# Utilization of Coconut Waste as a Basic Material for Making Carbon Dots with Acid Oxidation Method

Marpongahtun[1,2] , Irmayani[1] and Suci A.[1]
[1]*Chemistry Department, Universitas Sumatera Utara, Jalan Bioteknologi No. 1, Medan, Indonesia*
[2]*Laboratorium Penelitian Terpadu Universitas Sumatera Utara, Medan, Indonesia*

Keywords: Carbon Dots, Fluorescence, Soot, Acid Oxidation.

Abstract: Utilization of coconut shell soot as raw material for making carbon dots has been successfully carried out. This study used coconut shell soot with 5 M $HNO_3$ acid oxidation method which is then the Carbon dots characteristics were determined using UV light, UV-Vis, TEM analysis, and functional group analysis with FT-IR. Coconut shell soot is obtained by pyrolysis method. The heating process using a furnace carried out for 2 hours at 400 °C. Carbon dots produced by heating with 5 M $HNO_3$ for 12 hours at 100 °C, centrifuged and dialysis. The yield of carbon dots was 87%. The FT-IR spectrum shows that the Carbon dots formed produce OH group absorption at 3396 $cm^{-1}$, absorption of C = C at wave number 1637 $cm^{-1}$, absorption of CO groups, and CH at wave number 1339 $cm^{-1}$, and 835 $cm^{-1}$. Analysis with Transmission Electron Microscopy shows that carbon dots has an average diameter of 1.50 nm. Absorbance spectrum analysis (UV-Vis) results in the appearance of new uptake showing the electron transition at a wavelength of 307 nm and giving green fluorescence under UV light.

## 1 INTRODUCTION

Fluorescence nanoparticle material is receiving a lot of attention due to its superiority and wide application. One of the interesting properties of F-NPs is the ability to fluency, so it is widely applied as bioimaging. However, this material involves the use of heavy metals such as Cd and Pb which are known to have high toxicity so that their use are limited. Based on this problem, another alternative is used to replace the raw material from F-NPs, by using carbon-based nanoparticles or known as Carbon dots. Carbon dots is a material that has a zero dimensional structure and is a product of carbon nanotubes. Besides has properties that are almost similar to F-NPs, the ability of fluorescence, Carbon dots also have high solubility in water, environmentally friendly, have low toxicity. and low manufacturing costs (Baker & Baker, 2010). Carbon sources that can be used as starting material for C-dots include soot from burning candles (Liu et al., 2007), nanocrystal cellulose (Marpongahtun et al., 2018), burning plants (Tan et al., 2013) and coal (Ye et al., 2013). Carbon dots can also be synthesized from organic materials such as citric acid (Qu et al., 2012) and ascorbic acid (Nisa, 2014).

Coconut shell is one of the materials that can be used as charcoal, the residue produced when burning charcoal is smoke. The process of the smoke taking place in the combustion chamber is caused by the fuel droplets collected into soot because the heating is too large so that decomposition occurs. Decomposition will cause carbon solid (soot) (Arismunndar, 2002). Soot can be made by direct or indirect heating in the pile or by the pyrolysis method (Oladeji, 2010).

Various methods for the synthesis of Carbon dots have been developed by scientists. In general, the methods for carbon nanoparticle synthesis are based on 2 approaches, the top-down and bottom-up approaches. Bottom-up approach includes electrothermal synthesis, microgear or ultrasonic, hydrothermal, and acid oxidation (Li et al., 2012). Carbon dots synthesis in this study uses a bottom-up approach, through the $HNO_3$ acid oxidation method. In addition to acid treatment by $HNO_3$, the carbon dots synthesis is also carried out by the purification process by centrifugation, dialysis, and other separation techniques (Li et al., 2012). All of these processes are to obtain Carbon dots from a carbon soot combustion. Based on the description above, the author will conduct research on "Synthesis of



Marpongahtun, ., Irmayani, . and A., S.
Utilization of Coconut Waste as a Basic Material for Making Carbon Dots with Acid Oxidation Method.
DOI: 10.5220/0010139700002775
In *Proceedings of the 1st International MIPAnet Conference on Science and Mathematics (IMC-SciMath 2019)*, pages 232-236
ISBN: 978-989-758-556-2

Carbon dots from coconut shell soot by acid oxidation methods".

