CHEMIJA
2026. Vol. 37. No. 1. P. 53–62

ISSN 0235-7216 eISSN 2424-4538
DOI: https://doi.org/10.6001/chemija.2026.37.1.6

Evaluation of the efficiency of talc and bentonite in the dry purification of methyl and ethyl fatty acid esters as biodiesel from babassu

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The dry purification of biodiesel using natural adsorbents has emerged as a sustainable alternative to the traditional method of washing with water in biodiesel production. In this context, this study investigates the efficiency of talc and bentonite in the dry purification of biodiesel from babassu, a promising oilseed that is little used for biodiesel production. The biodiesel was produced by alkaline transesterification and purified with different concentrations of talc and bentonite. The adsorbents were characterised by X-ray diffraction, infrared spectroscopy and textural analysis. After purification, the physicochemical parameters of the biodiesel were analysed and compared with water-purified samples. Bentonite showed a higher surface area (76.685 m^2/g) and pore volume (0.121 cc/g) compared to talc (8.610 m^2/g and 0.029 cc/g). Both adsorbents efficiently reduced the acidity index, with bentonite being more effective, especially at a concentration of 3.0%. Moisture was significantly lower in samples treated with adsorbents, with bentonite standing out in ethyl biodiesel. The dielectric constant decreased with the use of adsorbents, indicating a greater removal of polar contaminants. Dry purification with talc and bentonite is efficient in improving the quality of babassu biodiesel, meeting the specifications of current legislation.

Keywords: adsorbents, sustainable treatment, unconventional oilseed, babassu

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INTRODUCTION

Recently, some government programs have encouraged the use of biodiesel, such as the European Union's Renewable Energy Directive (RED) [1], the Renewable Fuel Standard (RFS) in the United States [2] and, more recently in Brazil, the Fuel for the Future Law [3], which increases the limits for adding biodiesel to diesel. RED, for example, sets high targets for the use of renewable energies in total energy consumption, which increases the demand for sustainably produced biofuels [1]. In the United States, RFS establishes, by law, minimum quantities of renewable fuels that must be used each year, creating a predictable and secure market for biodiesel [2]. In Brazil, the Fuel of the Future Law stipulates that the percentage of biodiesel blended with regular diesel must gradually increase. The goal is to reduce greenhouse gas emissions in the transportation sector and strengthen domestic biofuel production [3].

To meet this demand, some oilseeds, such as babassu, are showing promise. One of the main advantages of babassu oil is its low cost, as well as its high content of saturated fatty acids, which can reach 91%, giving it oxidative stability and good properties, even at low temperatures [4]. Babassu biodiesel is rich in lauric acid (C12:0), a shorter-chain fatty acid that has important advantages such as flowing better at lower temperatures and being more resistant to degradation by heat and oxygen than biodiesels made from oils with longer-chain fatty acids, such as soybean oil [5, 6]. In addition, since most of the esters present in babassu are saturated, there is less oxidation than in soybean biodiesel, which contains more polyunsaturated esters. This lower tendency to oxidise is essential to ensure good performance and a longer shelf life during fuel storage and use [6]. During its conversion into biodiesel, impurities are formed that need to be properly eliminated, such as glycerol, unreacted alcohols, water, free fatty acids, soaps, and traces of catalysts. Removing these impurities is an essential step since their presence can negatively affect the quality of biodiesel, making it necessary to purify the final product to meet marketing standards [7, 8].

The conventional purification method involves washing with water, which is efficient in removing polar or water-dispersible impurities. It is a simple method that guarantees purity in the final product [9]. However, this technique uses large quantities of water and produces contaminated effluents, which is an environmental challenge [10]. These effluents contain high levels of organic matter and harmful chemicals, such as methanol and residues from catalysts used in biodiesel production. Therefore, they must undergo advanced and costly treatment processes before they can be safely disposed of in the environment [11]. Therefore, alternative approaches such as dry purification using solid adsorbents have been created to minimise water consumption and environmental impact [12]. Dry purification works on the basis of adsorption, where a solid adsorbent, which has a high affinity for impurities, attracts them and traps them on its surface. This process removes contaminants from biodiesel without the need for water or washing, thus avoiding the generation of liquid effluents [10].

