

A Comparative Study on the Performance of Fiber-Based Biosorbents in the Purification of Biodiesel Derived from *Camelina sativa*

Jie Yang¹, Quan (Sophia) He^{1,*}, Haibo Niu¹, Kenneth Corscadden¹ and Claude Caldwell²

¹Department of Engineering, Faculty of Agriculture, Dalhousie University, Truro, NS B2N 5E3, Canada

²Department of Plant and Animal Science, Faculty of Agriculture, Dalhousie University, Truro, NS B2N 5E3, Canada

Abstract: Biodiesel has received great interest as a promising substitute for petrodiesel. Biodiesel purification which follows the transesterification process is typically carried out using a wet washing process that generates large amounts of wastewater. Consequently, alternative methods are emerging as sustainable options for biodiesel purification. One of such methods is a dry washing process. In this paper, the performance of three dry washing media (commercially available BD-Zorb, sawdust and wood shavings) were evaluated as potentially suitable options for the purification of biodiesel derived from *Camelina sativa*. The results indicate that for the crude camelina biodiesel with an initial soap content of 9007 ppm, BD-Zorb exhibited the best purification performance. The soap removal capacity of BD-Zorb, sawdust, and wood shavings was 51.1 mL/g, 24.4 mL/g, and 9.4 mL/g respectively. The primary mechanism of soap removal using sawdust and wood shavings media was physical filtration and adsorption. While for adsorbent BD-Zorb, soap removal mechanism included adsorption and ion exchange due to the existence of a small amount of resins. The ion exchange led to a high acid number (1 mg KOH/g) of the purified biodiesel, and failed to meet the ASTM D6751 specifications (<0.5 mg KOH/g).

Keywords: Drying washing, purification of biodiesel, adsorbent, wood waste, *Camelina sativa*.

1. INTRODUCTION

The extensive use of fossil fuels has resulted in a dramatic decline in petroleum reserves, severe air pollution and growing concerns over global warming [1, 2] in recent years. These combined factors are driving researchers and industry practitioners to seek viable fuel alternatives. Biodiesel, a renewable, biodegradable and environmentally innocuous biofuel, has shown great potential to be used as a substitute for conventional petro-diesel [3-5].

Alkali-catalyzed transesterification is typically used for biodiesel production at an industrial scale due to fast reaction rate, mild reaction conditions, low cost of catalysts and easy operation. It is a process where vegetable oils (triglycerides) react with alcohol (usually methanol) to generate a mixture of fatty acid mono-alkyl esters (FAME or biodiesel) and a by-product (glycerol) in the presence of alkaline catalysts (KOH or NaOH). The majority of glycerol can be removed from biodiesel by phase separation because of the density and polarity difference between them. However, after glycerol removal, crude biodiesel still contains a variety of impurities, such as alcohol, catalyst, water, unremoved trace amounts of glycerol, unreacted

mono-, di- and tri-glycerides, free fatty acids and soap [6, 7]. The presence of those impurities may lead to adverse effects on fuel quality and the diesel engine [8, 9]. For example, an excess amount of total glycerol in biodiesel will cause undesirable deposits which may clog fuel lines and pumps [10]. Naturally occurring free fatty acids in vegetable oil can react with the catalyst, KOH or NaOH to form soap through a saponification process, which takes place in parallel with transesterification. Similar to glycerol, a high soap content can result in engine wear and negatively impact engine lifespan [6, 11]. Although there is no explicit specification of soap content in ASTM D6751, the generally acceptable soap limit should be lower than 66 ppm in the case where KOH is used as a catalyst in the biodiesel production processes. The existence of soap in biodiesel causes ash formation in diesel engine cylinders during biodiesel combustion. Water content (≤ 500 ppm) in biodiesel is another critical parameter associated with biodiesel quality. A high water content reduces the energy efficiency of fuel combustion, enhances the hydrolysis of FAME and even forms ice crystals resulting in gelling of fuels [12]. The FFA percentage in biodiesel is closely related to its acid number (≤ 0.5 mg KOH/g), which can cause corrosion of the engine's components [6]. Therefore, proper purification processes are essential to remove or minimize those impurities in crude biodiesel to meet standard specifications.

