

## Improved Biodiesel Production Yield Using Coconut Mesocarp-Based Catalyst

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### ABSTRACT

Current concern over the decrease in the use of fossil fuels has led to the study of various options as an alternative to replace them in the transportation and industrial sectors. Different materials, such as agricultural products, lignocellulosic residues, solid wastewater products, and algae, can be used in the production of biochar and, through a sulfonation process, it can be converted into a heterogeneous acid catalyst. The purpose of this study was employed coconut mesocarp as lignocellulosic biomass feedstock, obtaining sulfonated biochar (BACS), and evaluates its use in biodiesel production, comparing it with a KOH catalyst. The methodology included the pyrolysis of coconut mesocarp and the activation with H<sub>2</sub>SO<sub>4</sub> for BACS production. BACS was characterized by infrared spectroscopy, scanning electron microscopy, thermogravimetric analysis, BET surface area analysis and elemental analysis. The biodiesel obtained by BACS and biodiesel obtained by KOH were compared using international biodiesel standards. An activated sulfonated biochar with a sulfonation percentage of 15.23% was successfully obtained, providing a higher FAME conversion percentage than the KOH catalyst. During the characterization of the biodiesel obtained with both catalysts, it was found that KOH meets the specified standards, while the BACS catalyst requires variations in reaction temperature or blending with diesel to comply with the biodiesel characteristics. Additionally, it was observed that the coconut mesocarp-based catalyst showed a 2.78% reduction after the first working cycle, allowing for its reuse without the need for a new sulfonation process.

**Keywords:** biochar, biofuel, biomass, catalysis, waste.

### INTRODUCTION

The high energy demand currently faced by the global population, resulting from rapid technological development, high industrial demand, and an increase in the number of inhabitants worldwide, has led to a significant growth in the use of fossil fuels. Thus, it is projected that the demand for liquid fuels will increase by 32% between 2020 and 2050 to meet these needs (Xu et al., 2022). On the other hand, the great amount

of greenhouse gases released when using the energy source have turned it into a major cause of air pollution and climate change (Kumar & Raheman, 2022). However, during the last decades, efforts have been made to find alternative fuels that could be used as a replacement of fossil fuels, thus addressing the increasing demands while guaranteeing environmental sustainability (Ao & Rokhum, 2022).

Biodiesel has emerged as an alternative to replace petroleum-based fuels in the transportation

and industrial sectors due to its physical and chemical characteristics comparable to conventional diesel (Abdelmoez et al., 2016). Biodiesel fuels consist of mono-alkyl esters (fatty acid methyl esters, or FAME). They are obtained from the transesterification of fatty acids present in vegetable oils or animal fats through catalytic reactions with a short-chain alcohol (Pydimalla et al., 2023).

The catalytic transesterification process of fatty acids present in oils to FAME can occur by using biological agents such as enzymes or either homogeneous or heterogeneous catalysts (alkalis or acids) (Biernat et al., 2021). The choice will depend on the effectiveness of the process, that is, on the conversion rate and energy demand related to temperature, pressure and reaction time, as well as on the ease in the separation and purification process of biodiesel to obtain the final product (Thangaraj et al., 2019).

A homogenous catalyst exists in the same phase as the reactants and may be alkali or acid. In the first case, compounds such as NaOH, KOH, NaOMe, KOMe, etc. are dissolved in the short-chain alcohol (usually methanol) due to their affinity, solubility, and low cost (Kirubakaran & Arul Mozhi Selvan, 2018). However, one inconvenience is that secondary saponification reactions occur. Thus, to avoid this problem, they are frequently replaced with acid catalysts, such as p-toluenesulfonic acid or sulphuric acid. Due to its lowest cost, sulphuric acid is more commonly used (Marchetti et al., 2011; Singh et al., 2022). In general, the drawbacks of homogeneous catalysis include excess generation of waste waters, requiring large amounts of alcohol, deterioration of the reactors and pipelines, more expensive biodiesel purification, and ultimately, no catalyst recovery (Boro et al., 2014).

