Evaluation of Carica Papaya Seed Activated Carbon as Basic Catalyst Support: Extent and Base Concentration

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ABSTRACT

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Carica Papaya or Papaya seed is one of the abundant agricultural residues that contain carbon atoms. This study evaluated the role of activated carbon as essential catalyst support. Papaya seeds were dried, crushed, and activated using sulfuric acid. After being chemically activated, carbon was physically activated through heating. At 110 ºC. The synthesized activated carbon was then impregnated with NaOH for contact time variation (180, 240, 300, and 360 minutes) with varying alkaline concentrations (30, 40, 50, and 60%). The synthesized supported catalyst on activated carbon was morphologically characterized through SEM. Additionally, the structure and elemental composition of the supported catalyst were analyzed with EDX. It was found that morphologically, concentration enhanced NaOH dispersion onto activated carbon in positive means with a 20% NaOH concentration increase by 0.6%. On the other hand, impregnation time displayed a maximum point, after which the impregnation process did not proceed significantly from 240 to 360 minutes by 1.25%. Moreover, impregnated activated carbon also presented an identical trend, with Sodium and Oxygen substances being impregnated in the same manner for both impregnation parameter variations. On top of that, the *structure analysis through XRD depicted the potential heterogeneous solid catalytic performance of NaOH supported with activated carbon from papaya seed. The findings showed that activated carbon from Papaya seed with NaOH impregnation has the potential for Biodiesel production as a heterogeneous catalyst.*

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1.Introduction

Currently, over sixty-seven percent of global energy is supported by nonrenewable energy sources [1]. The rapid depletion of fossil fuel sources demands fast and readily available renewable energy sources to fulfill their constantly increasing demand. Biofuel, as an alternative to fossil fuel, has a lower carbon footprint [2]. Biomass that acts as a source of biofuel has a wide variety of potential biofuel products [3]. With more than 17,000 islands [4], Indonesia has substantial plants or biomass available. This enormous availability of biomass residues is still a tiny amount of energy utilized [5]. The area covered with biomass is almost 3 million hectares and its biomass waste potential for energy production is nearly 50,000 MW [6].

One of the readily available and abundant biomass sources in Indonesia is papaya. It is proven by being one of the most Papaya-producing nations among Brazil, Mexico, and India [7]. Papaya is one of the five major tropical fruits ranked after Banana, Mango, and Pineapple [8]. One underutilized residue from the papaya tree is its seed. Papaya seed contains compounds that remain underutilized for value-added applications. The content of Papaya seed is shown in Table 1 [9].

Papaya Seed Contents	Components				
	Fat	Protein	Ash	Carbohydrates	Liauids
Percentage			.47	9.44	71.89

Table 1. Papaya Seed Components

Table 1 shows that Papaya seed contains carbohydrates and ash, which comprise approximately ten percent of its seed, consisting of carbon atoms that can be converted into activated carbon. The use of papaya seeds as raw material for activated carbon synthesis has been studied. Its further use as an adsorbent is also widely understood for dying material, toxic material, and heavy metal adsorptions [10], [11], [12].

Another function of activated carbon, besides the mentioned application studies, is catalyst support. Catalyst support from activated carbon has advantages such as its high surface area, abundance, and ease of preparation [13], [14]. The catalyst support is necessary for heterogeneous catalysts to enhance catalyst performance by maximizing the contacting area of catalyst material to reactants [15]. Among catalytic reactions, transesterification extensively utilizes heterogeneous catalysts. Its product is methyl ester or Biodiesel, one of the biofuels synthesized from natural raw materials. The use of essential heterogeneous catalysts in such reactions, namely CaO, NaOH, KOH, and other necessary materials, has been investigated extensively.

Until now, research on activated carbon from Papaya seed as essential catalyst support has not been comprehensively investigated. This research aimed to analyze and evaluate the performance of papaya seed-based activated carbon for catalyst support function.

2. Research Methodology

2.1. Materials

In this research, the utilized materials were Papaya seed from the local market of Lhokseumawe, H2SO⁴ (98% purity) Merck used as an activating agent, sodium hydroxide (NaOH) Merck, litmus paper, and distilled water. On the other hand, the apparatus employed were analytical balance, hotplate, magnetic stirrer, volume pipette, filter paper, oven, furnace, porcelain cup, beaker glass (Pyrex), Erlenmeyer (Pyrex), volumetric flask, and sieve mesh 80.