Among the adsorbents with potential for dry purification are talc and bentonite. Talc, a hydrated magnesium silicate, due to its layered structure and inert surface, is capable of adsorbing impurities from crude biodiesel, significantly reducing the amount of soap and glycerol, as well as reducing the concentration of ethanol, being a low-cost and widely available material [13]. Bentonite, a smectite clay, stands out for its large surface area, layered organisation and ability to expand, which improves its efficiency in adsorbing contaminants. It is also abundant and inexpensive [14]. Bentonite has been optimised for use in dry purification of biodiesel. Its effectiveness comes from its lamellar structure and the possibility of chemically modifying its surface, which increases the area available for adsorption, giving it a high capacity to retain polar contaminants such as glycerol. The process occurs through physicochemical interactions, mainly Van der Waals forces and hydrogen bonds, which allow activated bentonites to remove impurities responsible for odours, moisture, and unwanted colour in crude biodiesel [15]. Both materials allow biodiesel to be obtained with characteristics suitable for use, making the process more economical and sustainable compared to conventional water washing methods.

Some studies have evaluated the use of talc and bentonite in biodiesel purification, but most studies use soybean, sunflower or waste frying oil [13, 14, 16]. Although adsorbents such as bentonite have already demonstrated their effectiveness in purifying biodiesel from other sources, including used cooking oil, castor oil, palm oil and jatropha oil [17–20], there is still a lack of studies on their specific application in babassu biodiesel. This gap is especially relevant because babassu oil has a unique composition, which gives its biodiesel superior oxidative stability. Understanding how dry purification affects or preserves this characteristic is critical to optimising the process and ensuring the quality of the final fuel. Therefore, this study aims to investigate the dry purification process of babassu biodiesel using talc and bentonite adsorbents.

EXPERIMENTAL

Materials

All chemicals and reagents used were of analytical grade. The babassu oil was purchased from OLEAMA (São Luís, Maranhão, Brazil). Potassium hydroxide (KOH), methanol and ethanol were obtained from Merck (Darmstadt, Germany). Talc and bentonite were supplied by Vetec Química Fina (Rio de Janeiro, Brazil) and Sigma-Aldrich (St. Louis, USA), respectively. Hydrochloric acid, diethyl ether and phenolphthalein (1.0%) were purchased from Synth (Diadema, Brazil) and Sigma-Aldrich (St. Louis, USA). Karl-Fischer reagent was obtained from Merck (Darmstadt, Germany). Helium gas (99.95% purity) was supplied by White Martins (Rio de Janeiro, Brazil).

Methods

Characterisation of babassu oil

Babassu oil was obtained commercially from the company OLEAMA (São Luis, MA, Brazil) and its properties were evaluated by acidity index (SMAOFD 2.201), peroxide index (SMAOFD 2.501), iodine index (SMAOFD 2.505), saponification index (SMAOFD 2.202), specific mass (ASTM D 4052) and viscosity (ASTM D 445).

Biodiesel production

Initially, the babassu oil was dried at 110°C for 4 h to eliminate any moisture content. At the same time, the catalyst solution was prepared by dissolving 2 g of KOH in 27 mL of methanol for the methyl route or 80 mL of ethanol for the ethyl route, both under constant stirring. For the transesterification reaction, 100 g of refined babassu oil was used, to which the alcoholic catalyst solution was added. The reaction continued for 60 min at room temperature. As the ethyl biodiesel contained excess ethanol, it was distilled to remove the residual alcohol. At the end of the process, both the methyl and ethyl reaction mixtures were transferred to a separating funnel and left to stand for 12 h.

Characterisation of the adsorbents

The adsorbents used, talc and bentonite, were structurally characterised by X-ray diffraction using a RIGAKU equipment, model DMax/2500PC (Japan), with CuKα radiation (λ = 1.5406 Å), in a 2θ range between 15 and 75° with a step of 0.0005°/s. Measurements in the infrared region were carried out using a SHIMADZU model IR PRESTIGE/21 Fourier Tranform Infrared Spectrophotometer with a scan between 400 and 4000 cm^{-1}. The textural analyses were carried out using nitrogen adsorption at 77 K in a MICROMERITICS model ASAP 2000 apparatus. Before analysis, the samples were treated at 100°C and subjected to a vacuum of 0.8 Pa.

