

Synthesis and Characterization of Hydrochar by Hydrothermal Carbonization of Pineapple Crown Waste

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Abstract. *Hydrochar, produced through the hydrothermal carbonization (HTC) of biomass, presents a sustainable alternative to conventional carbon-based environmental sorbents. In this study, pineapple crown waste, an often-discarded agricultural byproduct, was used as the feedstock for hydrochar synthesis via HTC. The process was optimized at 200°C for 15 hours to enhance carbonization efficiency and improve the hydrochar's surface characteristics. Structural, morphological, and functional group analyses were performed using scanning electron microscopy (SEM), Fourier-transform infrared spectroscopy (FTIR), and UV-visible spectroscopy. FTIR analysis revealed the presence of O-H, C-H, C=O, and C=C functional groups in the hydrochar. SEM analysis showed that the hydrochar had sheet-like structures. UV-vis spectroscopy further confirmed the presence of C=C and C=O bonds, highlighting their significant role in the material's functionality. Overall, the hydrochar demonstrated favorable porosity and surface functionalization, supporting its potential for further application.*

Keywords: *biowaste, pineapple crown, hydrochar, hydrothermal*

1. Introduction

Agricultural waste management is a pressing global issue, as the accumulation of waste often leads to significant environmental challenges, such as land and water pollution (Gontard et al., 2018). Among the various types of agricultural waste, pineapple crown waste, a byproduct of the pineapple industry, is typically discarded or underutilized, exacerbating waste management problems (Fouda-Mbanga & Tywabi-Ngeva, 2022). Pineapple crown waste, like other agricultural residues, is abundant in lignocellulosic components, which positions it as a promising feedstock for value-added products, including the production of hydrochar through hydrothermal carbonization (HTC) (Inoue, 2016; Luthfi, Fukushima, Wang, & Takisawa, 2024). This transformation not only addresses waste disposal concerns but also contributes to sustainable material production, highlighting the dual benefits of agricultural waste valorization.

Hydrochar is a carbon-rich material with a wide range of applications, including energy storage, soil enhancement, and environmental remediation as an adsorbent for pollutants (Ojewumi & Chen, 2024). The HTC process, a thermochemical method, converts biomass into hydrochar at relatively low temperatures (180-250°C) in the presence of water, making it an energy-efficient and environmentally friendly approach (Pauline & Joseph, 2020). One of the key advantages of HTC is its ability to process wet biomass directly, eliminating the need for energy-intensive drying steps (Petrović et al., 2024). Furthermore, the addition of activating agents during the HTC process can significantly improve the surface area, porosity, and functional groups of the resulting

hydrochar, enhancing its adsorption properties for environmental applications (Falco et al., 2013).

In this study, pineapple crown waste was converted into hydrochar using an HTC process optimized for 200°C and 15 hours of treatment time. This temperature and duration were found to enhance carbonization efficiency and improve the surface functionalization of the hydrochar. To fully characterize the structural and chemical properties of the produced hydrochar, scanning electron microscopy (SEM) was employed to analyze its morphology, while Fourier-transform infrared spectroscopy (FTIR) and UV-vis spectroscopy were used to identify the functional groups present. This study aims to demonstrate how pineapple crown waste can be transformed into a value-added material with potential environmental and industrial applications, contributing to both sustainable waste management and resource recovery.

2. Method

2.1. Materials and Chemicals

The pineapple fruit was purchased from a traditional fruit market in Aceh Besar district, Aceh province, Indonesia. The pure water was bought from the Chemical Engineering Lab, Universitas Syiah Kuala, Indonesia, and used throughout the preparation of solutions in this work and the whole experiment.

2.2. Preparation of Hydrochar

The hydrochar derived from pineapple crown was produced by a hydrothermal treatment method as reported in our previous paper with a little modification [9]. First, the pineapple crown waste was washed, dried for several days under sunlight, ground into powder, and sieved using a 40-mesh sieve. Then, 5 grams of the pineapple crown waste powder was placed in a 100 mL hydrothermal autoclave. Pure water (80 mL) was added, and the mixture was stirred for 5 minutes at room temperature using ultrasonication. The mixture was then heated in an oven at 200°C for 15 hours. The resulting product is cleaned using centrifugation (4000 rpm, 10 min) with pure water and ethanol several times until the supernatant becomes neutral. The product was dried at 100°C for 3 h and stored in a desiccator for 24 h before being demolished into fine powder as the hydrochar material.