2.2. Procedures

The procedures conducted in this research were first activated carbon preparation from Papay seed, followed by NaOH impregnation on the synthesized activated carbon. After that, the NaOHimpregnated activated carbon was characterized. The diagram of the research methodology is shown in Fig. 1.

1) Activated Carbon Preparation

The primary procedure in this research involved synthesizing activated carbon from papaya seeds and impregnating it with a primary chemical. Collected papaya seed was washed and dried under the sun's light for two days. Then, the dried papaya seed was mashed and sieved with 80 mesh size. To synthesize activated carbon, the dried papaya seed needed to be activated. This study managed chemical activation for activating carbon. [16] employed acid such as sulfuric acid. Dried papaya seed was soaked and stirred in a 10% sulfuric acid mixture for 3 hours to achieve a rough, irregular surface in addition to its cavities for surface improvement [17]. After being soaked in an acidic mixture, the activated carbon was dried in an oven at 110ºC for 12 hours and then rinsed with distilled water to neutralize its pH. To evaporate water, synthesized activated carbon was dried in an oven at 110ºC for 2 hours.

2) NaOH Impreganation

The synthesized activated carbon from papaya seed was then impregnated with sodium hydroxide. The method was based on [18], [19], [20]. Fifteen milliliters of NaOH mixture with varying concentrations of 30%, 40%, 50%, and 60% (wt/wt) was mixed with 10 grams of activated carbon. Moreover, the duration of the impregnation step also varied to 180, 240, 300, and 360 minutes, stirring at room temperature. After impregnation, the catalyst with activated carbon support was dried in an oven at 110ºC for 12 hours. To enhance the activity of metal on the activated carbon support, the dried supported catalyst was calcined at 300ºC for 1 hour, under which it was proven to synthesize outstanding products of supported carbon properties, such as dispersed chemicals and morphology [16], [21].

3) Supported Catalyst Characterization

The synthesized supported NaOH catalyst on activated carbon was characterized quantitatively to observe its morphological information and existing substance. Scanning Electron Microscope-Energy Dispersive X-ray spectroscopy (SEM-EDX) was conducted on the samples to comprehend such knowledge. The instrument utilized for analysis was Thermo Fisher Scientific with Oxford Instruments software. X-Ray Diffraction (XRD) was used to determine the crystallinity of the samples.

Fig. 1. Designated Research Procedure

3. Results and Discussion

3.1. Impergnation Time Effect

The impact of the impregnation process on the morphology of the synthesized NaOHimpregnated activated carbon catalyst was analyzed. Fig. 2 shows the effect of the duration of impregnation on morphology with 1500 times magnification.

From Fig. 2. a-d, it can be seen that the accumulation of Sodium particles improved as the extent of the impregnation process proceeded. This can be observed from increasing bumps on the surface of activated carbon [22]. The increasing sodium particle numbers as more time is provided for impregnation time was observed to contribute to a positive correlation between the number of impregnated particles. This is also proved by [23], whose study impregnated carbon dioxide with varying impregnation times. Although an increasing trend was observed in NaOH dispersion, the impregnation process at 360 minutes did not show a significant increase compared to 300 minutes. This was due to the tendency of solute to be desorbed from the surface of a solid [24].

Despite the increasing amount of NaOH being impregnated onto activated carbon with increasing duration time, the percentage of NaOH on the activated carbon was decreasing. This is shown in Fig. 3., which shows EDX analysis results.

Fig. 2. Effect of 40% NaOH impregnation time on activated carbon morphology at 1500x magnification, (a) 180 minutes, (b) 240 minutes, (c) 300 minutes, (d) 360 minutes

Fig. 3. Present Components of Impregnated AC with 40% NaOH (a) 240 minutes (b) 360 minutes

It can be inferred from Fig. 3. a and b that there is a decrease in NaOH content by 1.25% on activated carbon as the impregnation time increases from 240 to 360 minutes. This was possibly due to rising impurities in the samples that affected the depleting weight percentage of NaOH. Removing the impurities percentage, it still can be interpreted that proceeding impregnation time did not affect the impregnation significantly with the presence of maximum capacity for chemical dispersion. This phenomenon was also observed by [25], with their findings showing gradual loading on chemicals and becoming saturated on the surface of carbonous material.

3.2. Concentration Significance

The concentration of solute effects on the impregnation process with variation from 30 to 60% to the synthesized solid's morphology is presented in Fig. 4.

