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Original Research Article

# Hydrothermal Carbonization of Coconut Pulp and the Adsorption Activity toward Methylene Blue

Octakireina Liesaini Daefisal [1], Demi Dama Yanti [1], Muhamad Alvin Reagen [2], Salprima Yudha S.[2,3]*, John Hendri[4]

[1] *Department of Chemistry, Institut Teknologi Sumatera (ITERA), Jl. Terusan Ryacudu, Way Huwi, Kec. Jati Agung, Province of Lampung, Indonesia*
[2] *Department of Chemistry, Faculty of Mathematics and Natural Sciences, Universitas Bengkulu (UNIB), Jalan W.R, Supratman, Kandang Limun, Kota Bengkulu 38122, Indonesia*
[3] *Research Center of Sumatera Natural Products and Functional Materials (RC-SuNaPFuMa) Universitas Bengkulu (UNIB), Kandang Limun, Kota Bengkulu 38122, Indonesia*
[4] *Department of Chemistry, Faculty of Mathematics and Natural Sciences, Universitas Lampung (UNILA), Jalan Sumantri Brojonegoro No. 1 Bandar Lampung, 35145, Indonesia*

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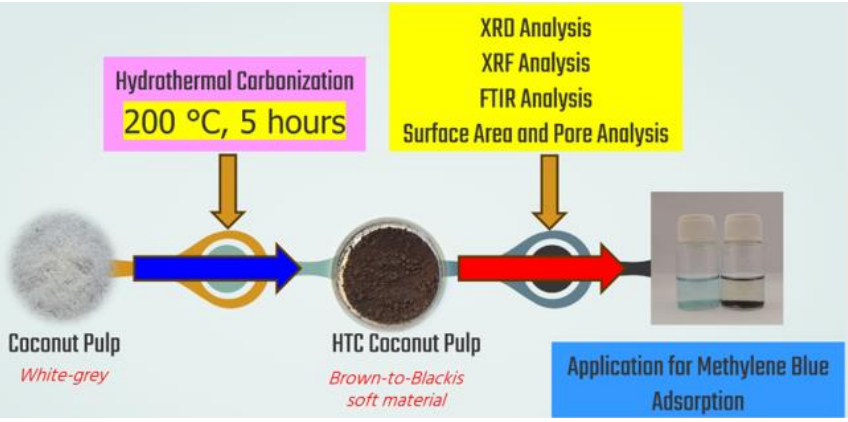
**ABSTRACT**

This study aimed to investigate alterations in the physical and chemical properties resulting from hydrothermal carbonization process applied to dried coconut pulp samples. The samples were passed through a 50-mesh sieve, immersed in demineralized water, and subjected to heating in an autoclave soaked in silicone oil at 200 °C for 5 hours. Hydrochar product (HTC-coconut pulp) of the treatment is a black powder characterized using several instruments. The results of X-ray diffraction (XRD) analysis showed that the peaks in coconut pulp occurred at 2θ: 16.1°, 20.3°, and in the corresponding HTC-coconut pulp at 2θ: 20.2°, 21.2°. Meanwhile, analysis using FTIR showed a significant change where the peaks were at wavenumber ($cm^{-1}$) 3603, 2926, 2855, 1746, 1462, 1372, and 1155. The peaks detected in HTC-coconut pulp were at wavenumber ($cm^{-1}$) 2929, 2849, 1713, 1468, 1290, 1117, and 1057. The results of X-ray fluorescence (XRF) analysis showed several elements such as Al, P, S, Cl, K, and Ca, while HTC-coconut pulp showed Al, Si, P, S, Cl, K, and Ca. A simple application of the two types of materials was as an adsorbent for a simulated methylene blue (MB) solution. According to UV-Vis spectrophotometry absorbance before and after treatment, HTC-coconut pulp showed a slightly higher absorbency compared to normal coconut pulp.

**GRAPHICALABSTRACT**




* Corresponding author: Yudha S., Salprima
✉ E-mail: salprima@unib.ac.id

## Introduction

Coconut milk preparation is conducted by squeezing a specific quantity of the fruit, resulting in pulp residue [1]. The limited instances of reported applications for coconut pulp contribute value to by-product and an example is the use as an additive in the manufacturing of cow dung biogas [2]. This material is also used as an emulsifier and stabilizer in ice cream making due to the protein and carbohydrate content [3-4]. Several studies examined the use as an additive or substitute in foods such as jam [5], steamed brownies [6], cereal [7], and klenyem cake [8]. In addition, the cellulose in coconut pulp has also been developed in the laboratory to make the paper used in the chromatographic process [9]. Coconut pulp protein was extracted using a range of NaOH concentrations, followed by salting with $(NH_4)_2SO_4$ producing a coconut pulp protein concentrate with a content of 71.30% and a yield of 14.42% [10].