Heterogeneous catalysts are usually solid, which facilitates their recovery and reuse, resulting in higher conversion rates and reduced operational costs of biodiesel production (Jayakumar et al., 2021). Previously, inorganic oxides with acid or alkaline properties were used as catalysts (Boro et al., 2014). Currently, there is a growing search for obtaining catalysts from different residues through conversion to biochar (Ínan et al., 2023; Mendonça et al., 2019). This is a material generated by means of biomass thermal degradation at high temperatures ranging between 300 °C and 600 °C, with no or limited levels of oxygen, and a high surface area and porosity (Panwar et al., 2019). Different materials, such as agricultural products, lignocellulosic residues, solid waste water products, and algae,

can be used in the production of biochar. Biochar can be converted into a heterogeneous acid catalyst by forming acid sites where transesterification using  $H_2SO_4 > 95\%$  occurs. This process is known as sulfonation of biochar and has proven to have enormous potential in biodiesel production (Garg et al., 2023; Hosseinzadeh-Bandbafha et al., 2023; Ínan et al., 2023).

According to Monge (2022), Costa Rica is a Central American country characterized by its agroindustrial production and environmental responsibility. That is why, in 2016, it became the first country to sign a National Pact for the Sustainable Development Goals (SDG), thus, laying the foundations for a collective commitment to foster environmental sustainability at a national level, by promoting biofuel production and harnessing agroindustrial waste (Corrales Aguilera et al., 2022; MIDEPLAN, 2023)

Coconut is a non-conventional export product, abundant in Costa Rican coastal areas, especially in the Caribbean area. It is predicted that coconut exports will annually increase by 10% between 2020 and 2026 (PROCOMER, 2020). The coconuts are daily harvested and transported to processing facilities where the main products and by-products are obtained. However, the husk is usually taken to unauthorized dumping sites. This, together with production concentration and little use of technology generates large amounts of waste, which is not properly managed or disposed of, resulting in increased pollution (Espinoza Montero et al., 2022). That is why, this study aimed to assess the use coconut mesocarp as lignocellulosic biomass feedstock to convert it into sulfonated biochar and evaluate its use in biodiesel production comparing it with a KOH catalyst.

## MATERIALS AND METHODOLOGY

### Sample processing

Sample collection, processing, and pyrolysis was carried out at the Caribbean Regional Campus of Universidad de Costa Rica (UCR). The coconut mesocarp samples were obtained from the Batán area, Limón, Costa Rica. They were subjected to a pretreatment process. They were dried in a binder natural convection oven (standard model) at 60°C for 24 hours. Finally, the coconut mesocarp was grinded in a Fritsch Pulverisette 19 blade mill using 4-mm sieves.

## Pyrolysis of coconut mesocarp, obtention of activated biochar

The pyrolysis was carried out in a Carbolite HZS 1200 three-zone tube furnace. The system kept a continuous nitrogen gas flow rate of 5 liter/minute. A constant heating rate of 10 °C/min was used to reach the final temperature, set at 550 °C. The pyrolysis process took an hour (Diaz-Caleño, 2020).

## Sulfonation

The methodology proposed by Bastos et al. was followed. To this end, 10 mL of H<sub>2</sub>SO<sub>4</sub> were used for every 1 g of activated biochar obtained via pyrolysis. The sulfonation was carried out in a flat-bottom balloon flask under constant agitation at 200 °C for 4 hours. After the reaction, the black solid was thoroughly washed with distilled water to neutral pH (pH ~ 7) and dried at 80°C for 24 h. The previous procedures were carried out in the Unit of Forest Resources (RE-FORESTA, acronym in Spanish) lab of the Research Institute in Engineering (INII, acronym in Spanish) at UCR.

## Biochar characterization

### Infrared (IR) spectroscopy

A PerkinElmer Frontier FT-IR spectrometer with ATR was used to identify the chemical compounds through the frequency ranges of different functional groups present in activated and sulfonated biochar. 32 scans per sample were performed in the spectral range from 4000 cm<sup>-1</sup> to 500 cm<sup>-1</sup>. The spectroscopy was carried out in the Materials Engineering and Science Research Center (CICIMA, acronym in Spanish) at UCR.

### Scanning electron microscopy (SEM)

A HITACHI S3700-N SEM, belonging to the Research Center in Microscopic Structures (CI-EMic, acronym in Spanish) at UCR, was used to determine the morphological changes of biochar after the sulfonation process.