*Address correspondence to this author at the Department of Engineering, Faculty of Agriculture, Dalhousie University, Truro, NS B2N 5E3, Canada; Tel: 902 893 6180; Fax: 902 8901859; E-mail: Quan.He@Dal.Ca

A traditional approach for the purification of biodiesel at a commercial production scale is wet washing, based on the removal of water-soluble contaminants. Warm distilled or deionized water can effectively remove residual glycerol, soap, alcohol and catalyst from crude biodiesel. However, this process consumes a great amount of fresh water and generates aqueous effluent that has a detrimental impact on the environment [10]. Therefore, efforts have shifted to develop dry washing processes, using adsorbents such as resins [13], silicate magnesol [14], activated carbon [15, 16] and fiber-based materials [17, 18], or using membranes [19]. The adsorption capacity of cellulose fiber, a natural polymer derived from biomass has been demonstrated in a number of applications [20-25]. Recently, several economical alternatives such as Eucalyptus pulp [17] and Rice husk ash [18] have been used to adsorb impurities in crude biodiesel. BD-Zorb is a fiber-based commercially available dry wash medium for biodiesel purification. From its appearance, it consists mainly of wood chips and a small amount of resins. Due to commercial confidentiality, it is difficult to obtain any information on the species of wood chips, physical/chemical treatment of wood chips and chemical composition of the resins. This encourages us to explore the possibility of using sawdust and wood shavings, large amounts of waste generated by wood processing plants as biosorbents for biodiesel purification. Additionally, more than 95% of biodiesel worldwide is currently derived from traditionally edible vegetable oils such as soybean, canola and sunflower [26, 27]; this competes with the food and feed supply, raising a heated debate on “fuel vs. food.” Recent research has identified *Camelina sativa*, as a promising and sustainable oilseed crop for biodiesel production in North America [28, 29]. Camelina seed has a fairly high oil content (35-43% on a dry matter basis) [30,31], has

a short growing season and is tolerant to drought, cool weather and insect pests [32,33].

In this study, using camelina biodiesel as a model crude biodiesel, we explored the potential of using wood wastes such as sawdust (SD) and wood shavings (WS) as biosorbents in the dry washing purification processes, and compared the performance of the wood waste to that of commercially available adsorbent, BD-Zorb. The soap level, acid number and water content of purified biodiesel were indicators for evaluating the purification performance of dry wash media.

2. MATERIALS AND METHODS

2.1. Materials

Unrefined camelina oil was cold pressed from seeds grown in Canning, Nova Scotia, Canada. Potassium hydroxide in the form of pellets, analytical grade methanol (>99%), hexane and ethanol were purchased from Fisher Scientific Ltd., Canada. Xylene, chloroform and two titrants (composite 5 and 0.1 N KOH) were purchased from Sigma Aldrich, Canada. A standard reference solution of camelina methyl esters (GLC 937, >99%) was purchased from Nu-Chek Prep. Inc. USA. Three fiber-based adsorbents used in this study are shown in Figure 1. BD-Zorb was purchased from Utah Biodiesel Supply Ltd., USA, and sawdust and wood shavings were provided by the wood shop in Dalhousie University, Agricultural Campus, Truro, NS, Canada.

2.2. Characterization of Adsorbents

The surface morphology of three adsorbents was studied by scanning electron microscopy (SEM) in a Hitachi S-4700 equipment with 10 kV and magnification