Coconut pulp contains cellulose fibers and other compounds attractive for conversion into carbon and other derivatives. Recent studies have shown that with the combination of hydrothermal treatment and acid hydrolysis, this material can be used as a feedstock for sugar production [11]. More recent results reported that coconut pulp could also serve as a source for the bioethanol production when pre-treated using hydrothermal process at 100 degrees with alkali treatment [12]. The material has been developed into carbon through a heating activation system using zinc chloride and a horizontal furnace system heated at 300 °C for 1 hour under a nitrogen atmosphere. The resulting carbon solids have been crushed and passed through a 30-mesh sieve. The solids obtained are effective as triclosan adsorbents following pseudo-second-order process kinetics [13]. Other studies examined the manufacture of carbon through roasting, and then activating the element using an HCl solution. The results had the highest ash and water content of 0.52% and 6.67%, respectively [14]. According to the reported results, there has been no investigation into alterations in the fundamental properties of coconut pulp following the conversion to carbon through hydrothermal carbonization route, and the subsequent use as an adsorbent for methylene blue (MB) in water. Hydrothermal carbonization includes the process of systematically converting a carbon-containing substance into a well-structured form through heat treatment while using a solvent. An advantageous aspect of hydrothermal carbonization is the capability to convert biomass into carbonaceous solids without energy-intensive drying methods [15-17]. Many studies developed a method to obtain materials with better characteristics than the precursors. Extensive study has been conducted to address the removal of detrimental dyes from water using diverse adsorbents, such as carbon materials, metal oxides, and polymer composites. Activated carbons derived from various biomass sources show varying capabilities in adsorbing MB [18]. This is interesting because the abundance promises the sustainability of adsorbent materials. Different studies developed a composite material using magnetic hydroxyapatite/$Fe_3O_4$ nanospheres to remove brilliant green (BG) and Coomassie brilliant blue (CBB) from the water environment. This composite has high adsorption capabilities in the removal of dye compounds from the water environment [19]. Moreover, an epichlorohydrin-crosslinked Schiff base chitosan/$Fe_2O_3$ composite was also developed in the laboratory and applied as an adsorbent for methyl green removal [20].

From the perspectives described, there are many opportunities to convert by-products from human activities into more functional materials, including applications for water environment management. This can be seen in active efforts to remove dyes from the water environment using

various types of adsorbents. Therefore, by-products of coconut milk production were analyzed as carbon materials through hydrothermal carbonization process. The characteristics of the carbonization product are interesting in fundamental chemical systems, and the implications for possible applications are an added value in environmental management.

## Experimental

### *Reagents and solutions*

The main materials used were coconut pulp obtained from a coconut milk sales local market in Bengkulu, Indonesia, demineralized water, and methylene blue (Merck).

### *Instrumentations*

The instruments used to study HTC-coconut pulp and coconut pulp samples were X-ray diffraction (XRD) (X'PERT Powder-PANalytical PW 30/60), X-ray Spectro-fluorescence (XRF) (NEX DE, Rigaku), Fourier transform infrared (FTIR) (Bruker), as well as a surface and porous analyzer ((TriStar II 302)). Furthermore, UV-Vis spectrophotometer (Agilent 60) was used to analyze the solution samples.

### *Methods*

Pulp was dried in the sun for 5 days and sieved using a 50-mesh sieve. The powder was accurately weighed, amounting to a maximum of two grams, and placed into PTFE autoclave reactor equipped with a magnetic rod. Subsequently, 20 mL demineralized water was carefully introduced into the container, which was put into the autoclave reactor and closed tightly. The reactor was put into silicon oil heated to 200 °C on a hotplate-magnetic stirrer for 5 h. After completion, the reactor was removed and the reaction mixture cooled to room temperature. The mixture of solids and liquids was subjected to repeated decantation separations. The main liquid obtained from the separation was measured for pH using a universal pH indicator. Furthermore, it was placed on Petri dish and heated in the sun to evaporate the remaining water attached to the sample.

The dried samples in the form of black powder were characterized using several analyses, namely XRD, FTIR, XRF, and surface analyzer. For comparison, the same analysis was carried out on dried coconut pulp powder samples. To examine the ability of the two materials to adsorb toxic dyes, MB was selected as a representation. In the first step, MB solution was made with a concentration variation of 0, 4, 8, 12, 14, and 16 ppm. The solution was analyzed using UV-Vis spectrophotometer, and the absorbance value was obtained. Meanwhile, plotting between the concentration and absorbance obtained a linear regression equation. The equation was used to determine the concentration of MB solution before and after treatment by an adsorbent. The initial and final concentration values were used to calculate the obtained percentage adsorption on MB.