Purification of biodiesel

The biodiesel purification process was carried out using talc and bentonite adsorbents, compared to the conventional washing method. For the adsorbent tests, talc or bentonite was added in different concentrations (0.5, 1.0, 2.0, 3.0 and 4.0% w/w) to 50 g of biodiesel (BE or BM). The selection of those concentrations was based on the established range of effective dosages reported in the literature for dry washing of biodiesel, typically between 1 and 5% w/w [21, 22]. The system was kept at room temperature under agitation for 120 min. After each adsorption step, the adsorbents were removed from the biodiesel by filtration using filter paper. For comparison purposes, the adsorbent purification method was compared with the conventional process, which consists of a wash with a 0.01% (v/v) HCl solution followed by four washes with distilled water.

Characterisation of biodiesel

After purification, the biodiesel was subjected to a series of analyses to verify its quality and conformity. The acidity index was determined according to the method of the Adolfo Lutz Institute [23]. 2.0 g of the sample was weighed, 25.0 mL of previously neutralised ether-alcohol solution (2:1) was added and the mixture was titrated with 0.01 mol/L KOH solution, using 1.0% phenolphthalein as an indicator. The water content was determined in accordance with EN ISO 12937 [24], whose method is based on Karl-Fischer titration. Capacitance measurements were carried out using an LCR 800 capacitance meter (GW Instek, Thailand) with a cylindrical capacitor. Analyses were carried out in triplicate at 27.0 ± 0.1 °C to avoid thermal interference in the sensor. The dielectric constant (ε) was calculated as the ratio between the sample capacitance (C) and the vacuum capacitance (C0). The ester content was quantified using gas chromatography. A Shimadzu GC-2010 chromatograph (Shimadzu Corporation, Kyoto, Japan) with a flame ionization detector and fused silica capillary column was used. The temperature of the injector and detector was 250°C. The oven temperature program was from 120°C (2 min) to 230°C. The carrier gas used was helium (99.95% purity) with a flow rate of 2.58 mL/min. The ester content was determined using methyl heptadecanoate as an internal standard by GCsolution 4.70 chromatograph software (Shimadzu Corporation, Kyoto, Japan).

RESULTS

Physicochemical characteristics of babassu coconut oil

The results of the physicochemical analysis of babassu oil showed the following parameters: acidity index of 0.3 mg KOH/g, peroxide index of 8.456 mEq/kg, iodine index of 17.22 g/100 g, saponification index of 249.98 mg KOH/g, specific mass at 20°C of 921.3 g/cm^3 and kinematic viscosity of 29.18 mm^2/s.

Physical characterisation of the adsorbents

The characterisation of the adsorbents used in the purification process showed differences between talc and bentonite (Table 1). Bentonite had a higher surface area and pore volume than talc, while talc had a higher mean pore diameter than bentonite.

Table 1. **Physical characteristics of talc and bentonite adsorbents**

Characteristics	Adsorbents	
	Talc	Bentonite
Surface area (m^2/g)	8.61	76.7
Pore volume (cc/g)	0.03	0.12
Average pore diameter (nm)	13.5	4.48

The X-ray diffraction (XRD) analysis of talc (Fig. 1a) showed that three main crystalline phases are present: muscovite (hexagonal), pyrophyllite (monoclinic) and quartz (hexagonal). The diffraction graph showed well-defined characteristic peaks, with the greatest intensity around 28° (2θ). For bentonite (Fig. 1b), the diffraction pattern revealed mainly two mineral phases: nontronite and montmorillonite, both with a hexagonal crystal structure. The point of the greatest intensity occurred near 7° (2θ).