Dry carbon dots are characterized by UV lamps, UV-Vis spectros, TEM, FE-SEM, and FT-IR.

# 2 RESEARCH METHODS

## 2.1 Tools

The tools used in this study are glassware, FT-IR, FE-SEM, TEM, UV lamps, UV-Visible spectra, pyrolysis, and centrifugation.

## 2.2 Material

The materials used in this study are NaOH, $HNO_3$ (p), distilled water, Coconut Shell, and Dialysis Membrane.

## 2.3 Research Procedure

### 2.3.1 Preparation of Coconut Shell

Coconut shell waste is cleaned from the remaining coconut husks that still attached, cut into small pieces and dried in the sun until the weight remains.

### 2.3.2 Making Soot from Coconut Shell

The dried coconut shell waste is put into a porcelain cup, then heated using a furnace at 400 °C for 2 hours. The resulting soot was then analyzed by FE-SEM.

### 2.3.2 Synthesis of Carbon Dots

Soot of carbon that has been obtained weighed as much as 500 mg, put in a three neck flask. The reflux assembly is arranged and a magnetic stir bar is inserted. Then 100 mL of 5N $HNO_3$ was added and refluxed at 100 °C for 12 hours. The result of reflux is cooled at room temperature, after that it is messed up using centrifugation at a speed of 4500 rpm for 30 minutes, then from the process will be obtained 2 phases, the brown supernatant phase and the solid phase in the form of black deposits, then the two phases are separated and neutralized with NaOH 5M (Li et al., 2012).

The neutralization is carried out until pH = 7, after that the neutralization results are filtered using filter paper and then dialyzed using a dialysis membrane for 24 hours by continuing to replace the aquadest water for 30 minutes 5 times at the beginning of dialysis.

# 3 RESULTS AND DISCUSSION

## 3.1 Results of Sample Preparation for Coconut Shell Soot

The soot preparation is carried out using a furnace tool. This preparation was carried out at 400 °C for 2 hours. The results of soot preparation can be seen in Figure 1.

Figure 1: Results of coconut shell soot.

From 30 grams of coconut shell used will obtained 5 grams of black carbon, 16.6% of the initial weight of the coconut shell. Obtained Carbon soot then analyzed using FT-IR to determine the functional groups contained in coconut shell soot and morphological analysis using FE-SEM.

## 3.2 Surface Analysis of Soot with FE-SEM

Surface analysis of coconut shell soot using FE-SEM can be seen in Figure 2.

The results shown in Figure 2 are the results of coconut shell soot with a heating of 400 °C for 2 hours where the resulting soot has an average surface diameter of 138.04 µm and appears to be random piles.

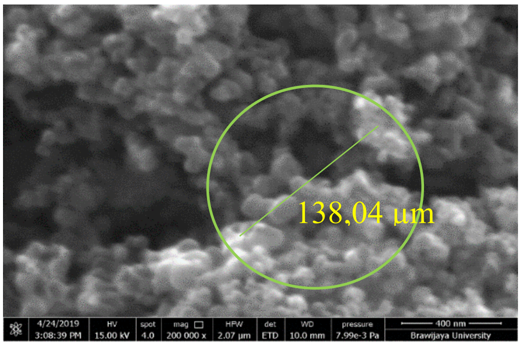


Figure 2: Results of morphological analysis of coconut shell soot with enlargement 400 times.

## 3.3 Soot Carbon dots from Coconut Shell with Acid Oxidation Method

Carbon dots solution obtained from the coconut shell in the previous stage placed into a beaker glass then evaporated to dry in an oven at 90 °C for 12 hours. The C-dot results obtained can be seen in Figure 3.

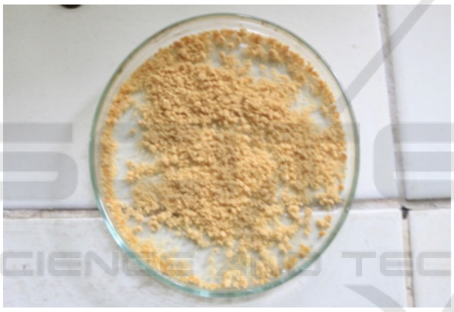

Figure 3: Carbon dots from coconut shell soot.

