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THE PRODUCTION OF COBALT METAL-IMPREGNATED-ACTIVATED CARBON FROM BAMBOO AS A CATALYST FOR WASTE COOKING OIL (WCO) PYROLYSIS

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ABSTRACT

Waste cooking oil (WCO) pyrolysis conversion process requires a catalyst to produce high-activity and selectivity products. This research aims to analyze the effect of metal-impregnated activated carbon catalysts in the pyrolysis of WCO. In this study, activated carbon from bamboo was impregnated with Co using carbonization at 450°C for 5 hours, physical activation using steam at 800°C for 75 minutes, and ultrasonic radiation for 60 minutes to form a catalyst. The products obtained were then characterized according to SNI 1683:2021 and 06-3730-1995. Several instrumental analyses, including FTIR, BET, and FESEM-EDS, were also utilized. The catalyst was used to crack waste cooking oil through fast pyrolysis to determine the contents produced. The results showed that the bamboo-activated carbon produced met Indonesia's standard. The samples contained several functional groups, including C=O and C=C. The FESEM results show a more developed and open pore morphology when the carbon is treated with steam activation, with an increase in carbon content of up to 91.5%, and the Co metal has been successfully impregnated into the activated carbon of about 2.7%. BET results showed that carbon type I isotherm. In contrast, activated carbon and Co-impregnated catalysts showed a type III isotherm pattern with a micro-mesopore size distribution and a surface area of Co-KABT 3% and Co-KABT 5% catalysts of 368,085 m^2/g and 388,853 m^2/g, respectively. The catalyst's selectivity relatively produced alcohol, hydrocarbons, fatty acids, including aldehydes and ketones. The highest yields of hydrocarbons and alcohol were achieved using a Co-KABT 3% catalyst in the cracking results. The catalyst has demonstrated significant results, increasing the production of alcohol and hydrocarbons by almost 400% and 30%, respectively.

Keywords: Bamboo, Fast Pyrolysis, Hydrocarbon, Waste Cooking Oil, SDGs: Affordable and Clean Energy, and Climate Action.

INTRODUCTION

The fossil fuels contribute significantly to global warming, making it necessary to find alternative energy sources derived from natural materials that are more environmentally friendly. The energy conversion process requires a catalyst to increase production yields. Catalytic cracking is a way to break down complex hydrocarbons into simpler molecules using a catalyst, improving the product's quality and quantity. It can also reduce the amount of residue produced. Heterogeneous catalysts are preferred because they can be