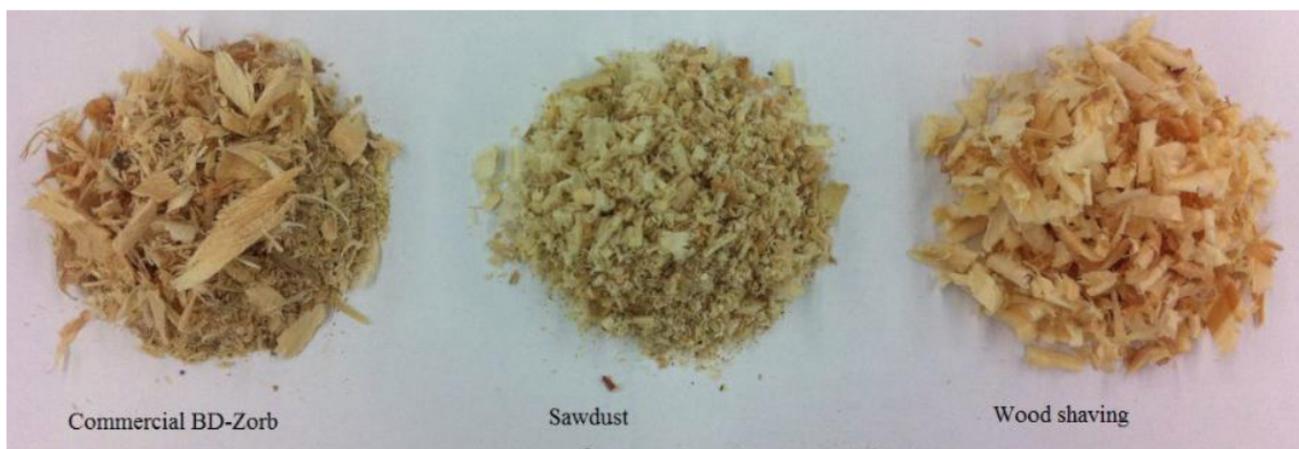


Figure 1: Three adsorbents applied for biodiesel purification: BD-Zorb, sawdust and wood shavings.

of up to 5000×. The Brunauer-Emmett-Teller (BET) specific surface area of adsorbents were determined by the Quantachrome Autosorb-3B with Krypton as the adsorbate. The functional groups of sawdust were analyzed using Fourier Transform Infrared (FT-IR) Spectrometer (PerkinElmer Spectrum One) in the range of 4000-400 cm^{-1} with a resolution of 4 cm^{-1} and 32 scans.

2.2. Camelina Biodiesel Synthesis

Camelina biodiesel was prepared by an ultrasound-assisted transesterification process [34]. 1 kg camelina oil was added to the reactor, and the temperature was set at 50 °C, a reaction time of 1 hour, methanol/oil molar ratio of 8:1 and 1.3 wt. % KOH catalyst with respect to oil feed [35,36]. The reaction mixture was transferred to a separatory funnel and allowed to stand for 30 min for phase separation. The glycerol layer under the crude biodiesel was drawn off, giving 0.93 kg crude biodiesel layer for further purification.

2.3. Purification Process

The purification of crude camelina biodiesel in a dry washing process was performed in three cylindrical separator funnels (125 mL), each filled with 18 g BD-Zorb, sawdust and wood shavings respectively. Cotton gauze was applied on the top of the packed adsorbent to distribute the added crude biodiesel evenly and minimize the channeling effect. Cotton gauze was also placed at the bottom of the adsorbent bed to hold the mass. The flow rate of biodiesel was 100mL/h and was controlled using a rotary knob and effluent was collected every 50 mL for further testing.

In a wet washing purification process, 50 mL of crude camelina biodiesel was transferred to a 250 mL separatory funnel and washed by adding 100 mL of distilled water until the water layer was transparent. The temperature of distilled water was set at 20 °C and 50 °C to compare the effect of water temperature on purification efficiency. All purification operations were conducted in duplicate.

2.4. Characterization of Camelina Biodiesel

The content of fatty acid methyl esters (FAME) at camelina biodiesel were determined using an Agilent 7890A Gas Chromatography (GC) equipped with a flame ionization detector (FID) and an Agilent DB-23 column (50%-Cyanopropyl-methylpolysiloxane; 30-m length × 0.25-mm internal diameter × 0.25 μm thickness; high polarity). The fatty acid methyl esters

were identified by comparing their specific retention times to those of a standard reference solution of camelina methyl esters (GLC 937).