Fig. 4. Effect of 6-hour NaOH impregnation concentration on activated carbon morphology at 1500x magnification (a) 30% (b) 40% (c) 50% (d) 60%

It is observed from Fig. 4. a-d that the solute concentration of NaOH during the impregnation process positively disperses the primary chemical. From Fig. 4. a-d, the impregnated NaOH rose as more NaOH was added to the solution. This is supported by [26], who observed an increasing trend of dispersing chemicals onto solids as solute concentration rose. Despite the rising dispersion of NaOH on the activated carbon, a reduction in the uniformity of dispersing material was exhibited. This was due to many basic chemicals being dispersed to several external spots of activated carbon surface instead of evenly distributed [27]. This phenomenon is distinguished from Fig. 3c-d at 60% NaOH. This was possibly from the impurities during the sample preparation and other study parts. The existence of impurities is confirmed in Fig. 5, which is the EDX analysis of impregnated activated carbon with NaOH.

Fig. 5. Present Components of AC for 6 hours Impregnation (a) 40% NaOH (b) 60% NaOH

It is evident from Fig. 5. a and b that impurities existed in the characterized samples. The impurities were identified as magnesium, silica, sulfur, and aluminum. Apart from presented impurities, NaOH particles dispersed onto activated carbon climbed as the solute (NaOH)

concentration advanced. In particular, Na weight percentage progressed by 0.6% from 31.5% to 31.7% as NaOH concentration as a solute in impregnation solution increased from 40% to 60%. This is coherent with findings by [28] that stated chemical concentration affected impregnated material onto the solid's surface in a positive trend.

3.3. Structure Analysis

The resulting graph of NaOH-impregnated activated carbon is shown in Fig. 6. The span of 2θ angle was conducted in the range of 0º to 80º.

Fig. 6. XRD Analysis on 60% NaOH Impregnated AC at 360 Minutes Impregnation

The structure analysis on impregnated activated carbon with 60% NaOH at 360 minutes shows broad peaks in the range of 2θ between 40º and 50º. Moreover, it also exhibits sharp peaks between 30º and 40º. This is potentially contributed by amorphous structure reflection of the solid's (002) dan (100) planes [29]. Hence, NaOH dispersed solid particles' structure was determined to be amorphous rather than crystalline [30]. This structure property possesses great performance as a solid heterogeneous catalyst that enhances more reactants to be contacted onto the catalyst's surface [31]. Apart from that, the broadening hump around 20º corresponded to a (002) peak with a small fraction of crystallite, a reflection of aromatic layers [32]. This might be due to some impurities during catalyst impregnation and a small amount of sulfuric-containing compounds left over during rinsing. This finding potentially affects the performance of synthesized impregnated activated carbon in transesterification reaction, which was proven to perform more catalytic activity [33].

4. Conclusion

Using papaya seed as a support material for NaOH as a catalyst shows promise due to positive morphological, compositional, and structural findings. Based on the variation of time and elemental concentration during impregnation, these two parameters affected Sodium compound dispersion onto solid particles. NaOH concentration positively affected the dispersion process, while impregnation time experienced a maximum point after which the primary compound did not significantly impregnate activated carbon. Moreover, the structure of impregnated activated carbon

was found to carry substantial performance as a heterogeneous solid catalyst with a porous material to enhance more contacting area. Other potential biomass residues should be explored for their valorization, ensuring more hygienic handling and preparation. Furthermore, Further development could involve optimizing papaya seed-derived activated carbon for industrial transesterification reactions in biodiesel production.