separated from the reactants and formed from materials such as activated carbon from lignocellulose. Activated carbon catalysts from lignocellulosic biomass attract attention because they are considered cheaper and easily renewable with a high surface area, pore structure, inertness, and thermal stability.[1] Bamboo is a renewable lignocellulosic biomass with a branchless and hollow structure. It is known for its rapid growth and excellent regeneration capacity.[2] To obtain bamboo, the right harvesting technique is needed, which is cutting ripe bamboo that is ready to harvest. The type of bamboo, the purpose of use, and the season affect the best time for harvesting bamboo. Additionally, bamboo is an effective conservation plant, capable of absorbing significant amounts of carbon dioxide. It offers multiple benefits, serving as a material for construction and crafts and as a raw material for advanced applications like activated carbon. *Tali* bamboo, known as *apus* bamboo, has lignin, alpha cellulose, and hemicellulose up to 22%, 53%, and 23%, respectively. A high fixed carbon in bamboo up to 48.64%, while its nitrogen, sulfur, and hydrogen contents are 0.14, 0.11, and 6.75%, respectively.[3] This composition makes bamboo suitable for activated carbon material.[4,5] Activated carbon materials are gaining attention due to their significant potential for development. In Indonesia, the import value of activated carbon has been increasing annually, reaching 36 million USD in 2024.[6] Traditionally, commercial activated carbon is manufactured by a non-renewable source like coal and coke, which have a high production cost. As a result, there is a growing preference for using biomass precursors to reduce these costs.[7] Activated carbon can be developed into a catalyst matrix, a material for providing active sites for reactants and metals. This matrix offers good mechanical strength and thermal stability, facilitating the dispersion of metals and enhancing acidity. Particularly after impregnation with nickel (Ni) and Cobalt (Co), the application of this catalyst can achieve hydrogen yields of up to 93.8% through steam reforming.[8] Due to their vigorous catalytic activity, metals such as Ni, Co, iron (Fe), and molybdenum (Mo) are commonly used as catalysts. However, these metals have several drawbacks, including a tendency to agglomerate and deactivate, a short lifespan, and high cost.[9] Modifications are necessary to develop cheaper, environmentally friendly catalyst materials that are easy to regenerate and exhibit strong catalytic performance to address these issues. One approach to modification involves depositing the metal onto a solid carrier or matrix, such as activated carbon. This method ensures the metal is evenly dispersed within the carrier material, producing an activated carbon-based solid catalyst as a heterogeneous catalyst. This heterogeneous catalyst is expected to have high catalytic activity with low corrosion.[10] Recent research on metal impregnation in activated carbon as a solid catalyst has explored the uses of bamboo-activated carbon as a metal matrix for rhodium (Rh) and nickel (Ni). This metal-impregnated carbon catalyst effectively produced hydrogen through the dehydrogenation reaction of ammonia boron.[11] A synthesized metal-impregnated carbon catalyst demonstrated an impressive tetracycline degradation efficiency of 94.5%. Additionally, carbon proposes an organic pollutant degradation when combined with cobalt metal.[12] Yiyun Zhang[13] synthesized Ni-Co/C catalysts applied to PC plastic waste's hydropyrolysis and hydrodeoxygenation (HDO) reactions to generate jet fuel fractions. A catalyst based on an H_3PO_4-impregnated activated carbon-based catalyst was also successfully synthesized and produced 86.11% aromatics and 64.01% selectivity towards mono-aromatic products from wood-plastic composite reactants.[14] Furthermore, Ni and Fe/bamboo carbon catalysts were developed for lignin degradation using microwave heating, resulting in up to 80.4% bio-oil yields.[15] Bamboo biochar catalysts are also considered adequate in bamboo pyrolysis reactions to produce up to 82% phenol products and show good stability for adsorbents, catalysts, and reactants simultaneously during the catalytic pyrolysis process.[16] The activated carbon catalysts impregnated with 1% Co metal can also produce oil yields of 32.65% with the lowest activation energy.[17] Co-metal can serve as a catalyst in the catalytic cracking of waste cooking oil with various solution concentrations. The average conversion percentages (%) of waste cooking oil cracking at 1%, 2%, and 3% catalyst concentrations are 31.83%, 8.51%, and 11.43%, respectively. The optimal cracking product was obtained at 450°C with a Co-carbon concentration of 1%.[18] In this research, activated carbon-based catalysts were produced by carbonization and steam activation. These products were then impregnated with Co metal using the ultrasonication method to obtain Co/activated carbon catalyst solids, which were then applied to the pyrolysis of WCO. WCO poses environmental pollution risks and health concerns when reused repeatedly for food function; therefore recently it used for other functions, as a reactant for energy production due to its abundant availability. Utilizing WCO is a promising alternative for generating renewable energy with catalysts. Based on this

context, this research was conducted to synthesize Co/active carbon catalysts derived from bamboo and assess their effectiveness in cracking WCO to produce more valuable products. This research is in line with SDG number 7 (affordable and clean energy) because the research supports the SDG 7 goal, namely, access to affordable, reliable, and sustainable energy. The novelty of this research lies in exploring the properties of the Co/bamboo activated carbon catalyst, created through the combined processes of carbonization, activation, and Co impregnation using sonication. Additionally, the fast pyrolysis uses the (py-GCMS) model to analyze the composition of the resulting cracking products.