2.3. Characterization Methods of Hydrochar

The hydrochar was identified to obtain the best candidate for adsorbent. The optical characteristics of the hydrochar were measured and analyzed. The UV-Vis spectrum was recorded by Agilent 8453 UV-Vis Spectrometer using a quartz cuvette of 10 mm path length and 3 mL volume. The surface functional groups of the hydrochar were examined by an ALPHA FTIR spectrophotometer from Buker. The surface morphology was determined using a SEM microscope from JEOL (JSM-6510LA).

3. Results and Discussions

3.1. Production of Hydrochar from Pineapple Crown Waste

The one-step HTC process successfully transformed pineapple crown waste into hydrochar. The process parameters, optimized at 200°C for 15 h, resulted in efficient carbonization and enhanced surface properties. The physical and chemical characteristics of the hydrochar were assessed through various analytical techniques to understand its properties (Andrade, Porto, Moreira, Batistela, & Scaliante, 2023). In this study, an HTC treatment to obtain hydrochar from pineapple crown waste as a precursor was performed

as illustrated in Figure 1. The pineapple crown waste powder was transformed into microsphere cores through dehydration and polymerization in the hydrothermal autoclave at 200°C for 15 h. The dispersed colloidal particles were then formed from the microsphere cores by the adsorption of surrounding molecules and lastly developed into hydrochar material (Zulfajri, Kao, & Huang, 2021). The separation of the solution phase from the upper solution and bottom precipitate was carried out using centrifugation. The hydrochar was obtained from residue after washing several times with excess water and ethanol was dried in an oven to obtain a brownish-black powder.

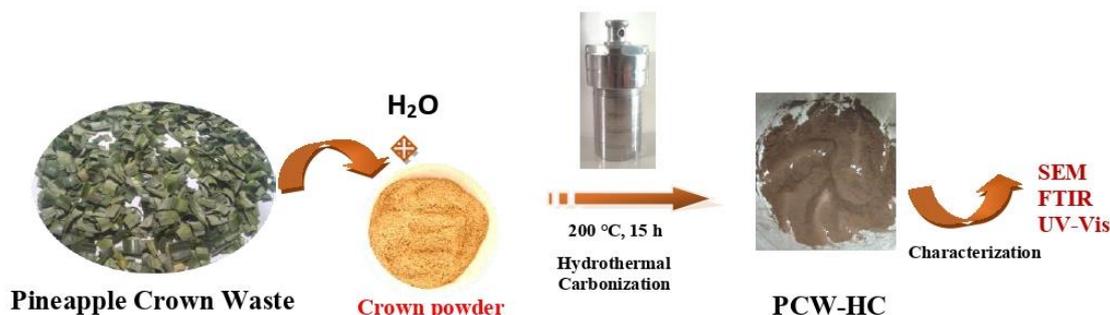


Figure 1. The preparation and characterization of hydrochar

3.2. Structural and Morphological Characterization

The result of the hydrochar synthesis was further characterized using a Scanning Electron Microscope (SEM) with a magnification of 2000x. The result of SEM observations of hydrochar can be seen in Figure 2. The SEM analysis revealed the surface morphology of the hydrochar. The SEM image of hydrochar showed relatively well-defined surfaces with little flaking observed on the surface. The porosity is vital for increasing the interaction sites between the adsorbent and molecules, facilitating higher pollutant removal efficiency (Haleem, Shafiq, Chen, & Nazar, 2023).