### Thermogravimetric analysis (TGA)

The analysis was performed in a TGA equipment from TA Instruments, model SDT-650, in the temperature range of 0 °C to 1000 °C at a heating rate of 15°C/min, under an inert atmosphere. The

analysis was carried out by the School of Chemical Engineering at UCR to determine the sulfonation percentage of activated biochar according to ISO 16948:2015 standard.

### BET surface area analysis

An Autosorb IQ gas sorption analyzer, from Quantachrome Instruments, was used. The high-vacuum physisorption/chemisorption analyzer, from the Research Center in Electrochemistry and Chemical Energy (CELEQ, acronym in Spanish) at UCR, was used to determine the surface area and pore size of an activated biochar sample of coconut mesocarp and an activated biochar sample of sulfonated coconut mesocarp.

### Elemental analysis CHONS

A Thermo Fisher Flashmart elemental analyzer with multivalve enabled to determine the elemental composition of the sample according to ISO 16948:2015 standard. The analysis was performed at the School of Chemical Engineering (UCR).

## Biodiesel production

The evaluation of biodiesel production was performed using two catalysts. The conditions and reaction parameters were based on Lathiya et al. (2018) and Lugo et al; (2015). A nested experimental design was used to compare biochar obtained from coconut mesocarp with potassium hydroxide, used as reference. To this end, high-speed homogenizer IKA T25 Ultra-Turrax was used. The agitation speed was maintained at 7000 rpm throughout the process, at a temperature of 60°C with a reaction time of one hour.

For the biodiesel synthesis using potassium hydroxide (KOH) as the catalyst, firstly, solutions of 1.0% and 0.5% KOH solid catalyst in methanol were prepared to homogenize the mixture. Then, soy oil was added in the reactor using a oil to methanol molar ratio of 6:1, and the reaction was allowed to proceed. Once the reaction was completed, the mixture was transferred into a separating funnel, and was left for 24 hours to settle. Lastly, the resulting glycerol was separated from biodiesel by liquid-liquid extraction.

For the synthesis of biodiesel using activated and sulfonated biochar as the catalyst, the oil to methanol molar ratio used was 30:1. Two different weight percentages of catalyst were

employed: 1.0% and 3.0%. After the reaction was completed, a centrifuge was used to separate the solid residues of activated and sulfonated biochar at 6500 rpm for 20 minutes.

#### FAME biodiesel conversion

The biodiesel conversion percentage was determined using the method described by Lugo et al. (2015). In this method, the biodiesel density is determined by pycnometry at  $15.0 \pm 0.1^\circ\text{C}$ . Afterwards, the kinematic viscosity was determined by using ARES-G2 rheometer, from Texas Instruments, at a temperature of  $40.0 \pm 0.1^\circ\text{C}$ , using 25 mm parallel plates at a shear rate of  $100 \cdot 1 \text{ s}^{-1}$ . This determination was carried out by the Materials Science and Engineering Research Center (CICIMA, acronym in Spanish) at UCR. Then, the dynamic viscosity was obtained from these data. The dynamic viscosity was used as a parameter of the transesterification reaction, since it is supposedly related to FAME content and the conversion rate.

$$FAME = (-0.2187 \ln \mu + 0.9466) \quad (1)$$

Likewise, a balanced hierarchical statistical design was performed using the type of biodiesel (Factor A), referring to either potassium hydroxide catalyst (B. KOH) or sulfonated biochar catalyst (B. BCAS), and the amount of catalyst used (Factor B).

#### Heat of combustion

An AC500 isoperibolic calorimeter was used to precisely determine the heat of combustion. This analysis was carried out by the Analytical Services Laboratory of the School of Chemistry at UCR.