A total of 10 mg coconut pulp or HTC-coconut pulp was put into different bottles, each containing 5 mL MB 14 ppm, followed by gentle shaking, and the mixture was allowed to stand for 24 hours. In addition, a pipette was used to transfer 3 mL liquid into a cuvette for analysis using UV-Vis spectrophotometer. To calculate the final concentration of MB after treatments, the absorbance value obtained is entered into the previously derived linear regression equation.

## Results and discussion

Coconut pulp that passes the 50-mesh sieve has a grayish-white color, as displayed in Figure 1a. The results of heating coconut pulp at hydrothermal reaction temperature produced a reaction mixture in the form of black solids and

blackish-brown solutions. Examination of the solution following the reaction cessation showed that pH of the solution resulting from hydrothermal reaction was approximately ±5. This phenomenon may be attributed to the decomposition process of coconut pulp, leading to the formation of intermediate compounds showing acidity and solubility in water. The separation of the mixture using the decantation method is considered sufficient to separate the two different phases. The black solid left at the bottom of hydrothermal reactor (Figure 1b) is the main sample in the characterization process.

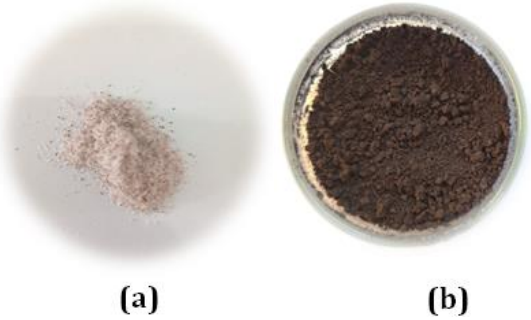



**Figure 1.** Image of (a) coconut pulp and (b) HTC-coconut pulp.

The results of XRD analysis (in Figure 2) showed that the peaks in coconut pulp occurred at 2θ: 16.1°, 20.3°. HTC-coconut pulp obtained distinct peaks at 2θ values of 20.2° and 21.2°. The results were in line with hydrothermal synthesis of carbon derived from other biomass or precursors [21-25]. Analysis using FTIR (Figure 3(a and b)) showed a significant change where the peaks in coconut pulp were at wavenumbers 3603, 2926, 2855, 1746, 1462, 1372, and 1155 $cm^{-1}$.

The peaks detected in hydrocar were at wavenumber 2929, 2849, 1713, 1468, 1290, 1117, and 1057 $cm^{-1}$. The results were in line with previous studies examining FTIR analysis of several cellulose sources and other carbohydrates [26-30]. The proximate analysis reported protein, carbohydrate, and fat content as major compound groups [31].