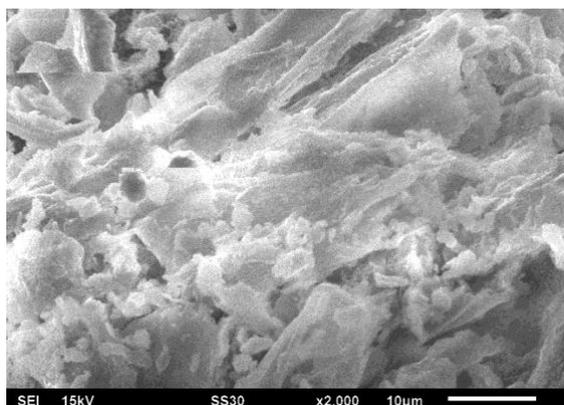


Figure 2. SEM images of the hydrochar with 2000x magnification

3.3. Functional Group Analysis

FTIR was employed to identify the functional groups present on the surface of hydrochar. The FTIR spectrum indicated the presence of hydroxyl (-OH), carboxyl (-COOH), and carbonyl (C=O) groups (Jawad, Abdulhameed, & Mastuli, 2020; Xie, Jin, Chen, & Jiang, 2017). The typical FTIR spectrum of the hydrochar is shown in Figure 3. The hydrochar has several bands observed at 3250 cm^{-1} (O-H bonds), 2900 cm^{-1} (C-H bonds), 1600 cm^{-1} (C=O bonds), 1550 (C=C bonds), 1400 cm^{-1} (-COOH bonds), and 1300

cm^{-1} (C-H bonds) and 1250 cm^{-1} (C-O-C bonds). Several other bands observed at lower wavenumbers also demonstrated the existence of various bonds such as C-H, O-H, and C-O (Elhassan, Kooh, Chou Chau, & Abdullah, 2024). The obtained data shows that the hydrochar contained oxygen-functional groups that derived during the carbonization process of the pineapple crown waste.

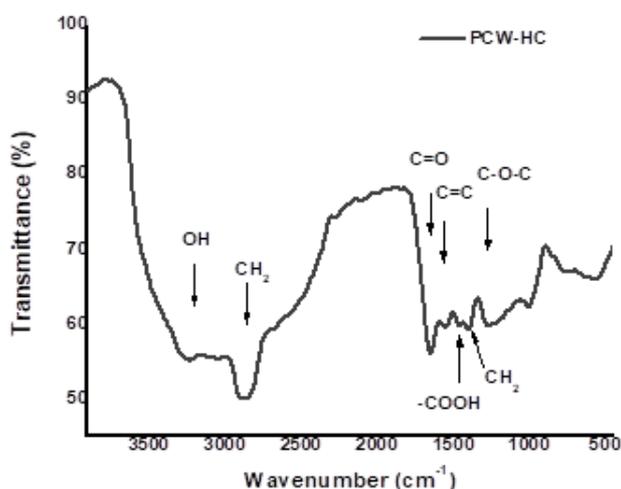


Figure 3. FTIR spectrum of hydrochar

Figure 4 depicts the UV-vis spectrum of the hydrochar solution. UV-vis spectrum was measured by mixing 10 mg of hydrochar with 3 mL ultrapure water and sonicating the mixture for 5 min to break up the hydrochar particles into smaller fractions (Zulfajri et al., 2021). The absorbance peaks of C=C with $\pi-\pi^*$ energy transitions and C=O bonds with $n-\pi^*$ energy transitions were observed at $\sim 275 \text{ nm}$ and $\sim 325 \text{ nm}$, respectively. The UV-Vis spectrum proved the existence of oxygen-containing groups that attached to the carbon skeleton in the hydrochar surface.

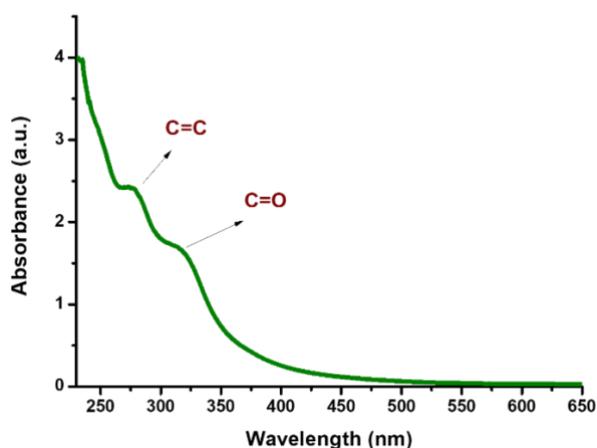


Figure 4. The UV-Vis spectrum of hydrochar solution

4. Conclusions

This study demonstrates that pineapple crown waste can be effectively utilized as a sustainable feedstock for producing hydrochar through the HTC process. The hydrochar exhibits excellent structural, morphological, and surface properties, making it a promising

material for environmental remediation as a sustainable adsorbent. Characterization using SEM, FTIR, and UV-vis spectroscopy revealed its surface structure, morphology, and functionalization, highlighting its potential in waste valorization. This approach contributes to sustainable agricultural waste management and advances the development of biowaste-based adsorbents for environmental applications, showcasing the valuable use of agricultural waste as a resource.