From 0.5 grams of coconut shell soot 0.48 grams of Carbon dots was obtained equal to 87% of the initial weight of soot. Carbon dots from coconut shell soot produce a yellowish brown color. The oxidation reaction scheme can be seen in Figure 4.

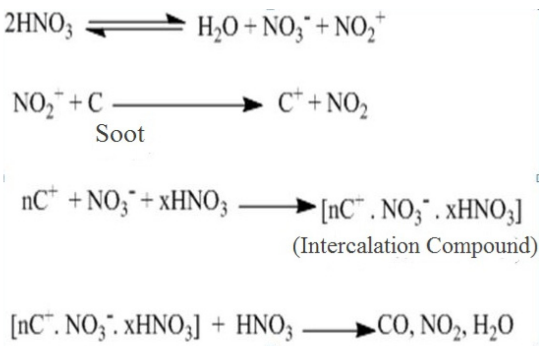


Figure 4: Oxidation reaction scheme.

The addition of $HNO_3$ aims to disperse by oxidizing molecules that have undergone agglomeration. The reaction between carbon from soot and $NO_3^-$ ions causing electrons displacement by the formation of complex compounds produced Carbon dots are different from the base material. Further interaction of complex compounds with $HNO_3$ forms intercalation compounds (Savitskii, 2017).

## 3.4 Characterization

### 3.4.1 Analysis with UV Lamps

Fluorescence testing is carried out physically through observation under a UV lamp. Carbon dots produced through the acid oxidation method produce flourescence under UV light as shown in Figure 5.

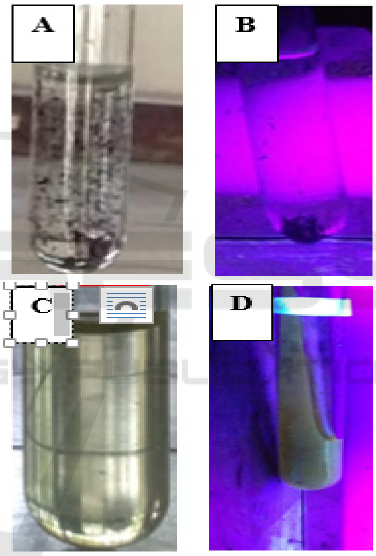


Figure 5: Carbon dots irridiation with UV lamps results: (a) Negative control of coconut shell soot in visible light; (b) Negative control of coconut shell soot under UV light; (c) Carbon dots in visible light; (d) Carbon dots under UV light.

The success of Carbon dots synthesis can be seen from the fluorescence analysis using UV lamps. Irradiation is carried out on a Carbon dots solution with a concentration of 1000 ppm which can be seen in Figure 5. These results show a green glow and in accordance with the results reported by Ray et al. (2009). Fluorescence occurs when electrons move from the valence band to thes conduction band after being excited by the excitation source in this case UV light. Besides having good fluorescence intensity, Carbon dots also have high solubility in water (Liu et al., 2007). Figure 5 also shows that

Carbon dots from coconut shell soot have higher solubility compared to negative control. The negative control made from a soot solution of pure coconut shell before oxidation, dissolved in water at concentration of 1000 ppm. The high solubility point of Carbon dots is due to the successful functioning of the Carbon dots surface. The functionalization process will bring up functional groups such as hydroxyl and carboxyl which will cause the surface of Carbon dots to be negatively charged, so that Carbon dots will be hydrophilic (Liu et al., 2007).

### 3.4.2 Absorbance Spectrum Analysis with UV-Vis Spectrophotometry

The synthesis results of Carbon dots obtained were then characterized using UV-Vis spectrophotometry. Measurements were made at a wavelength of 200-700 nm with wavelength interval of 100 nm.

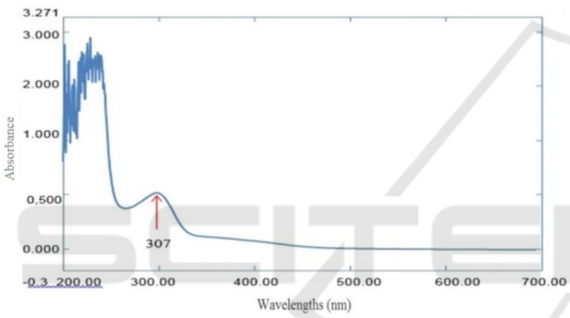


Figure 6: Absorbance curve with wavelengths from coconut shell soot carbon dots.