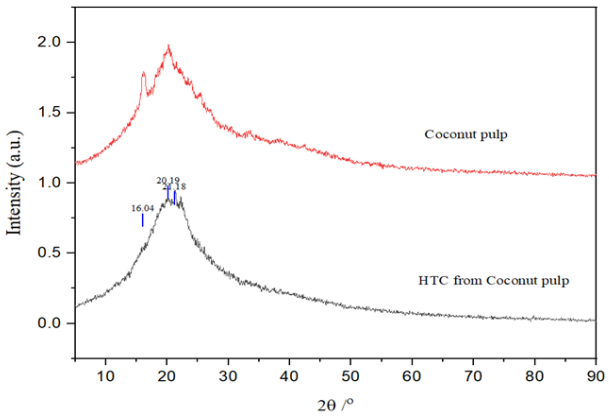



**Figure 2.** XRD pattern of coconut pulp and HTC from coconut pulp.

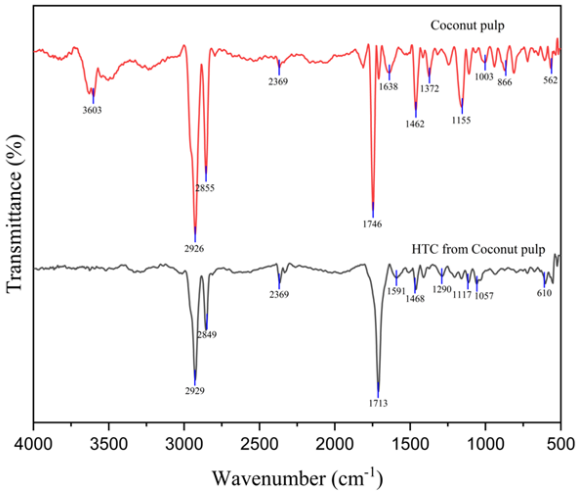



**Figure 3**. FTIR spectroscopy pattern of coconut pulp and HTC from coconut pulp.

Further study based on XRF analysis showed changes in the content of some mineral elements. Table 1 shows the presence of major elements such as Al, P, S, Cl, K, and Ca in low concentrations in coconut pulp. This is most likely due to the decomposition of the main compound in coconut pulp, namely cellulose, which has turned into carbon. Another interesting point is the detection of silicon in HTC-coconut pulp (Table 2).

**Table 1.** XRF elemental analysis of coconut pulp

| Element | Al | P | S | Cl | K | Ca |
|---|---|---|---|---|---|---|
| % | 2.06 | 0.276 | 0.203 | 0.302 | 0.537 | 0.150 |

**Table 2.** XRF elemental analysis of HTC-coconut pulp

| Element | Al | Si | P | S | Cl | K | Ca |
|---|---|---|---|---|---|---|---|
| % | 2.26 | 0.309 | 0.426 | 0.356 | 0.432 | 0.727 | 0.223 |

Surface analysis, pore volume, and diameter were investigated to determine the possible changes caused by heat treatments, as shown in Figure 4 (a and b). BET surface area analysis showed that the original and HTC-coconut pulp had BET surface area of 9.8750 $m^2/g$ and 8.5317 $m^2/g$, respectively. These results showed that heating the sample at 200 °C decreased the surface area. The phenomenon was due to the aggregation during the final treatment of the materials after hydrothermal reaction was terminated. Some studies have been carried out to examine the impact of pressure and temperature on hydrothermal carbonization reactions derived from biomass [32-36]. In contrast, the pore volume of coconut pulp was 0.015 $cm^2/g$ with an adsorption pore diameter of 6.06 nm. The corresponding HTC-coconut pulp had a pore volume of 0.076 $cm^2/g$ with an adsorption pore diameter of 35.76 nm.

The adsorption ability of samples toward MB in a simulated solution was tested to investigate the ability of these two types of sustainable materials to work. MB is a compound often used as a textile dye and toxic when discharged into the environment [37-40]. Several concentrations of MB solution were plotted against absorbance at a wavelength of 665 nm. The measurement results of these standard solutions in the range of 200-800 nm can be seen in Figure 5.

Figure 5 showed a direct correlation between the concentration of MB and the absorbance measured by UV-Vis spectrophotometer under the Lambert-Beer law. In addition, a linear regression equation can be derived by plotting concentration against absorbance by assessing the absorbance values of a range of MB standard solutions.