EXPERIMENTAL

Materials: bamboo (*Gigantochloa Apus*) from Bogor-Indonesia. WCO derived from fast food chicken business, 0.1% iod solution (Merck), $Co(NO_3)_26H_2O$ (Merck), methylene blue (Merck), distilled water, benzene (Smart lab), 0.1 N sodium thiosulfate (Merck), and starch indicator.

Equipment

Equipment includes beakers, glassware, Erlenmeyer flasks, burettes, pipettes, spatulas, an Ohaus analytical balance, a stirrer, aluminum foil, a desiccator, an oven, 1 set of raw material carbonization tools, and 1 set of carbon activation tools. Analytical instruments included a Shimadzu UV-Vis spectrophotometer UV-1700, a FESEM EDS (Bruker), and SAA Meso 112.

Methods

Carbonization and Activation of Bamboo

The bamboo was cleaned and cut into 3-5cm pieces. The bamboo pieces were aerated for 7 days and analyzed for moisture content. The bamboo carbonization process is carried out at 450°C, and then pulverized and filtered with a size of 40 mesh for activation. Activation of carbon was carried out by a physical method using steam at 800°C for 75 minutes to obtain activated carbon as a catalyst matrix precursor.

Co-metal Impregnation

20 grams of activated carbon were dissolved in 250 mL of distilled water, and 3% and 5% $Co(NO_3)_26H_2O$ solutions were added. The mixture was stirred with a magnetic stirrer at 350 rpm for 30 seconds and ultrasonically irradiated for 60 minutes at 20 kHz. The mixture was dried at 105°C and calcined at 550°C in a furnace. The solids were denoted as Co-KABT 3% and Co-KABT 5% and then applied as WCO cracking catalysts.

Catalyst Characterization

The quality of activated carbon was assessed through characterization according to the Indonesian National Standard (SNI No.06-3730-1995) method, which includes moisture, volatile matter, fixed carbon, ash, iodine number, benzene, and methylene blue adsorption. Then, the catalyst's acidity was also analyzed using the ammonia and pyridine base adsorption method. Other characterizations included functional groups using FTIR, morphology, and activated carbon content using FESEM-EDS, and porosity using BET.

Application of Activated Carbon Catalyst in Cracking Used Cooking Oil

Waste cooking oil preparation. WCO is added to the water and then heated until the water remains partially. Then, the WCO was given activated carbon heated at 70°C and stirred until the temperature reached 100°C. Then, the oil is filtered using Whatman 110mm, and the filtering results are ready to be used (Taufik, 2007; Muallifah, 2009; Novtriani, 2013). The oil was weighed with a ratio of 1:5 with Co-KABT 3% and Co-KABT 5% catalyst. The application was carried out on a py-GCMS device at 500°C to obtain chromatogram data on the relative compounds formed from the pyrolysis of used WCO using a Co metal-impregnated bamboo activated carbon catalyst.

RESULTS AND DISCUSSION

The production process of activated carbon begins with carbonization, where bamboo as a raw material is heated at 450°C using a carbonization instrument with minimal oxygen presence. This process yields carbon in a solid form and liquid smoke, which is a byproduct of gas condensation. The carbon product is further activated with steam, which opens the carbon pores and enhances the surface area, resulting in activated

carbon suitable for use as a metal matrix. Different methods of producing activated carbon from bamboo can lead to variations in physicochemical characteristics, including product yield. The yield is calculated as the ratio of the carbon of dry weight and the activated carbon products' initial weight of the raw materials used. In this calculation, W1 represents the dry weight of the produced carbon (in grams), while W0 denotes the initial weight of the bamboo raw material (in grams). This relationship can be expressed using eqn.-1.

$$Yield\ (100\%) = \frac{W1}{W0} \text{ x } 100\% \qquad (1)$$

In this study, the carbon product yield was 29.087%, with liquid smoke by-products of 41.4%. Furthermore, about 300 grams of carbon were activated using steam to produce 230 grams of activated carbon or 76.6% of the dry weight of carbon. Raw materials, temperature, and activator affect the yield of activated carbon.[19] Higher carbon content generally leads to more excellent activated carbon production, while higher temperatures release more molecules, producing a lower amount of activated carbon product. The respective analysis was carried out by SNI 1683: 2021 and SNI 06-3730-1995 to determine the quality of the carbon and activated carbon products against a national standard.