The soap level of biodiesel was determined by titration examination: 12 mL biodiesel and 1 mL 0.04% aqueous Bromophenol blue were added into 100 mL of isopropanol. The solution was mixed well using magnetic stirring, followed by dropwise adding 0.01 N hydrochloric acid (HCl) until the color of the solution changed from blue or/and green to yellow. The amount of HCl used (mL) was recorded and the soap level of biodiesel was determined as follows:

$$\text{Soap content (ppm)} = \frac{\text{the amount of HCl added (mL)} \times 320}{\text{mL of biodiesel}}$$

Where 320 is the average molecular weight of soap in biodiesel.

The water content of biodiesel was determined by Karl Fisher volumetric testing method in Mettler Toledo T7 titrator. Biodiesel sample of 3 g was added into titration vessel containing 40 mL methanol and 30 mL chloroform and xylenes mixture (1:1 in volume), followed by adding titrant (composite 5) automatically until reaching the ending point. The water content was calculated shown in the panel of titrator. The acid number of camelina biodiesel was measured by an accessory electrode coupled in Mettler Toledo titrator. 15 grams of biodiesel was dissolved in 60 mL of diethyl and ethanol mixture (1:1 in volume) in titration vessel. 0.1 N KOH titrant was stepwise purged into the titration vessel to determine the acid number.

3. RESULTS AND DISCUSSION

3.1. Characterization of Adsorbents

The macroporous structures of the three fiber-based biosorbents of interest in this study were captured using a SEM under two different magnifications (500× and 2000×) as shown in Figure 2. BD woodchips (Figure 2a and 2b) have fewer pores compared to sawdust (Figure 2c and 2d) and wood shavings (Figure 2e and 2f). BET analysis indicated that SD had the largest specific surface area (1.06 m^2/g) of these three dry wash media, followed by WS (0.8 m^2/g) and BD (0.49 m^2/g) as shown in Table 1.

3.2. Characterization of Crude Camelina Biodiesel

The properties of crude biodiesel and the composition of fatty acid methyl esters were presented