The adsorbents showed different characteristic bands in the infrared spectra (Fig. 2). For bentonite,

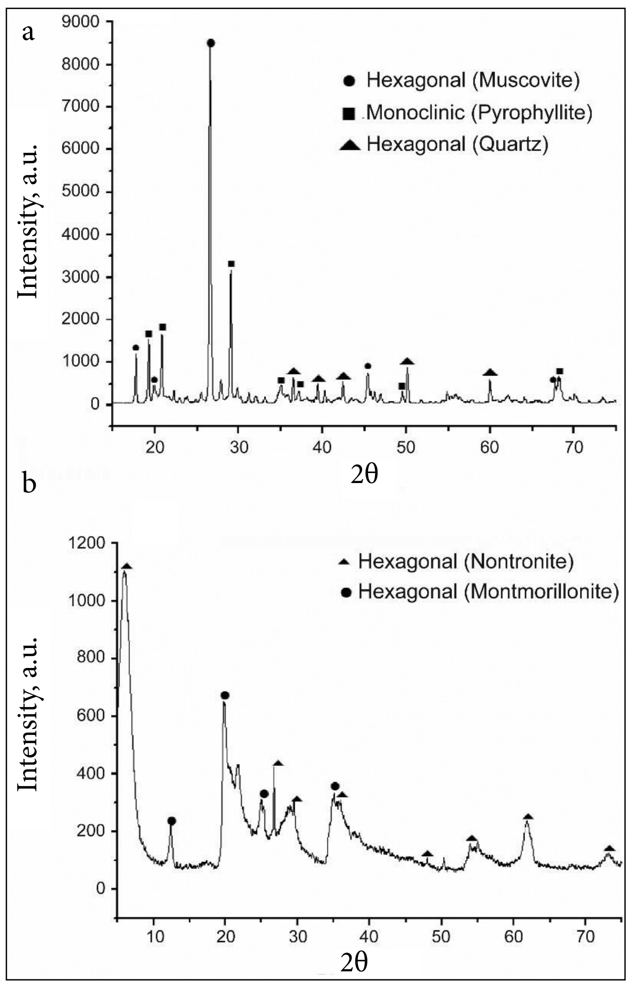

Fig. 1. X-ray diffractogram for talc (a) and bentonite (b)

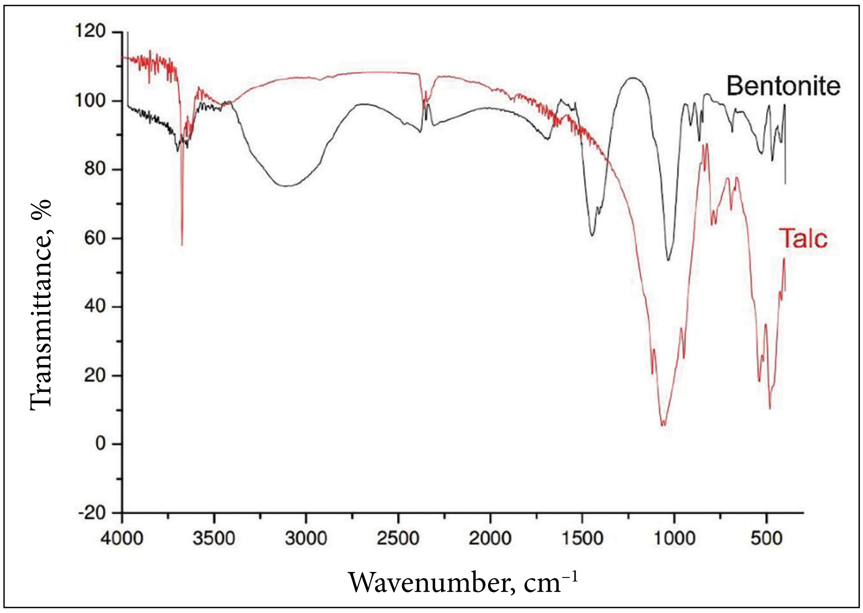

Fig. 2. Infrared spectra of bentonite and talc

bands were identified at 3105.39 cm^{-1} (axial deformation of the O–H bond of H_2O), 1033.84 cm^{-1} (Si–O–Si), 686.66 cm^{-1} (Mg–O), 526.57 cm^{-1} (axial deformation) and 468.70 cm^{-1} (Si–O–Al). Talc showed bands at 3674.39 cm^{-1} (O–H), 1055.20 cm^{-1} (Si–O–Si), 694.37 cm^{-1} (Mg–O), 540.07 and 480.20 cm^{-1} (Si–O–Al).