Table-1: Proximate Analysis of Carbon and Activated Carbon Bamboo

No	Testing parameter	KBT	SNI (1683:2021)	KABT	SNI (06-3730-1995)
1.	Moisture(%)	4.145	≤ 8	7.398	Max 15
2.	Ash (%)	3.493	≤ 4	4.145	Max 10
3.	Volatile matter(%)	29.535	10 –17	21.463	Max 25
4.	Fixed carbon (%)	66.972	≥ 79	74.387	Min 65
5.	Iodine number ($^{mg}/_{g}$)	435.894	-	801.116	Min 750
6.	Methylene blue adsorption($^{mL}/_{g}$)	93.442	-	101.912	Min 120
7.	Benzene adsorption (%)	6	-	20	-
8.	Pyridine adsorption($^{mmol}/_{g}$)	10.86	-	12.78	-
9.	Ammonia adsorption($^{mmol}/_{g}$)	7.65	-	11.17	-

Table-1 shows that the quality of carbon does not entirely follow SNI requirements, especially in the value of volatile matter substances. Hence, this also affects the bound carbon content of carbon samples, which is below standard. In the carbonization process, bamboo raw material molecules change or break down into several products: carbon as a solid product, liquid smoke due to condensation, and gas or other by-products. In this process, the quality of carbon, especially the sorption capacity, tends to be low because there is still much residue covering the pore surface. Carbon is then activated using steam to improve the quality characteristics of activated carbon by SNI standards, and the carbon activation process improves the value of the SNI parameter test results. The activation process can increase the surface area and absorption capacity of carbon.

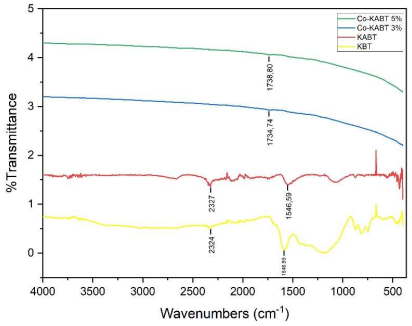

Fig.-1: FTIR Spectra of Activated Carbon Impregnated with 3% and 5% Co Metal

The data showed that the chemical structure was decomposed during the carbonization process. The heat treatment of carbonization resulted in degradation and changes in functional groups. Chemical compounds are degraded, mainly cellulose and lignin compounds. In bamboo carbon without activation treatment, there are several wave numbers, including 2663, 2323, 1683, 1176, 872, 811; 750, and 458 cm^{-1}, while in bamboo

activated carbon after steam treatment only gives rise to wave numbers 2323; 2111, 1548; 1060; 458; 454; 407 cm^{-1} where the wave number of 2663, 2323, 2162 cm^{-1} are representations of the triple C functional group.[20] The wavelengths of 1683 and 1734 cm^{-1} represent the C = O functional group, a stretch vibration of carboxylic acids, ketones, or lactones.[21] Ahmad et al. and Rattanapan,[20] found the C = O group at wave numbers 1640-1700 cm^{-1}. The wavelength of 1400-1583 cm^{-1} is the aromatic ring C=C functional group,[22] which in this study is found in the KABT sample shown at wave number 1548 cm^{-1}. The wave number 1060 cm^{-1} for C-O stretches from esters, ethers, or phenols. The low wavenumber region can reflect the absorption of C-H bonds from aromatic rings. The surface of activated carbon has many functional groups, especially hydroxyl groups and ether bonds, so these functional groups usually contribute as active sites of chemical adsorption.[19] Several factors, including activator, temperature, and impregnation treatment, influence the activated carbon functional groups' surface. After activation, some wave numbers can remain, disappear, shift, or decrease in intensity compared to those without activation.