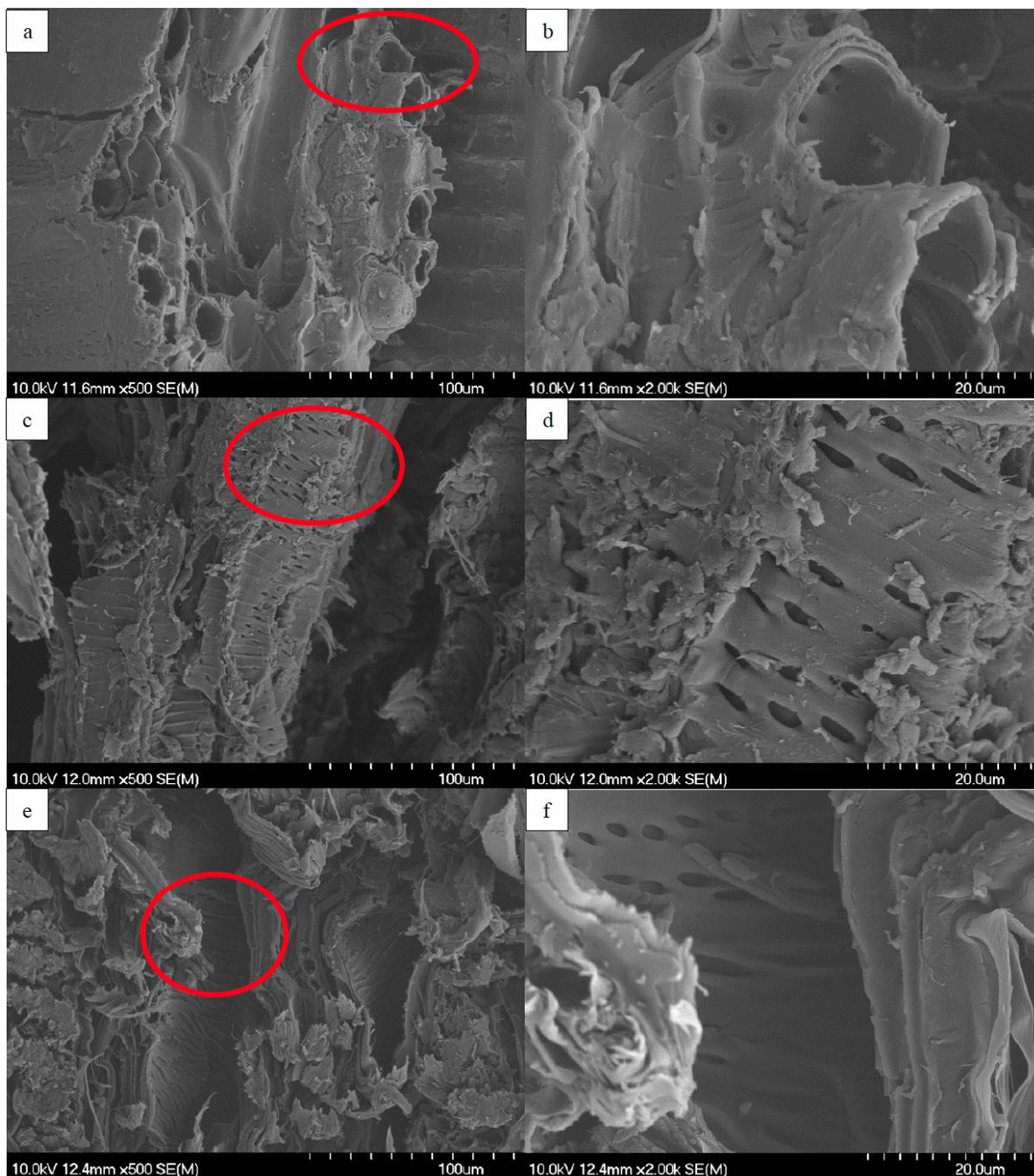


Figure 2: SEM images of BD woodchips (a & b), sawdust (c & d) and wood shaving (e & f) under two different magnifications (500× & 2000×).

Table 1: Specific Surface Areas of Three Fiber-Based Biosorbents

	BD-Zorb	Sawdust	Wood Shavings
Specific surface area, (m ² /g)	0.49	1.06	0.80

in Table 2. Crude camelina biodiesel contained 97.5 wt. % of FAME, complying with EN14214 standard. The water content of crude camelina biodiesel of 2011 ppm

was much higher than the minimum values specified in biodiesel quality references, ASTM D6751 or EN 14214. The acid number was 0.07 mg KOH/g, a pretty

Table 2: The Properties of Fatty Acid Profile of Crude Camelina Biodiesel

Properties	ASTM D6751	EN 14214	Crude Biodiesel
Soap content (ppm)	-	-	9007
Acid number (mg KOH/g)	0.5	0.5	0.07
Water content (ppm)	500	500	2011
FAME (wt. %)	-	96.5	97.5
Myristic methyl ester, C14:0			0.1
Palmitic methyl ester, C16:0			5.5
Palmitoleic methyl ester, C16:1			0.1
Stearic methyl ester, C18:0			2.4
Oleic methyl ester, C18:1			14.4
Linoleic methyl ester, C18:2			19.1
Linolenic methyl ester, C18:3			33.5
Arachidic methyl ester, C20:0			1.5
Gadoleic methyl ester, C20:1			15
Eicosadienoic methyl ester, C20:2			2.2
Arachidonic methyl ester, C20:4			1.4
Behenic methyl ester, C22:0			0.3
Erucic methyl ester, C22:1			3.1
Clupanodinic methyl ester, C22:2			0.2
Docosatrienoic methyl ester, C22:3			0.4
Lignoceric methyl ester, C24:0			0.2
Nervonic methyl ester, C24:1			0.6

Note: the individual percentage of fatty acid methyl esters were adapted from our previous work [38].

low value against biodiesel specifications. The soap content in crude camelina biodiesel was 9007 ppm, relatively high compared to other studies [13, 37].

The high-level of soap resulted from a stoichiometric excess of KOH (1.3 wt. %), and thus saponification reaction between FFA in parent camelina oil and KOH occurred during the transesterification process. The reaction mechanism of saponification was shown in Figure 3. The hydrogen ions in FFA can be readily replaced by potassium ions (K^+) from KOH and then form soap and water. It is worthwhile to point out that the soap level was high because the soap measurement was taken right after the glycerol removal in the present study. If the crude

biodiesel after separating glycerol had settled for a period of time, the soap would partially concentrate at the bottom of the storage tank, and could then be separated by density difference, which would greatly reduce the burden for downstream purification. In this study, the crude camelina biodiesel with a soap level of 9007 ppm was used for evaluating the performance of the three biosorbents in the following sections.