Spectrum of the negative control Carbon dots of coconut shell soot does not produce new absorption at certain wavelengths (Figure 6), different from the results of the Carbon dots spectrum of coconut shell soot that produce new absorption at wavelengths of 307 nm with absorbance of 0.5040.

### 3.4.3 Analysis of C-dot Average Diameter by using TEM

TEM analysis is used to find the average diameter of the surface and to magnification of objects in a small size. Carbon dots shaped like a ball in the form of dots.

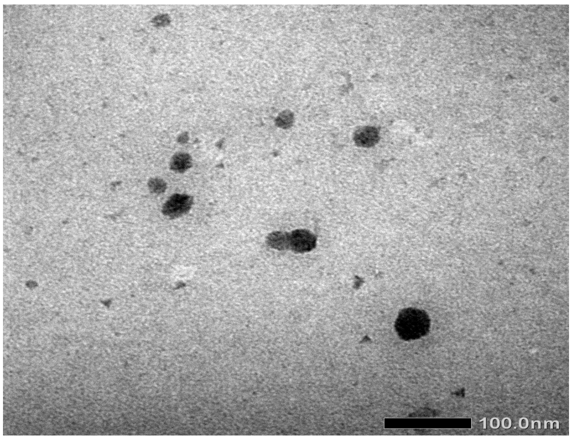


Figure 7: Results of TEM carbon dots with a dispersion of 400000 times.

Carbon dots is a new class of carbon nanomaterials under 10 nm in size (Baker & Baker, 2010) based on the calculation of Carbon dots diameter measurements that have been carried out using the Image J application, obtained the average diameter of C-dot soot in coconut shell is 1.50 nm. The particle size distribution illustrated in Figure 8.

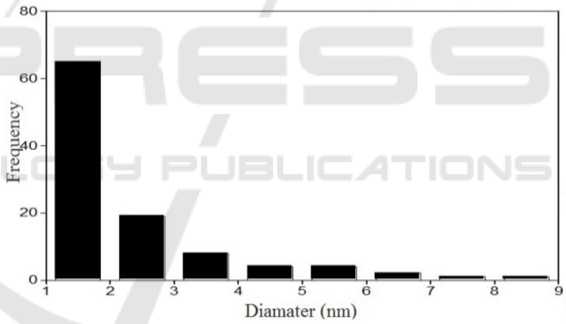


Figure 8: Result average diameter.

### 3.4.4 Analysis of the Function Group with using FT-IR

Functional group analysis is performed using Opus Alpha's Bruker FT-IR. Wave numbers obtained from coconut shell soot and Carbon dots can be seen in Figure 9.

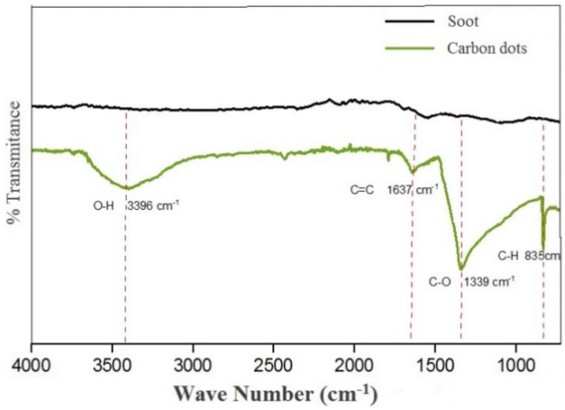


Figure 9: Wave numbers of coconut shell soot and C-dots obtained using FT-IR.

In addition to measurements taken for Carbon Dots samples, FT-IR measurements were also carried out for samples of coconut shell soot. This can be seen in Figure 9, and the interpretation of functional groups seen in the picture explain that there are differences in the absorption peaks between Carbon dots samples and soot of coconut shells. Coconut shell soot does not produce peak O-H absorption. Carbon dots of coconut shell soot produce peak O-H at wave number 3396 $cm^{-1}$. These results explain that in the structure of Carbon dots that have been synthesized there are carboxylic functional groups. This is consistent with the results on Carbon dots coconut shell soot. Based on experiments reported by Hu et al. (2014), coal also has a C = C uptake at a wavelength of 1585 $cm^{-1}$. These results are in accordance with the Carbon dots that have been synthesized, although experiencing a shift that is at wave number 1637 $cm^{-1}$ and at wave number 1339 $cm^{-1}$ for C-O and 835 $cm^{-1}$ for C-H uptake.