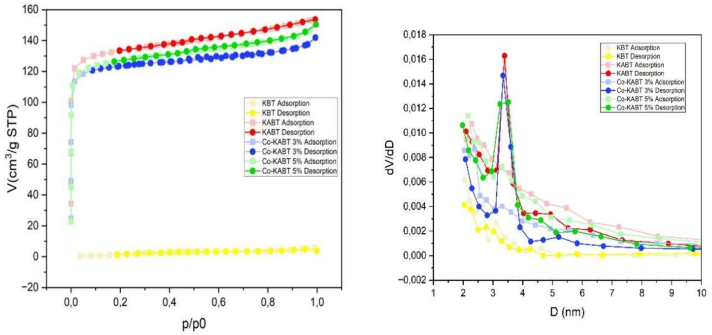

Fig.-2: Nitrogen Adsorption and Pore Diameters of Samples using the BET Method

Figure-2 illustrates nitrogen adsorption and desorption isotherm patterns on samples in this study. The isotherm curve pattern of carbon resembles a type III isotherm curve depicting a macroporous surface. Meanwhile, the activated carbon and Co-impregnated carbon show isotherm curves that resemble type I with very few hysteresis loops, so they are suspected to contain micro-meso-sized pores. It is known that carbon activation using physical methods, such as water vapor, mainly produces meso-sized pores.

Table-2: Pore Structure of Carbon, Activated, and Activated Carbon with 3 and 5% Co Metal Impregnation

Sample	Surface area(m^2/g)	Pore volume (cm^3/g)	Pore size (nm)
KBT	24.058	0.009	1.535
KABT	419.755	0.238	2.264
Co-KABT 3%	368.085	0.219	2.378
Co-KABT 5%	388.853	0.231	2.381

Table-2 shows the evidence that the surface area of activated carbon increased significantly compared to that of non-activated carbon. This increase can be attributed to the removal of impurities from carbon surfaces and pores and the formation of new pores. A higher surface area enhances the adsorption capacity of activated carbon, leading to greater absorption of adsorbate compounds such as iodine, methylene blue, pyridine, and ammonia. Activation using a steam activator is believed to expand the carbon surface and create meso-sized pores, in contrast to chemical activation. However, using the sonication method, the impregnation process of activated carbon with Co metal resulted in a decreased surface area. This reduction is probably due to the undispersed metal blocking the surface or pores of the carbon and forming aggregates, which ultimately reduce the calculated surface area. Materials with a high surface area are more conducive to bonding with substrates. Additionally, a high pore volume promotes product formation by significantly increasing contact opportunities between reactants and catalyst-active sites, enhancing product yield, and reducing reaction rates.[10]

The surface morphology and elemental analysis of carbon (KBT), activated carbon (KABT), and Cobalt-impregnated carbon (Co-KABT 3% and Co-KABT 5%) samples are presented in Fig.-3. Figure-3a demonstrates the surface morphology of bamboo carbon before the activation process, where the pore size

varies and has not evolved much, with a carbon content of 83 %. Following the activation procedure (Fig-3b), the carbon content increased to 91.5%, along with more developed pore apertures.