3.3. Purification of Crude Biodiesel Using Different Fiber-based Dry Wash Media

Crude camelina biodiesel with an initial soap level of 9007 ppm was purified by using three fiber-based adsorbents, including commercially available BD Zorb

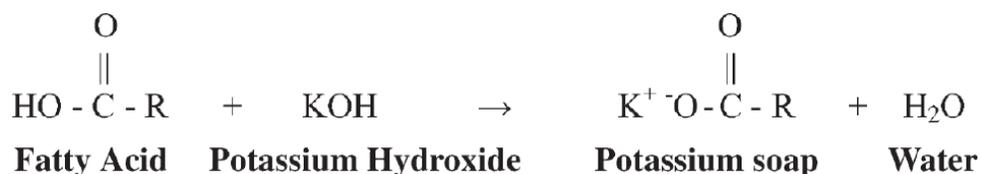


Figure 3: Formation of soap between fatty acid and excessive KOH catalyst [37].

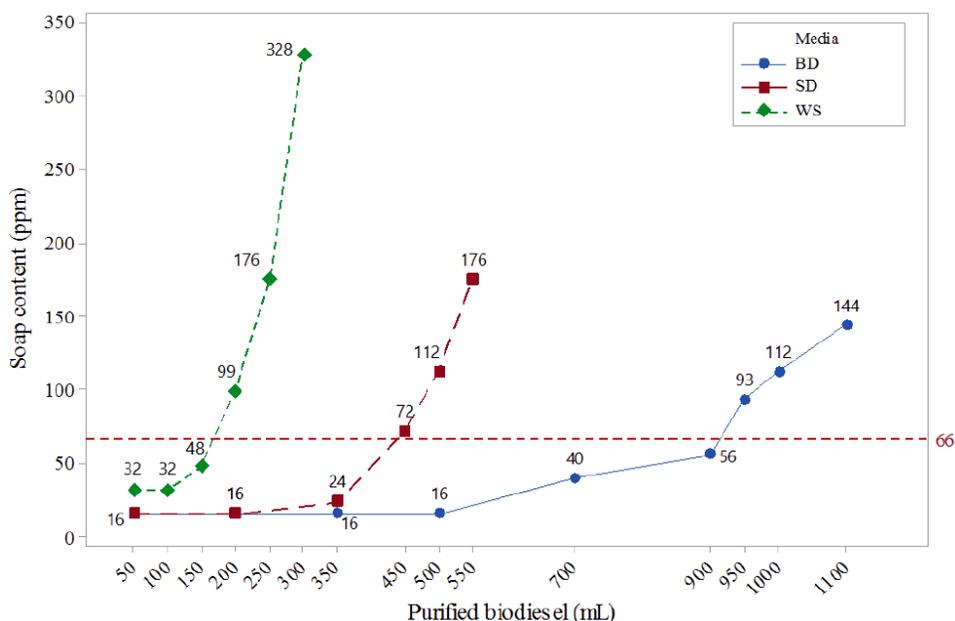


Figure 4: Comparison of soap removal efficiency of the three dry wash media, BD Zorb (BD), Sawdust (SD) and Wood shavings (WS) (18 g each).

and waste products from wood processing plants (sawdust and wood shavings). Figure 4 presents a comparison of the soap removal efficiency of these three adsorbents. Clearly, all of them were effective in removing soap and decreased the soap content from the initial concentration of 9007 ppm to less than 66 ppm for the first 150 mL of biodiesel treated. However, wood shavings were saturated by soap quickly and their adsorption ability decreased. The soap level increased to 99 ppm, 176 ppm and 328 ppm when treating 200 mL, 250 mL and 300 mL of biodiesel respectively. Sawdust displayed a better soap removal ability than wood shavings, and the soap level was 72 ppm after treating 450 mL crude biodiesel as shown by the red dashed line. This was mainly attributed to a larger specific surface area of SD than that of WS as shown in Table 1. Compared to WS and SD, BD Zorb exhibited much higher soap removal efficiency even though its specific surface area was lower than that of SD. The soap level of biodiesel purified using BD Zorb was 56 ppm after treating 900 mL of crude biodiesel. This is because a small amount of resin exists in BD Zorb, which enabled soap removal more effectively than could be achieved using fibers [13].