Applicability of adsorbents

The acidity index was evaluated for methyl (BM) and ethyl (BE) biodiesel after being purified with different concentrations of adsorbents (Fig. 3). With talc, the acidity levels of BM were between 0.20 and 0.27 mg KOH/g, while for BE the variation was between 0.21 and 0.26 mg KOH/g. Using bentonite, it was observed that the BM index ranged from 0.19 to 0.23 mg KOH/g, while the BE index ranged from 0.19 to 0.25 mg KOH/g. The acidity index of crude biodiesel (0%) was 0.29 mg KOH/g (BM) and 0.32 mg KOH/g (BE), while biodiesel washed with water had an index of 0.17 mg KOH/g (BM) and 0.19 mg KOH/g (BE).

After being purified with different concentrations of adsorbents, the moisture contents of the BM and BE were analysed and compared with the biodiesel washed with water (Fig. 4). For talc, the moisture content of BM ranged from 185.94

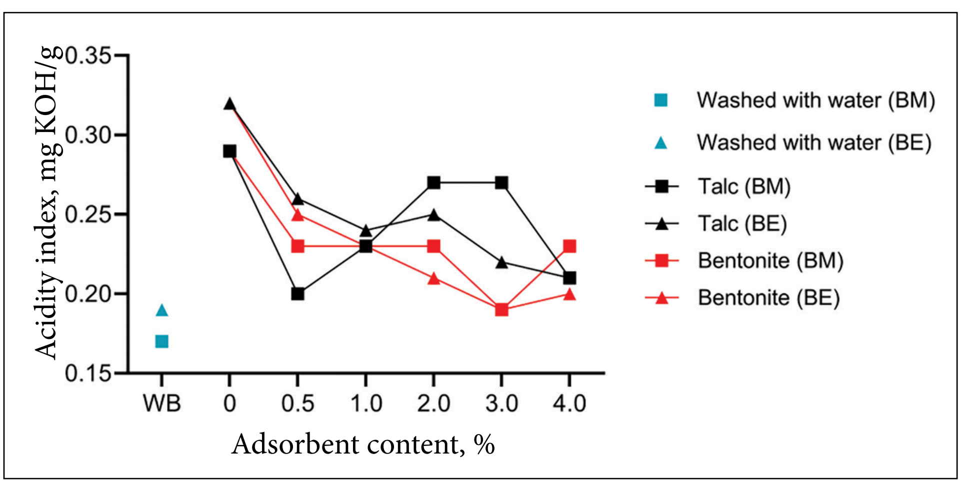

Fig. 3. Acidity index of methyl (BM) and ethyl (BE) biodiesel purified with talc and bentonite over a contact time of 120 min

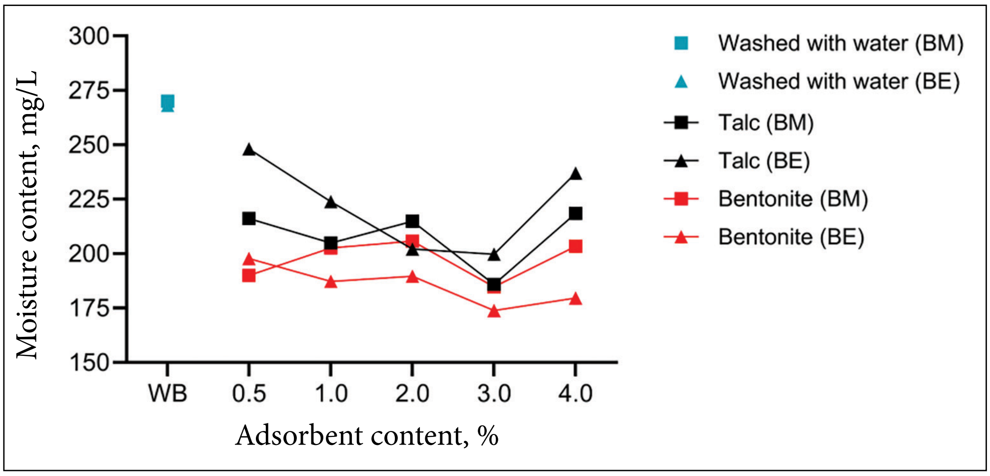

Fig. 4. Moisture content of methyl (BM) and ethyl (BE) biodiesel purified with talc and bentonite during a contact time of 120 min

to 218.52 mg/L, while for BE the range was 199.84 to 248.23 mg/L. With bentonite, the BM recorded values between 184.85 and 205.76 mg/L and the BE between 173.98 and 197.85 mg/L. The moisture content of the biodiesel washed with water was 270.11 mg/L (BM) and 268.13 mg/L (BE).