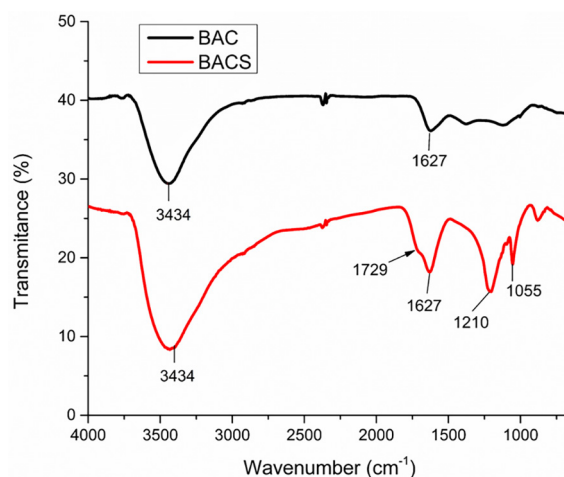
#### Sulfated ash

The sulfated ash content was determined by means of the calcination of the sulfated sample with 1 mol/L sulfuric acid. The Analytical Services Lab of the School of Chemistry (UCR) made the determination of sulfated ash content.

## RESULTS AND DISCUSSION

Figure 1 shows two infrared spectra. A spectrum for a biochar activated by pyrolysis (BAC) and a spectrum for a biochar activated by pyrolysis and sulfonation (BACS) can be observed. Sulfonation is the most common method for converting biochar obtained by pyrolysis into a catalyst for biodiesel production, resulting in higher biodiesel yields (Hosseinzadeh-Bandbafha et al., 2023). Table 1 shows a summary of typical bands for both materials. In both spectra, the typical bands of -OH and C=C bonds attributable to lignocellulosic compounds carbonized at low temperatures are present (Konwar et al., 2014). In the BACS (sulfonated biochar), the presence of the sulfonic group (-SO<sub>3</sub>H) is shown, which is not observed in the BAC (biochar). Also, the appearance of the -COOH group band attributable to the hydrolysis of some groups on the cross-linked surface of the biochar can also be observed, hence its low intensity (Ma'rifah et al., 2019). Thus, it is known that there is a degree of functionalization of the material, which is confirmed with elemental analysis and quantified in the thermogravimetric analysis.

From the elemental analysis of data in Table 2, it is shown that BACS contain sulphur (S) in their



**Figure 1.** Infrared spectrum for BAC and BACS samples

**Table 1.** Summary of typical bands for the identification for activated and sulfonated biochar (Lathiya et al., 2018)

Wavelength (cm <sup>-1</sup> )	Identification
≈ 3434	Stretching vibrations of -OH bonds of lignocellulose
≈ 1729	Generation of -COOH group
≈ 1627	Polyaromatic vibrations C=C from groups of lignin
≈ 1055-1210	Presence of -SO <sub>3</sub> H functional groups

**Table 2.** CHONS elemental analysis produced biochars

Sample	Mass percentage of elements (%)				
	C	H	O	N	S
BAC	74.6 ± 0.4	2.2 ± 0.1	8.5 ± 0.5	0.60 ± 0.01	< 25 mg/kg
BACS	64.4 ± 0.7	1.7 ± 0.4	24.2 ± 0.4	0.47 ± 0.06	1.5 ± 0.1

structure. At the same time, oxygen (O) increases and hydrogen (H) decreases when compared to BAC. This proves that  $-SO_3H$  groups were added to the structure, as shown in the FTIR in Figure 1 (Fraile et al., 2014). Sulphur content in the sample shows a similar amount to the one obtained by Malaika et al. (2021) for a sulfonated biochar. At the same time, there is also coincidence with the materials synthesized by Panwar et al. (2019), This proves that this is an efficient process for material sulfonation.

Through thermogravimetric analysis, three distinct mass losses were observed, as shown in Table 3. The first mass loss is consistent with the unbound moisture in the sample, and therefore occurs between 30 °C and 100 °C. The second mass loss is consistent with water occluded within the material's structure, hence its temperature limit is 130 °C. These temperature intervals align with findings from studies conducted by Espinoza-Montero et al., (2022); Jirón-García & Rodríguez-Mora, (2022) on materials synthesized from agro-industrial residues. Additionally, it is observed that the sulfonated biochar (BACS) does not contain occluded water, as it underwent two thermal treatments: pyrolysis at 550 °C and sulfonation at 200 °C.