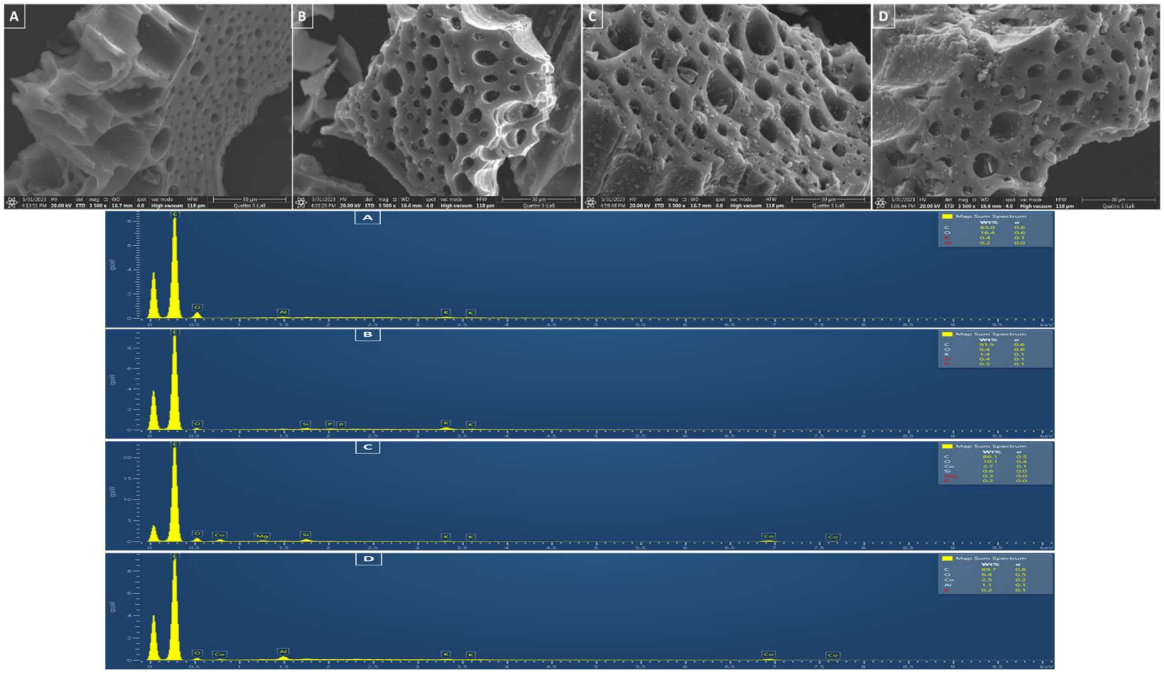

Fig.-3: FESEM-EDS Images for KBT (a), KABT (b), Co-KABT 3% (c), and Co-KABT 5% (d)

The activation procedure can eliminate contaminants from the carbon surface or pores, making the carbon pores more open.[17,23] Figure-3c and 3d show that the impregnated Cobalt metal attaches to the activated carbon's surface and pores, reducing the activated carbon's surface area. The optimum percentage of Cobalt impregnated into activated carbon is 2.7% (Co-KABT 3%). The more Cobalt attached to the active carbon side, the more significant the decrease in surface area, as evidenced by an increase in Cobalt and a decrease in carbon content in a sample.[24] Calculating the specific surface area using BET can also demonstrate the reduction in surface area on Cobalt-impregnated carbon.

Waste Cooking Oil Cracking using Activated Carbon Impregnated with Co-Metal

Activated carbon is a porous material with distinct physicochemical properties. The performance of activated carbon-based catalysts varies depending on several production parameters, including activation temperature, duration, and impregnation ratio.[25] The material is widely used in various applications, particularly in the catalysis process of biofuel production, which can be synthesized from vegetable or animal fats, including waste cooking oil. However, using vegetable oil directly can lead to issues such as inefficient heating and significant carbon deposits, primarily due to the high viscosity of the vegetable oil.[1] Waste cooking oil can be converted into renewable energy and other chemicals with higher yields than biomass feedstocks with cellulose content.[26] The content of palmitic, stearic, and other long-chain fatty acids has the energy potential as feedstocks. The cracking/pyrolysis technique can convert oils. The pyrolysis temperature was measured at 500°C, where fatty acids are generally decomposed at 320°C.[26] The high temperatures of pyrolysis break down the organic bonds in fatty acids due to the sufficient energy provided.

From the py-GCMS results, various chemical compounds were formed from the pyrolysis of used cooking oil, both using and without catalysts. These compounds are grouped into several types: alcohols, hydrocarbons, fatty acids, polycyclic aromatic hydrocarbons (pAh), and others (ketones, aldehydes). Figure-4 shows that the number of alcohol compounds increases with the addition of catalysts. Specifically, the percentage of alcohol reaches 11.8% with a Co-KABT 3% catalyst and 5.91% with a Co-KABT 5% catalyst. In contrast, without any catalysts, alcohol production is only 2.98%.