The dielectric constant of BM and BE was evaluated after the purification process using various adsorbent concentrations (Fig. 5). For talc, the BM values ranged from 1.368 to 1.373, and for BE from 1.307 to 1.359. With bentonite, the BM values ranged from 1.364 to 1.369, and for BE from 1.326 to 1.345. Crude biodiesel (0%) showed values of 1.398 (BM) and 1.364 (BE), while the biodiesel washed with water (BA) showed values of 1.375 (BM) and 1.359 (BE).

The chromatographic analysis (GC/DIC) (Fig. 6) showed the following ester contents for the purified samples with talc, BM showed 94.4 and BE 96.9%, with bentonite, BM reached 95 and BE 92%; by the traditional method (washing with water), BM reached 80.1 and BE 92.5%.

DISCUSSION

The results obtained for the physicochemical characteristics of babassu oil suggest that it is suitable for use as a raw material in biodiesel production. The low acidity index (0.3 mg KOH/g) below the EN 14214 recommendation (0.5 mg KOH/g) [25] is favourable, as values below 1.0 mg KOH/g are ideal for efficient transesterification [26, 27].

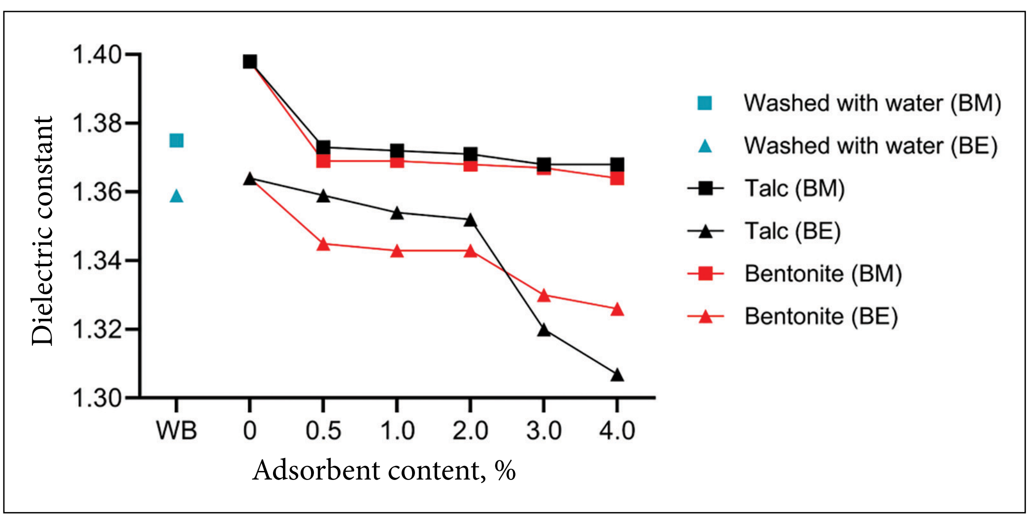

Fig. 5. Dielectric constant of methyl (BM) and ethyl (BE) biodiesel purified with talc and bentonite during a contact time of 120 min