The third mass loss occurs within a temperature range from 200 °C to 700 °C, and its broad amplitude is due to the differential degradation of compounds present in the coconut mesocarp, such as hemicellulose, cellulose, and lignin, which have pyrolysis temperatures of 281 °C, 339 °C, and 356 °C, respectively. Polysaccharides decompose through dehydrogenation and cleavage of glycosidic bonds, while lignin generates a variety of volatile alcohols, carboxylic acids, aldehydes, and phenols. Additionally, within this range, the degradation of the  $-SO_3H$  group is observed, as the highly cross-linked aromatic residue surface derived from lignin readily undergoes functionalization with acidic catalytic groups (Meng et al., 2021). Thus, the sulfonation percentage in the sulfonated biochar (BACS) is determined to be  $15.23 \pm 0.43\%$ , which aligns with expectations considering the initial high lignin content, estimated to be around  $51.8 \pm 2.9\%$  (Espinoza-Montero et al., 2022). Following pyrolysis, the degradation produces large molecules together with a surface with numerous active sites and a high surface area.

Table 4 shows a comparison of a variety of catalysts. It includes data related to the process

**Table 3.** Decomposition of BAC and BACS samples by TGA, in percentages

Range °C	Identity	Percentage of BAC mass loss (%)	Percentage of BACS mass loss (%)
30–100	Humidity	4.21±0.09	11.00±0.07
100–130	Occluded water	1.61±0.21	-
200–700	-	20.48±0.42	35.71±0.09

**Table 4.** Comparison of the sulfonation percentage and biodiesel production yield from sulfonated biochar

Biomass	Acid group mmol/g	Sulfonation %	Yield %	Reference
Coffee residue	0.45	3.65%	71.5	(Ngaosuwan et al., 2016)
Coconut shell	1.0	8.11%	86.03	(Zhong et al., 2019)
Orange	1.5	12.16%	91.68	(Lathiya et al., 2018)
Coconut mesocarp	1.9	15.23%	98.5	This study
Oiled canola meal	3.29	26.65%	93.8	(Rao et al., 2011)
Peanut shell	6.85	55.53%	90.2	(Zeng et al., 2014)
Oat hull	7.00	56.75%	89.97	(González et al., 2017)

feedstock, the sulfonation percentage, and the conversion yield for the biodiesel reaction. It is important to remark that, according to provided data, there is a slight trend between the sulfonation percentage and the reaction yield. This occurs because the increase of the number of active sites favors the transesterification reaction.

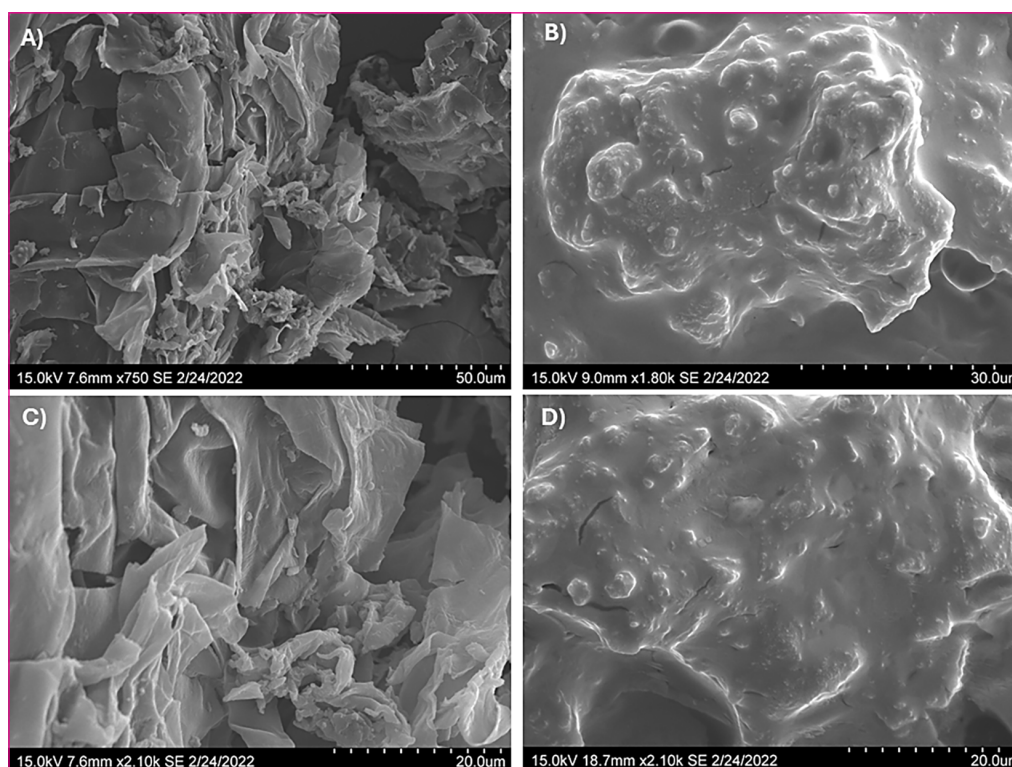
Figure 2 shows scanning electron microscopy images illustrating a change in the morphology of BAC compared to BACS. In these micrographs, a significant decrease in porosity in the biochar is observed. In Figure 2a, cavities or pores are visible in the structure of the activated biochar, while in Figure 2b, this morphology is no longer evident. This confirms that the sulfonation process utilized the active sites of the biochar to be occupied by  $\text{SO}_3\text{H}$  groups, thus allowing for increased catalyst contact, and consequently, an increased active site/surface area ratio in the transesterification reaction.