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6. References

- Andrade, J. G. da S., Porto, C. E., Moreira, W. M., Batistela, V. R., & Scaliante, M. H. N. O. (2023). Production of hydrochars from *Pinus caribaea* for biosorption of methylene blue and tartrazine yellow dyes. *Cleaner Chemical Engineering*, 5, 100092. doi:10.1016/j.clee.2022.100092
- Elhassan, M., Kooh, M. R. R., Chou Chau, Y. F., & Abdullah, R. (2024). Hydrochar from *Shorea* spp.: a dual-purpose approach for sustainable biofuel and efficient methylene blue adsorbent. *Biomass Conversion and Biorefinery*. doi:10.1007/s13399-024-05376-w
- Falco, C., Marco-Lozar, J. P., Salinas-Torres, D., Morallón, E., Cazorla-Amorós, D., Titirici, M. M., & Lozano-Castelló, D. (2013). Tailoring the porosity of chemically activated hydrothermal carbons: Influence of the precursor and hydrothermal carbonization temperature. *Carbon*, 62, 346–355. doi:10.1016/j.carbon.2013.06.017
- Fouda-Mbanga, B. G., & Tywabi-Ngeva, Z. (2022). Application of Pineapple Waste to the Removal of Toxic Contaminants: A Review. *Toxics*, 10(10), 561. doi:10.3390/toxics10100561
- Gontard, N., Sonesson, U., Birkved, M., Majone, M., Bolzonella, D., Celli, A., ... Sebok, A. (2018). A research challenge vision regarding management of agricultural waste in a circular bio-based economy. *Critical Reviews in Environmental Science and Technology*, 48(6), 614–654. doi:10.1080/10643389.2018.1471957
- Haleem, A., Shafiq, A., Chen, S. Q., & Nazar, M. (2023, February 1). A Comprehensive Review on Adsorption, Photocatalytic and Chemical Degradation of Dyes and Nitro-Compounds over Different Kinds of Porous and Composite Materials. *Molecules*. MDPI. doi:10.3390/molecules28031081
- Inoue, S. (2016). Hydrothermal Carbonization of Lignocellulosic Biomass. *Carbon*, 104, 263–264. doi:10.1016/j.carbon.2015.12.021
- Jawad, A. H., Abdulhameed, A. S., & Mastuli, M. S. (2020). Acid-fractionalized biomass material for methylene blue dye removal: a comprehensive adsorption and mechanism study. *Journal of Taibah University for Science*, 14(1), 305–313. doi:10.1080/16583655.2020.1736767

- Luthfi, N., Fukushima, T., Wang, X., & Takisawa, K. (2024). Hydrochar as an Alternative to Coal: A Comparative Study of Lignocellulosic and Nonlignocellulosic Biomass. *Resources*, 13(4), 49. doi:10.3390/resources13040049
- Ojewumi, M. E., & Chen, G. (2024). Hydrochar Production by Hydrothermal Carbonization: Microwave versus Supercritical Water Treatment. *Biomass (Switzerland)*, 4(2), 574–598. doi:10.3390/biomass4020031
- Pauline, A. L., & Joseph, K. (2020). Hydrothermal carbonization of organic wastes to carbonaceous solid fuel – A review of mechanisms and process parameters. *Fuel*, 279, 118472. doi:10.1016/j.fuel.2020.118472
- Petrović, J., Ercegović, M., Simić, M., Koprivica, M., Dimitrijević, J., Jovanović, A., & Janković Pantić, J. (2024, January 1). Hydrothermal Carbonization of Waste Biomass: A Review of Hydrochar Preparation and Environmental Application. *Processes*. Multidisciplinary Digital Publishing Institute (MDPI). doi:10.3390/pr12010207
- Xie, R., Jin, Y., Chen, Y., & Jiang, W. (2017). The importance of surface functional groups in the adsorption of copper onto walnut shell derived activated carbon. *Water Science and Technology*, 76(11), 3022–3034. doi:10.2166/wst.2017.471
- Zulfajri, M., Kao, Y. T., & Huang, G. G. (2021). Retrieve of residual waste of carbon dots derived from straw mushroom as a hydrochar for the removal of organic dyes from aqueous solutions. *Sustainable Chemistry and Pharmacy*, 22, 100469. doi:10.1016/j.scp.2021.100469