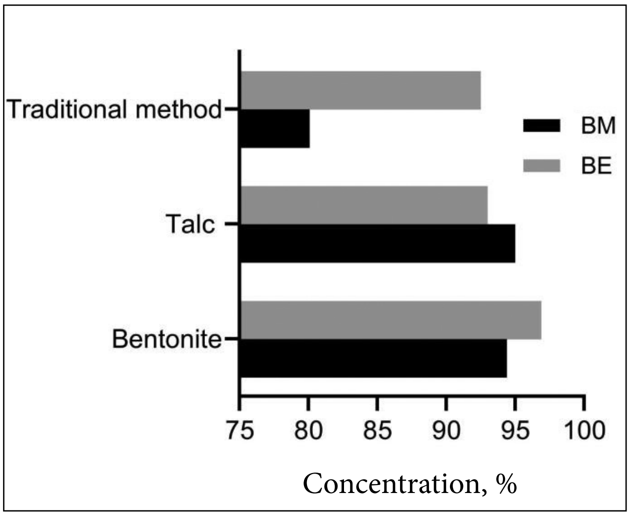

Fig. 6. Content of methyl (BM) and ethyl (BE) esters in biodiesel samples monitored by gas chromatography/DIC

The peroxide index (8.456 mEq/kg) is within the acceptable limits (<10 mEq/kg) [28], indicating that the oil has a good oxidative stability [29]. The iodine index of 17.22 g/100 g is significantly lower than the maximum of 120 g/100 g recommended by EN 14214 (120 g/100 g) [25], indicating few unsaturations in the oil, a characteristic that contributes to the stability of biodiesel, reducing its tendency to oxidise during storage [30]. The saponification index value (249.98 mg KOH/g) is within the common range for vegetable oils used in biodiesel production, which varies from 169.2 to 312.5 mg KOH/g [31]. The density and viscosity of the oil in natura are higher compared to the legislation [25]; however, these parameters are considerably reduced after conversion into biodiesel.

With regard to the physical properties of the adsorbents, it can be seen that bentonite had a larger surface area compared to talc. This gives it a better ability to adsorb contaminants since the greater the surface area, the greater the number of active sites where contaminant molecules can bind [32]. The greater pore volume of bentonite is also greater than that of talc, indicating a greater ability to retain contaminant molecules [33]. In terms of pore diameter, bentonite had smaller values than talc. This characteristic is disadvantageous as it limits the adsorption of larger molecules such as residual glycerol, as shown by previous studies [34, 35], which indicated an ideal pore size for adsorbents for this purpose of 20 to 50 nm, which is higher than both adsorbents studied.

XRD analysis showed the presence of muscovite and pyrophyllite in the talc. These compounds may contribute to the ability to adsorb polar compounds, especially pyrophyllite, given its previously reported ability to absorb anionic and cationic contaminants [36]. Bentonite, on the other hand, showed a characteristic composition of smectitic clay minerals, with montmorillonite and nontronite as the main constituents. The presence of these mineral phases justifies the high surface area observed in the physical analyses, since it has an expandable structure, as demonstrated by Widjaya et al. [37]. These characteristics favour the removal of polar contaminants from biodiesel through adsorption processes. Analysis of the infrared spectra showed the presence of hydroxyl groups (O–H) in both materials, indicating their ability to form hydrogen bonds with polar impurities such as glycerol and residual soaps [16]. The bands related to the Si–O–Si, Mg–O and Si–O–Al bonds, present in both materials, show a potential structural stability at higher temperatures and provide adsorption sites for the retention of impurities [37, 38].

The purification efficiency of the adsorbents was assessed by means of physicochemical analysis and compared with traditional cleaning with water. The acidity index results were below the limit set by the ANP (0.50 mg KOH/g) [39], both with talc and bentonite, indicating the effectiveness of both adsorbents in purifying biodiesel. The results were comparable to commercial adsorbents such as Magnesol® and silica, which generally have acidity values below 0.17 mg KOH/g [21]. Bentonite showed a slight advantage in reducing acidity, especially at a concentration of 3.0%, where it reached the lowest values for both BM and BE. This is because bentonite has a structure with a greater surface area and a high ion exchange capacity, which facilitates the removal of free fatty acids and consequently reduces acidity [40, 41]. When comparing the purification of babassu biodiesel with biodiesel from other sources, it is noted that the type of raw material directly influences how biodiesel responds to adsorbents. Studies indicate that babassu biodiesel has a slightly higher energy efficiency than soybean biodiesel, which can be attributed to its high content of medium-chain saturated fatty acids, such as lauric acid [42]. This characteristic helps make the transesterification reaction more efficient and produces biodiesel with fewer impurities from oxidation when compared to highly unsaturated oils, such as sunflower and soybean oils [43]. In the case of babassu oil, the use of bentonite was more effective in reducing the acidity of biodiesel. For biodiesels made from soybean and canola oils, however, the literature indicates that the use of magnesium silicates, such as talc, is the most recommended option for achieving low acidity levels and removing soaps efficiently [21]. Furthermore, the effectiveness of bentonite in removing moisture observed in this study with babassu biodiesel is consistent with previous results in palm biodiesels and waste oils, in which activated bentonite eliminated up to 90% of residual water [17]. Thus, although both talc and bentonite are effective adsorbents for different raw materials, babassu has the advantage of greater oxidative stability, observed by its low iodine index, which makes the adsorption purification process more efficient.