This is also reported by Bora et al. (2018) and Endut et al. (2017). In their studies, coconut waste-based carbon catalysts were used for biodiesel production. These studies showed a significant number of micropores after thermal activation. However, after sulfonation, a blockage of those pores and a smoother surface were observed, indicating the adsorption of catalytic

$\text{-SO}_3\text{H}$  groups onto the carbon support, which increases the active sites of the synthesized BACS. This reduction in pore size is also evident in the surface analysis, as presented in Table 5.

The surface properties shown in Table 5 reveal that BAC has considerable surface area and pore volume for the adsorption of  $\text{-SO}_3\text{H}$  groups. In this case, the average pore diameter is larger, although the contact surface area is smaller compared to BACS. The increase in surface area and reduction in pore size in BACS can be attributed to the chemical activation process with  $\text{H}_2\text{SO}_4$ . This increases the active sites of the catalyst and enhances the transesterification reaction (Lathiya et al., 2018).

According to Li et al. (2008) and Fan et al. (2022) larger surface areas are obtained at higher temperatures for activated biochar. The study de Igboke et al. (2023) reported a value of  $33 \text{ m}^2 \cdot \text{g}^{-1}$  when pyrolysis is conducted at  $500 \text{ }^\circ\text{C}$ . This result is consistent with the area found in BAC. However, the synthesized BACS has a higher surface area than reported in these studies. This is due to the type of activation applied to the biochar prior to sulfonation since thermal treatments improve properties, such as surface area, porosity, and crystallinity compared to those without such activation (Ma'rifah et al., 2019; Yu et al.,



**Figure 2.** Morphological analysis by SEM (a) y (c) activated biochar by pyrolysis (b) y (d) activated biochar by sulfonation

**Table 5.** Specific surface area, total pore size, and average pore diameter of biochar samples

Type of sample	Specific surface area / m <sup>2</sup> g <sup>-1</sup>	Total pore size / cm <sup>3</sup> g <sup>-1</sup>	Average pore diameter / nm
Activated biochar (BAC)	30.516	0.03681	4.825
Sulfonated biochar (BACS)	68.714	0.08022	4.67

2016). Table 5 shows the obtained data on viscosity, density, and FAME conversion percentage of biodiesel produced using activated and sulfonated biochar (B.BACS) and biodiesel produced using KOH (B.KOH). Viscosity and density are relevant properties to be considered when comparing obtained products with commercial diesel, as they directly influence the performance of compression ignition engines or diesel engines' fuel injection systems (Benjumea et al., 2006).

In this study, it is observed that B.BACS showed relatively low viscosities compared to the viscosity range of commercial biodiesel and biodiesel reported by other authors, whereas B.KOH produced viscosity values similar to those established for commercial diesel. This difference is primarily due to the difference in the chemical nature of the catalysts, as one is acidic and the other is alkaline, which directly affects the amount of free fatty acids and unsaturated lipids in the oil (Maleki et al., 2022; Zheng et al., 2020).