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References

- [1] L. Pang, L. Liu, X. Zhou, M. Hafeez, S. Ullah, and M. T. Sohail, "How does natural resource depletion affect energy security risk? New insights from major energy-consuming countries," *Energy Strategy Reviews*, vol. 54, p. 101460, Jul 2024, doi: 10.1016/j.esr.2024.101460.
- [2] F. U. Nisa, "Chapter 12 Biofuel: A unique solution for the future energy crisis," in *Environmental Sustainability of Biofuels*, K. R. Hakeem, S. A. Bandh, F. A. Malla, and M. A. Mehmood, Eds., Elsevier, 2023, pp. 219–236 doi: 10.1016/B978-0-323-91159-7.00008-4.
- [3] R. K. Srivastava, N. P. Shetti, K. R. Reddy, E. E. Kwon, M. N. Nadagouda, and T. M. Aminabhavi, "Biomass utilization and production of biofuels from carbon neutral materials," *Environmental Pollution*, vol. 276, p. 116731, May 2021, doi: 10.1016/j.envpol.2021.116731.
- [4] L. Dsikowitzky et al., "Java Island, Indonesia," in *World seas: An environmental evaluation*, Elsevier, 2019, pp. 459–490.
- [5] S. Yana, M. Nizar, and D. Mulyati, "Biomass waste as a renewable energy in developing bio-based economies in Indonesia: A review," *Renewable and Sustainable Energy Reviews*, vol. 160, p. 112268, May 2022, doi: 10.1016/j.rser.2022.112268.
- [6] E. Mahidin, M. Zaki, M. Hamdani, R. M. Hisbullah, and H. Susanto, "Potential and utilization of biomass for heat energy in Indonesia: A review," *International Journal of Scientific & Technology Research*, vol. 2, no. 10, pp. 331–344, Oct 2020.
- [7] V. N. Villegas, "Carica papaya L," *Edible fruits and nuts*, vol. 2, 1997.
- [8] H. Wijaya, "Flavour of papaya (*Carica papaya L.*) fruit," *Biotropia (Bogor)*, vol. 20, no. 1, Sep 2013.
- [9] M. Yuniwati and A. Purwanti, "Optimasi kondisi proses ekstraksi minyak biji pepaya," *Jurnal Teknologi Technoscientia*, pp. 75–82, Aug 2008, doi: 10.34151/technoscientia.v1i1.382.
- [10]D. Krishnaiah, C. G. Joseph, S. M. Anisuzzaman, W. Daud, M. Sundang, and Y. C. Leow, "Removal of chlorinated phenol from aqueous solution utilizing activated carbon derived from papaya (*Carica Papaya*) seeds," *Korean Journal of Chemical Engineering*, vol. 34, no. 5, pp. 1377–1384, Mar 2017, doi: 10.1007/s11814-016-0337-6.
- [11]E. A. Idohou et al., "Preparation of activated carbon/chitosan/Carica papaya seeds composite for efficient adsorption of cationic dye from aqueous solution," *Surfaces and Interfaces*, vol. 21, p. 100741, Dec 2020, doi: 10.1016/j.surfin.2020.100741.
- [12]S. K. Yadav, D. K. Singh, and S. Sinha, "Chemical carbonization of papaya seed originated charcoals for sorption of Pb (II) from aqueous solution," *J Environ Chem Eng*, vol. 2, no. 1, pp. 9–19, May 2014, doi: 10.1016/j.renene.2018.11.006.
- [13]B. Narowska, M. Kułażyński, M. Łukaszewicz, and E. Burchacka, "Use of activated carbons as catalyst supports for biodiesel production," *Renew Energy*, vol. 135, pp. 176–185, May 2019, doi: 10.1016/j.renene.2018.11.006.
- [14][W. Mateo et al., "Synthesis and characterization of sulfonated activated carbon as a catalyst for bio-jet fuel production from biomass and waste plastics," *Bioresour Technol*, vol. 297, p. 122411, Feb 2020, doi: 10.1016/j.biortech.2019.122411.
- [15]T. W. van Deelen, C. Hernández Mejía, and K. P. de Jong, "Control of metal-support interactions in heterogeneous catalysts to enhance activity and selectivity," *Nat Catal*, vol. 2, no. 11, pp. 955–970, Nov 2019, doi: 10.1038/s41929-019-0364-x.
- [16]M. Z. Siswarni, L. I. Ranita, and D. Safitri, "Pembuatan Biosorben Dari Biji pepaya (*Carica Papaya L*) untuk penyerapan zat warna," *J. Tek. Kim. USU*, vol. 6, no. 2, pp. 7–13, Jun 2017.
- [17]P. M. Thabede, N. D. Shooto, and E. B. Naidoo, "Removal of methylene blue dye and lead ions from aqueous solution using activated carbon from black cumin seeds," *S Afr J Chem Eng*, vol. 33, no. 1, pp. 39–50, Jul 2020, doi: 10.1016/j.sajce.2020.04.002.