Compared to both adsorbents, water showed a better efficiency in reducing acidity indices. This is because water is able to solubilise the polar compounds dissolved in biodiesel responsible for the increase in acidity, such as free fats, soaps, catalyst residues almost completely, unlike bentonite and talc, which act mainly by surface adsorption [21, 44]. Although washing with water is more efficient in reducing acidity, the use of bentonite and talc is offset by the environmental and economic advantages of the dry process, since it reduces the use of water in the purification of biodiesel [45].

With regard to moisture reduction, bentonite outperformed talc in eliminating moisture, especially for ethyl biodiesel, possibly due to its greater surface area and pore volume. These properties allow bentonite to have a higher retention capacity, absorbing more moisture [16]. This result is compatible with previous studies [46], which show that bentonite is capable of removing up to 90% of the moisture in oils. In addition, the hydroxyl groups identified in the infrared contribute to a greater retention of water molecules, reducing moisture. It was noted that the purification process with water resulted in an increase in moisture. Higher humidity can result in problems during storage, such as a higher rate of hydrolysis and oxidation reactions, leading to an increase in the acidity index, as well as promoting the growth of microorganisms [47, 48]. Therefore, bentonite and talc are more efficient at removing wastewater.

Both adsorbents showed a reduction in dielectric constant compared to water, which indicates the adsorbents' greater ability to remove polar contaminants, while water can increase the dielectric constant through the addition of water molecules [16, 49], as demonstrated in the physicochemical analysis, where purification with water increased the moisture content of the biodiesel. The dielectric constant is related to the presence of polar compounds in biodiesel and is one of the indicators of the efficiency of the purification process. The results show a reduction in the dielectric constant with increasing adsorbent concentration, indicating a greater efficiency in removing polar contaminants such as glycerol and water [50, 51]. We also noticed a lower constant in BE compared to BM for all purification processes, which can be attributed to the greater polarity of methanol compared to ethanol [52].

Chromatography showed that dry purification with the adsorbents was more efficient than the traditional method with water, especially for BM, indicating a better efficiency in removing contaminants that could interfere with the transesterification reaction. For BE, among the methods tested, talc showed the highest yield, outperforming both bentonite and the conventional method. The better performance of talc can be explained by its larger pore diameter, which favours the adsorption of larger molecules. Despite this, it should be noted that the values obtained using the adsorbents meet ANP specifications (ANP, 2023), which establish a minimum ester content of 96.5%.

CONCLUSIONS

The dry purification method using talc and bentonite has been confirmed as a viable and more sustainable alternative to traditional water washing for babassu biodiesel. Both adsorbents were able to bring the final product up to the specifications required by current quality standards. The study showed that bentonite is more effective in the overall removal of polar contaminants, especially acidity and moisture. Talc, although less efficient in this removal, had the advantage of preserving the ester content in ethyl biodiesel, thus maintaining a higher yield of this compound. For future research, it is recommended to explore ways to regenerate and reuse both talc and bentonite, which could make the process more economical. It is also suggested to investigate chemical modifications in bentonite, with the aim of combining its high adsorption capacity with talc's ability to preserve esters, seeking an even more efficient and balanced purification for babassu biodiesel.

Received 8 December 2025
Accepted 12 January 2026

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TALKO IR BENTONITO EFEKTYVUMO ĮVERTINIMAS METILO IR ETILO RIEBALŲ RŪGŠČIŲ ESTERIŲ (BIODYZELINO) IŠ ATALIŲ ALIEJAUS SAUSOJO VALYMO METU