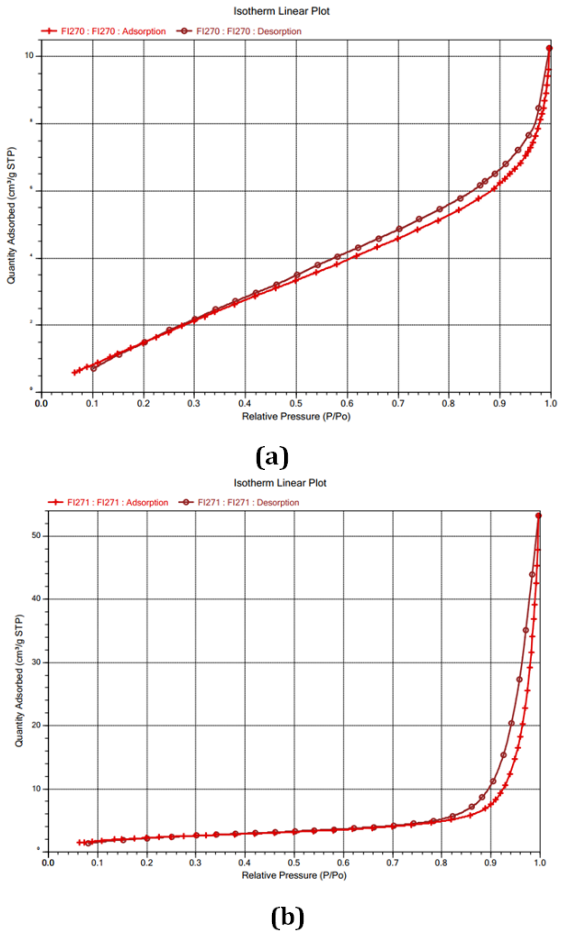



**Figure 4**. BET surface analysis of (a) coconut pulp and (b) HTC-coconut pulp.

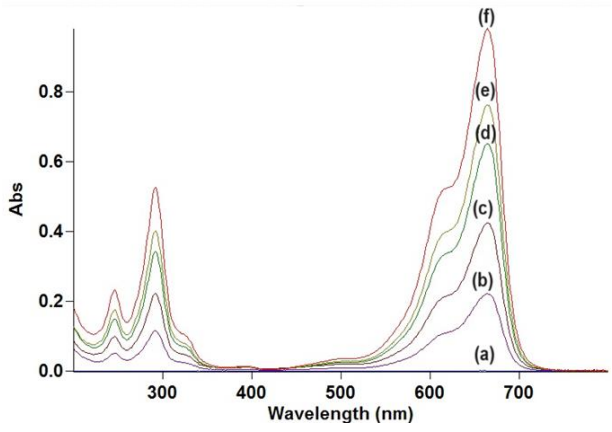



**Figure 5**. UV-Vis spectroscopy analysis of the MB standard solution: (a) blank solution, (b) 4 ppm, (c) 8 ppm, (d) 12 ppm, (e) 14 ppm, and (f) 16 ppm.

This equation serves as the foundation for determining the initial concentration of the solution used as the treated sample and the final solution after treatment by the two available types of adsorbents. Furthermore, two types of adsorbents were applied to a simulated 14 ppm MB solution for an equivalent duration and mass to assess the dye absorption capabilities. After separating both solutions from their solids, distinctive color variations were observed. The solution treated with coconut pulp powder adsorbent retained a vibrant blue color, while treated with HTC-coconut pulp appeared clear. Based on the visual observation, HTC-coconut pulp showed a greater ability for absorbing the blue dye from MB solution. This conclusion is supported by the absorbance analysis of the final solution conducted using UV-Vis spectrophotometer, as depicted in Figure 6.

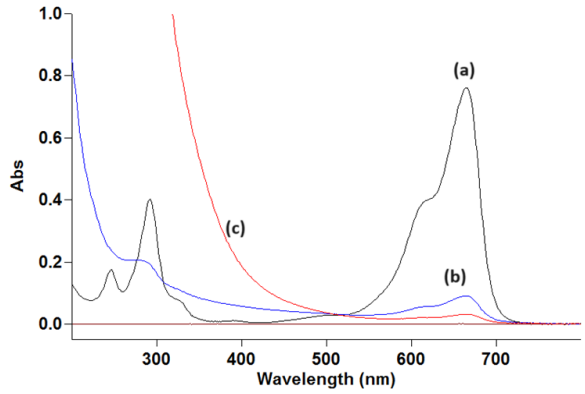



**Figure 6.** UV-Vis spectroscopy analysis of the MB standard solution (a) initial solution, (b) solution after adsorbed by coconut pulp, and (c) solution after adsorbed by HTC-coconut pulp.

The analysis results showed that the peak of MB at 665 nm (Figure 6a) was reduced in the solution subjected to treatment using coconut pulp adsorbent (Figure 6b). The solution treated with HTC-coconut pulp had a lower absorbance at the wavelength detected by UV-Vis spectrophotometry (Figure 6c). Based on the absorbance value of MB solution, coconut pulp and HTC-coconut pulp provided an adsorption percentage of 87.8% and 99.7%, respectively. It is clear that the HTC-coconut pulp had a greater adsorption percentage due to larger pore area and volume. The size of the adsorption pore diameters significantly influenced the power of the materials on MB compounds. The results using HTC produced from various biomass sources also showed a good percentage, which could reduce the risk of side effects from toxic compounds used as dyes [41-45].

## Conclusion

To sum up, coconut pulp was converted to carbon through hydrothermal carbonization process. HTC-coconut pulp showed new characteristics, such as different XRD patterns and XRF analysis identified compounds containing silicon. Furthermore, the adsorption percentage of methylene blue by HTC-coconut pulp and coconut pulp was 99.7% and 87.8%, respectively. The greater adsorption power of HTC-coconut pulp was attributed to the larger pore and volume compared to coconut pulp. The results were expected to provide new insights into the use of by-products of human activities in the framework of chemical development and the applications.

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## Disclosure statement

No potential conflict of interest was reported by the authors in this study.

## Orcid

*Octakireina Liesaini Daefisal*: 0009-0005-4451-9781
*Demi Dama Yanti*: 0000-0002-9693-0872
*Muhamad Alvin Reagen*: 0009-0005-8693-1539
*Salprima Yudha S.*: 0000-0002-3095-5284
*John Hendri*: 0000-0002-8119-0701

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