- [18]S. O. B. Ginting, D. Daniel, and N. Hindryawati, "Impregnasi natrium hidroksida pada karbon aktif cangkang jengkol sebagai katalis dalam pembuatan biodiesel," in *Prosiding Seminar Nasional Kimia*, 2017, pp. 143–147.
- [19]N. Nurjannah, "Preparasi katalis dari cangkang telur dengan metode impregnasi untuk pembuatan biodiesel dari minyak kelapa," *JCPE*, vol. 5, no. 1, pp. 41–44, 2020, doi: 10.33536/jcpe.v5i1.543.
- [20]Y. H. Taufiq-Yap, N. F. Abdullah, and M. Basri, "Biodiesel production via transesterification of palm oil using NaOH/Al2O³ catalysts," *Sains Malays*, vol. 40, no. 6, pp. 587–594, Jun 2011.
- [21]U. C. Abubakar, K. R. Alhooshani, S. Adamu, J. Al Thagfi, and T. A. Saleh, "The effect of calcination temperature on the activity of hydrodesulfurization catalysts supported on mesoporous activated carbon," *J Clean Prod*, vol. 211, pp. 1567–1575, Feb 2019, doi: 10.1016/j.jclepro.2018.11.177.
- [22]N. S. Lani, N. Ngadi, N. Y. Yahya, and R. Abd Rahman, "Synthesis, characterization and performance of silica impregnated calcium oxide as heterogeneous catalyst in biodiesel production," *J Clean Prod*, vol. 146, pp. 116–124, Mar 2017, doi: 10.1016/j.jclepro.2016.06.058.
- [23]A. Ameri, G. Sodeifian, and S. A. Sajadian, "Lansoprazole loading of polymers by supercritical carbon dioxide impregnation: Impacts of process parameters," *J Supercrit Fluids*, vol. 164, p. 104892, Oct 2020, doi: 10.1016/j.supflu.2020.104892.
- [24]A. Bouledjouidja, Y. Masmoudi, M. Sergent, V. Trivedi, A. Meniai, and E. Badens, "Drug loading of foldable commercial intraocular lenses using supercritical impregnation," *Int J Pharm*, vol. 500, no. 1, pp. 85–99, Mar 2016, doi: 10.1016/j.ijpharm.2016.01.016.
- [25]J. Li et al., "Synthesis and adsorption performance of La@ ZIF-8 composite metal–organic frameworks," *RSC Adv*, vol. 10, no. 6, pp. 3380–3390, 2020.
- [26]N. Fitriana, H. Husin, D. Yanti, K. Pontas, P. N. Alam, and M. Ridho, "Synthesis of K2O/Zeolite catalysts by KOH impregnation for biodiesel production from waste frying oil," in *IOP Conference Series: Materials Science and Engineering*, IOP Publishing, 2018, p. 012011.
- [27]L. Fan, D. Cheng, F. Chen, and X. Zhan, "Preparation of highly dispersed iron species over ZSM-5 with enhanced metal-support interaction through freeze-drying impregnation," *Chinese Journal of Catalysis*, vol. 40, no. 7, pp. 1109–1115, Jul 2019, doi: https://doi.org/10.1016/S1872-2067(18)63198-5.
- [28]J. H. Lee, M. J. Kim, E. J. Lee, D.-W. Lee, C. H. Kim, and K.-Y. Lee, "Promoting effect of Rhimpregnation on Ag/CeO2 catalyst for soot oxidation," *Appl Surf Sci*, vol. 572, p. 151504, Jan 2022, doi: 10.1016/j.apsusc.2021.151504.
- [29]L. Muniandy, F. Adam, A. R. Mohamed, and E.-P. Ng, "The synthesis and characterization of high purity mixed microporous/mesoporous activated carbon from rice husk using chemical activation with NaOH and KOH," *Microporous and Mesoporous Materials*, vol. 197, pp. 316–323, Oct 2014, doi: 10.1016/j.micromeso.2014.06.020.
- [30]L. Fu and W. Xia, "MAX phases as nanolaminate materials: chemical composition, microstructure, synthesis, properties, and applications," *Adv Eng Mater*, vol. 23, Jan 2021, doi: 10.1002/adem.202001191.
- [31]Y. Zhai, X. Ren, J. Yan, and S. (Frank) Liu, "High density and unit activity integrated in amorphous catalysts for electrochemical water splitting," *Small Struct*, vol. 2, no. 4, p. 2000096, Apr 2021, doi: https://doi.org/10.1002/sstr.202000096.
- [32]H. Takagi, K. Maruyama, N. Yoshizawa, Y. Yamada, and Y. Sato, "XRD analysis of carbon stacking structure in coal during heat treatment," *Fuel*, vol. 83, no. 17–18, pp. 2427–2433, Dec 2004, doi: 10.1016/j.fuel.2004.06.019.

[33]A. Munyentwali, H. Li, and Q. Yang, "Review of advances in bifunctional solid acid/base catalysts for sustainable biodiesel production," *Appl Catal A Gen*, vol. 633, p. 118525, 2022.