# 4 CONCLUSIONS

Carbon dots can be made from coconut sheell soot with acid oxidation method. The yield of Carbon dots obtained was 87% of the initial weight of the soot. The FT-IR spectrum shows that the Carbon dots formed produce OH group absorption at 3396 $cm^{-1}$, absorption of C = C at wave number 1637 $cm^{-1}$, absorption of CO groups, and CH at wave number 1339 $cm^{-1}$, and 835 $cm^{-1}$. Analysis with Transmission Electron Microscopy shows that carbon dots has an average diameter of 1.50 nm. Absorbance spectrum analysis (UV-Vis) results in the appearance of new uptake showing the electron transition at a wavelength of 307 nm and giving green fluorescence under UV light.

# REFERENCES


Arismunndar, W. (2002). *Motor Bakar Torak* (5th ed.). Institut Teknologi Bandung.

Baker, S. N., & Baker, G. A. (2010). Luminescent Carbon Nanodots: Emergent Nanolights. *Angewandte Chemie International Edition*, *49*, 44. https://doi.org/10 .1002 / anie. 200906623

Hu, C., Yu, C., Li, M. Y., Wang, X., Yang, J., Zhao, Z., Eychmuller, A., Sun, Y. P., & Qiu, J. (2014). Chemically tailoring coal to fluorescent carbon dots with tuned size and their capacity for Cu(II) detection. *Small*, *10*(23), 4926–4933.

Li, H., Kang, Z., Liu, Y., & Lee, S. T. (2012). *Carbon nanodots: synthesis, properties and applications*. https://doi.org/doi:10.1039/c2jm34690g

Liu, H., Ye, T., & Mao, C. (2007). Preparation Of Flourescent Carbon Nanoparticles From Candle Soot. *Angewandte Chemie International Edition*, *46*, 6473–6475. https://doi.org/10.1002/ anie.200701271

Marpongahtun, Gea, S., Muis, Y., Andriayani, Novita, T., & Piliang, A. F. (2018). Synthesis of Carbon Nanodots from Cellulose Nanocrystals Oil Palm Empty Fruit by Pyrolysis Method. *Journal of Physics: Conference Series*, *1120*(1), 1–6. https://doi.org/10.1088/1742-6596/1120/1/012071

Nisa, A. K. (IPB). (2014). *Sintesis nanopartikel karbon berfluoresens*.

Oladeji, J. (2010). Fuel characterization of briquettes produced from corncob and rice husk resides. *Pacific Journal of Science and Technology*, *11*(1), 101–106.

Qu, S. G., Wang, X., Lu, Q., Lie, X., & Wang, L. (2012). A Biocompatible Fluorescent Ink Based on Water-Soluble Luminescent Carbon Nanodots. *Angewandte Chemie*, *124*, 1–5. https://doi.org/10.1002/amge.201206791

Ray, S. C., Saha, A., Jana, N. R., & Sarkar, R. (2009). Fluorescent Carbon Nanoparticle: Synthesis, Characterization, and Bioimaging Application. *J Phys C*, *113*(43), 18548–18551. https://doi.org/10.1021/jp905912n

Savitskii, D. P. (2017). Preparation and characterization of colloidal dispersions of graphene-like structures from different ranks of coals. In *Ranliao Huaxue Xuebao/Journal of Fuel Chemistry and Technology* (Vol. 45, Issue 8, pp. 897–907).

Tan, M., Zhang, L., Tang, R., Song, X., Li, Y., Wu, H., Wang, Y., Lv, G., Liu, W., & Ma, X. (2013). Enhanced Photoluminescence and Characterization of Multicolor Carbon Dots Using Plants Soot as a Carbon Source. *Talanta*, *115*, 950–956.

Ye, R., Xiang, C., Lin, J., Peng, Z., Huang, K., Yan, Z., Cook, N. P., Samuel, E. L. G., Hwang, C. C., Ruan, G., Ceriotti, G., Raji, A. R. O., Martí, A. A., & Tour, J. M. (2013). Coal as an abundant source of graphene quantum dots. *Nature Communications*, *4*, 1–7. https://doi.org/10.1038/ncomms3943