The pyrolysis process also produced hydrocarbon content, which increased from 25.32% to 35.2%, especially when treated with Co-KABT 3% catalyst. This result is corroborated by the EDS results that the highest Co metal content (2.7%) is found in the Co-KABT 3% catalyst, where the high Co content provides a better active site to break down more compounds. Hydrocarbons are chemical components in energy that are expected to form when pyrolysis occurs. These hydrocarbons can be aliphatic, aromatic, or alicyclic. The higher the hydrocarbon content, the quality of the energy product could be better.

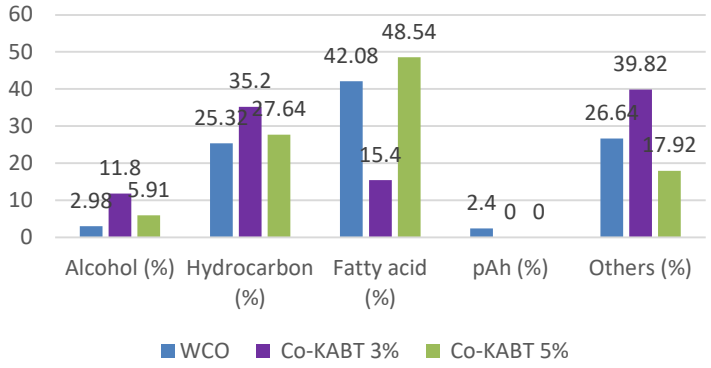

Fig.-4: Pyrolysis of WCO using Co Metal-Impregnated Activated Carbon Catalyst

The formation of aliphatic hydrocarbons is possible due to the deoxygenation of hydroxyl, carbonyl, and carboxyl compounds. Waste cooking oil also contains high fatty acids, incredibly saturated fatty acids such as palmitic and oleic acids, around 19.89% and 22.15%. Meanwhile, the samples of WCO pyrolysis results using catalysts showed the presence of unsaturated fatty acids such as stearic acid and heptadecane (8) carbonic acid of 3.47% and 5.51%, respectively, in the addition of Co-KABT 3% catalyst and 18.4% and 11.5% in the addition of Co-KABT 5% catalyst. This data follows Ibrahim *et al.,*[27] who obtained carboxylic acid compounds of 24% in the cracking of WCO using catalysts. The WCO pyrolysis without using catalysts resulted in pAh at 2.4%, where these compounds are carcinogenic and undesirable in a product. The presence of a catalyst, especially with a high surface area, allows for a longer residence time of the reactants and initiates subsequent thermal cracking with low molecular weight.[26] Pyrolysis products with high hydrocarbon content have the potential to be developed as energy feedstock; therefore, this result can contribute to the SDGs goal number 7, in order to get affordable and clean energy for society.

CONCLUSION

The utilization of bamboo biomass as raw materials to create a metal catalyst matrix that supports Cobalt (Co) is crucial for enhancing the pyrolysis of waste cooking oil (WCO). This approach adds value to abundant biomass resources and emphasizes the importance of managing natural resources through technological advancements. A key aspect of this research is the application of green chemistry to produce renewable products, such as biocatalysts and bioenergy, from biomass resources. The process of impregnating bamboo activated carbon with Co metal and its subsequent application in the cracking of WCO has demonstrated significant results, increasing the production of alcohol and hydrocarbons by almost 400% and 30%, respectively. Along with this result, this research is in line with SDG Goal 7 to get affordable and clean energy for society.

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CONFLICT OF INTERESTS

The author (s) declare no conflict of interest in this research and manuscript.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-7: Affordable and Clean Energy.* All the authors contributed significantly to this research and manuscript, participated in the idea, methodology, analysis, writing,

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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