azmani, I. I. Hassan, *International Journal of Basic and Applied Sciences,* **14**, 1(2014), ISSN: 2077-1223 (Online) 2227-2720 (Print)
10. B. L. F. Chin, S. Yusup, A. Al Shoaibi, P. Kannan, C. Srinivasakannan, S. A. Sulaiman, *Energy Conversion and Management*, **87**, 746(2014), https://doi.org/10.1016/j.enconman.2014.07.043
11. Z. Chen, M. Hu, X. Zhu, D. Guo, S. Liu, Z. Hu, *Bioresource Technology,* **192**, 441(2015), https://doi.org/10.1016/j.biortech.2015.05.062
12. C. Wood, K. A. Rosentrater, K. Muthukumarappan, *Industrial Crops and Products,* **56**, 118(2014), https://doi.org/10.1016/j.indcrop.2014.02.039
13. A. S. Reshad, P. Barman, A. J. Chaudhari, P. Tiwari, V. Kulkarni, V. V. Goud, *Energy Fuels,* **29**, 5136(2015), https://doi.org/10.1021/acs.energyfuels.5b01033
14. A. S. Reshad, P. Tiwari, V. V. Goud, *Fuel*, **150**, 636(2015), https://doi.org/10.1016/j.fuel.2015.02.058
15. A. A. Ghani, A. Mohd, G. da Silva, R. T. Bachmann, Y. H. Taufiq-Yap, U. Rashid, *Industrial Crops and Products,* **44**, 8(2013), https://doi.org/10.1016/j.indcrop.2012.10.017
16. A. Shaaban, S. Se, M. F. Dimin, J. M. Juoi, M. M. Husin, N. M. M. Mitan, *Journal of Analytical and Applied Pyrolysis,* **107**, 31(2014), https://doi.org/10.1016/j.jaap.2014.01.021
17. K. Sun, J. C. Jiang, *Biomass and Bioenergy*, **34**, 539(2010), https://doi.org/10.1016/j.biombioe.2009.12.020
18. A. M. Leman, S. Zakaria, M. N. M. Salleh, N. M. Sunar, D. Feriyanto, A. A. Nazri, *AIP Conference Proceedings*, **1885(1)**, 020001(2017), https://doi.org/10.1063/1.5002195
19. D. Feriyanto, S. Zakaria, S. Alva, H. Pranoto, A. F. Sudarma, A. O. L. Siang, *International Journal of*

Advanced Technology in Mechanical, Mechatronics and Materials, **1(2)**, 39(2020)
20. S. Ucar, S. Karagoz, *Journal of Analytical and Applied Pyrolysis,* **84**, 151(2009), https://doi.org/10.1016/j.jaap.2009.01.005
21. M. Morshed, K. Ferdous, M. R. Khan, M. S. I. Mazumder, M. A. Islam, M. T. Uddin, *Fuel,* **90**, 2981(2011), https://doi.org/10.1016/j.fuel.2011.05.020
22. D. Balkose, T. Egbuchunam, F. Okieimen, *Journal of Thermal Analysis and Calorimetry*, **101**, 795(2010), https://doi.org/10.1007/s10973-010-0940-4
23. Y. Hu, G. Zhu, J. Zhang, J. Huang, X. Yu, Q. Shang, R. An, C. Liu, L. Hu, Y. Zhou, *Molecules*, **26(18)**, 5455(2021), https://doi.org/10.3390/molecules26185455
24. R. O. Ebewele, A. F. Iyayi, F. K. Hymore, *International Journal of The Physical Sciences*, **5**, 826(2010), ISSN 1992 - 1950
25. S. H. Mohd-Setapar, L. N. Yian, M. A. C. Yunus, I. I. Muhamad, M. A. A. Zaini, *Journal of Biobased Materials and Bioenergy*, **7**, 213(2013), https://doi.org/10.1166/jbmb.2013.1314
26. E. Baumler, G. Crapiste, A. Carelli, *Journal of The American Oil Chemists' Society,* **87**, 1489(2010), https://doi.org/10.1007/s11746-010-1637-3
27. K. G. Michael, O. A. Sogbesan, L. U. Onyia, S. M. Shallangwa, *Direct Research Journal of Agriculture and Food Science*, **7(6)**, 131(2019), https://doi.org/10.5281/zenodo.3242756
28. Y. W. Sari, M. E. Bruins, J. P. M. Sanders, *Industrial Crops and Products,* **43**, 78(2013), https://doi.org/10.1016/j.indcrop.2012.07.014
29. S. Latif, F. Anwar, *Food Chemistry,* **125**, 679(2011), https://doi.org/10.1016/j.foodchem.2010.09.064
30. A. O. L. Siang, A. M. Leman, D. Feriyanto, S. Zakaria, *Regional Journal in Mechanical Engineering and Advanced Materials (RJMEAM)*, (2016), https://doi.org/10.1038/s41598-019-55403-4
31. E. Lazzari, T. Schena, C. T. Primaz, G. P. S. Maciel, M. E. Machado, C. A. L. Cardoso, *Industrial Crops and Products,* **83**, 529(2016), https://doi.org/10.1016/j.indcrop.2015.12.073
32. Z. Ma, D. Chen, J. Gu, B. Bao, Z. Qisheng, *Energy Conversion and Management,* **89**, 251(2015), https://doi.org/10.1016/j.enconman.2014.09.074
33. A. S. Reshad, P. Barman, A. J. Chaudhari, P. Tiwari, V. Kulkarni, V. V. Goud, *Energy Fuels,* **29**, 5136(2015), https://doi.org/10.1021/acs.energyfuels.5b01033
34. Z. Chen, M. Hu, X. Zhu, D. Guo, S. Liu, Z. Hu, *Bioresource Technology,* **192**, 441(2015), https://doi.org/10.1016/j.biortech.2015.05.062
35. A. Noviyanto, T. Nishimura, M. Kitano, N. Ohashi, *Journal of the European Ceramic Society,* **36(16)**, 4083(2016), https://doi.org/10.1016/j.jeurceramsoc.2016.06.031

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