In both cases, the reported values are below the limit established by the Central American Technical Regulation (upper limit of kinematic viscosity of 6.5 mm<sup>2</sup>/s) (ASTM, n.d.). However, B.KOH falls within the range defined by ASTM 6751-08 standard, which sets the kinematic viscosity range for biodiesel between 1.9–6.0 mm<sup>2</sup>/s. This results from the conversion percentage of the reaction, which affects the density and viscosity of biodiesel, as the unreacted methanol lowers the average of these properties. It is possible to observe that B.BACS tends to generate biodiesel with low density and viscosity because increasing the catalyst percentage in the reaction decreases these properties, but increases the conversion percentage. On the other hand, KOH generates a product with high density and viscosity, not only compared to B.BACS, but also to what have been reported in other studies, such as Lugo et al (2014), who reported a density of 830.2 kg/m<sup>3</sup> and a viscosity of 2.400 ± 0.003 mm<sup>2</sup>/s.

At the same time, the achieved FAME conversion percentage is higher than that reported by other authors for sulfonated biochar. For example, Bora et al. (2018) reported a maximum

conversion of 95.57%. In the mean time. Lathiya et al. (2018) achieved a conversion percentage of 91.58%. Therefore, the results obtained with B.BACS, produced with a mass percentage of 1.0% and 3.0% of the catalyst, are higher than those reported by these authors.

When comparing B.KOH, the study by Marcano et al. (2014) reported conversion percentages lower than 50%, while Rashid et al. (2014) obtained a FAME value of approximately 60%. The results obtained in this research, although higher, are similar to those reported in previous studies, thus demonstrating the effectiveness of the process.

Table 7 shows that there are statistical differences in one of the factors. For Factor A (biodiesel), it is observed that P-value < 0.05, rejecting the null hypothesis, and thus, the factor has an influence, demonstrating that the yield is indeed affected depending on the catalyst used. For the analysis of Factor B (catalyst percentage), it is observed that P-value > 0.05, accepting the null hypothesis, indicating a null effect of the factor. Therefore, the studied percentages of each catalyst affect the reaction yield. This suggests that, in terms of cost, it is recommended to use the catalysts in their lower percentages.

Considering the statistical design and the data in Table 4 and Table 6, higher FAME conversion yield is demonstrated for BACS compared to other sulfonated biochar catalysts. This difference in yield performance is observed even when compared to other feedstock with a higher sulfonation percentage, such as oat hull (González et al., 2017). This proves that obtaining a BACS with a high surface area positively influences B.BACS. Likewise, it is worth noting that coconut mesocarp is a material that is abundantly generated. Currently, this feedstock has no use, and is disposed of in open-air landfills. Additionally, it does not require costly pretreatment to obtain biochar. The characterization of the obtained biodiesel (B100), using the two catalysts under study, was performed to quantify the most important properties to consider when comparing the obtained products with commercial diesel. These values are displayed in Table 8

**Table 6.** Values obtained for kinematic and dynamic viscosity, density, and FAME of synthesized biodiesels from BACS and KOH\* catalysts

Sample	Mass percentage of catalyst	Viscosity dynamic (cP)	Viscosity kinematic (mm <sup>2</sup> /s)	Density 15°C (kg/m <sup>3</sup> )	FAME conversion percentage (% mol/L)
B. BACS 1	1.0%	0.92 ± 0.11	0.75 ± 0.11	818 ± 20	96.6 ± 2.5
B. BACS 1	3.0%	0.84 ± 0.04	0.68 ± 0.04	805.7 ± 1.5	98.5 ± 1.1
B.KOH 1	0.5%	4.28 ± 0.24	3.82 ± 0.22	892.5 ± 1.5	62.8 ± 1.2
B.KOH 2	1.0%	3.88 ± 0.01	3.46 ± 0.01	890.85 ± 0.49	65.01 ± 0.07

**Note:** \*B.BACS refers to biodiesel synthesized from activated and sulfonated biochar from coconut mesocarp, while B.KOH is an abbreviation used for biodiesel synthesized from potassium oxide as catalyst.

Considering the statistical analysis and the conversion percentages from Table 6, B.BACS 2 was selected for characterization and comparison with the commercial diesel. B.BACS 2 is biodiesel synthesized using sulfonated biochar as the catalyst, with a molar ratio of 30:1, catalyst percentage of 3%, and a maximum conversion of 98.5% in accordance with Central American Technical Regulation (Reglamento Técnico Centroamericano RTCA 75.02.43:07, 2007).

For sulfated ashes, the quantification limit of the equipment employed is 0.31% m/m, which is below the allowed limit (2% m/m for sulfated ashes.) Also, the reported kinematic viscosity does not exceed the allowed upper limit of 6.5 mm<sup>2</sup>/s, although it is below the minimum limit (1.9 mm<sup>2</sup>/s). Density and viscosity are temperature-dependent, and since the kinematic viscosity does not comply with existing regulations, it is recommended to vary the reaction temperature and blend with diesel (Ramírez Verdusco, 2013). For biodiesel synthesized using the commercial catalyst, B.KOH 1 was selected. This is biodiesel synthesized using potassium hydroxide as the catalyst, with a molar ratio of 6:1, catalyst percentage of 1%, and a maximum conversion of 65.01%, which is in accordance with current technical regulation. For sulfated ashes, the quantification limit of the equipment employed is 0.31% m/m, which is below the allowed limit (2% m/m for sulfated ashes). Thus, this indicates that the biodiesel complies with existing technical regulations.

Regarding density, it was reported at 15°C in this study. Both B.KOH and B.BACS fall within the ranges reported by Hoekman et al. (2012); Kirubakaran & Arul Mozhi Selvan (2018); and Yusuff et al. (2022). Also, although heat of combustion is not a required physicochemical characteristic to report biodiesel (B100), the values obtained in both cases are lower than the value for petroleum diesel (47.0 ± 0.2 kJ g<sup>-1</sup>). In this case, the value obtained for B.KOH 1

is closer to the values reported for biodiesel by other authors such as Akers et al. (2006) (41.2 ± 0.2 kJ g<sup>-1</sup>). Similarly, although the value shown for B.BACS 2 is lower, the value found for B.BACS is close to those reported by other authors such as Falbo et al. (2022) and Mehta & Anand (2009, which are 35 and 38 kJ g<sup>-1</sup> respectively. This demonstrates that BACS is a viable catalyst to obtain biodiesel with suitable physico-chemical properties.

Besides, it was demonstrated that, after a transesterification cycle, the percentages of occluded water decreased, while the moisture content in BACS increased. The occluded water content decreased from 11.00 ± 0.07% to 3.74 ± 0.13%, while the moisture content increased from 0% to 0.66 ± 0.08%, possibly due to diffusion during the aqueous reaction. It was also confirmed that sulfonation decreased from 15.23 ± 0.43% to 12.45 ± 0.57% after this cycle. This reflects the potential of BACS for further studies to determine the variability of the conversion percentage based on the catalyst's functionalization percentage and to explore possible reuse cycles.

## CONCLUSIONS

A BACS catalyst with a sulfonation percentage of 15.23 ± 0.43% was successfully obtained. When analyzed by SEM, a morphological change was observed due to a decrease in porosity. This was confirmed by the BET isotherm, which showed an increase in surface area and a decrease in pore size, which was caused by the increase in active sites of the catalyst, thereby aiding the transesterification reaction.

Upon studying the FAME conversion percentage in BACS, it is observed that it has a high production. No significant differences were shown between the proportion of catalyst used and the conversion percentage obtained. Using 1%



BACS, a conversion of 96.6+2.5% was reached. Employing the same percentage of KOH, a conversion to FAME of 65.01+0.07% was obtained. The characterization of the biodiesels obtained by using both catalysts revealed that the one produced with KOH complies with density, sulfated ashes, heat of combustion and viscosity according to the specifications of the RTCA 75.02.43:07 standard, while the one produced with BACS complied with all except viscosity. This parameter values are below the lower limit, so it is recommended to vary the reaction temperature in further experiments or blend it with diesel fuel. The BACS catalyst showed a reduction of  $2.78 \pm 0.71\%$  after the first work cycle, thus demonstrating its potential for reuse without having to sulfonate again.

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