The acid number of biodiesel is a good indicator of free fatty acid (FFA) content in biodiesel, representing its degree of acidity. In the transesterification process, excessive KOH was applied to obtain high quality biodiesel. Therefore, the initial acid number of crude camelina biodiesel was very low (0.07 mg KOH/g). The

effects of dry wash media on the acid number of the purified biodiesel are shown in Figure 5. The acid number of BD Zorb purified for the first 50 mL of biodiesel was 1.73 mg KOH/g, which was higher than the initial value of 0.07 mg KOH/g. This can be explained due to the small amount of resin existing in BD Zorb removing soap *via* ion exchange and thus generated a considerable amount of FFA, leading to an increased acid number. A steadily declining trend of acid number was observed as the volume of treated biodiesel increased, indicating that the ability of resins to remove soap decreased gradually when the crude camelina biodiesel was continuously fed to the BD Zorb bed. The acid number of purified biodiesel using sawdust and wood shavings also increased to 0.43 mg KOH/g and 0.38 mg KOH/g respectively from the initial value of 0.07 mg KOH/g when the first 50 mL crude biodiesel was treated, and then decreased with increasing amounts of crude biodiesel. The acid number of the biodiesel purified by sawdust and wood shavings were lower than 0.5 mg KOH/g, the limit specified in the ASTM standard, but BD Zorb purified biodiesel had a much higher acid number (1.01 mg KOH/g) and failed to meet biodiesel standards.

Figure 6 showed the water removal efficiency of the three dry wash media. It was found that the three media were able to adsorb water from the crude camelina biodiesel to a certain degree, however failed to meet the ASTM D6751 (<500ppm) requirement. The water content decreased from an initial level of 2011

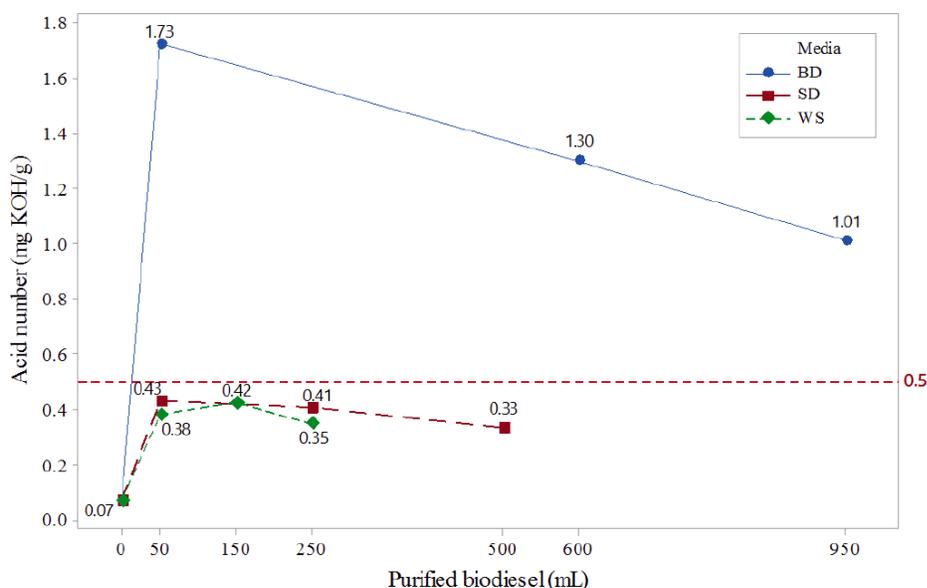


Figure 5: Acid number of the purified camelina biodiesel treated by dry washing media, BD Zorb (BD), Sawdust (SD) and Wood shavings (WS) respectively (18 g each).

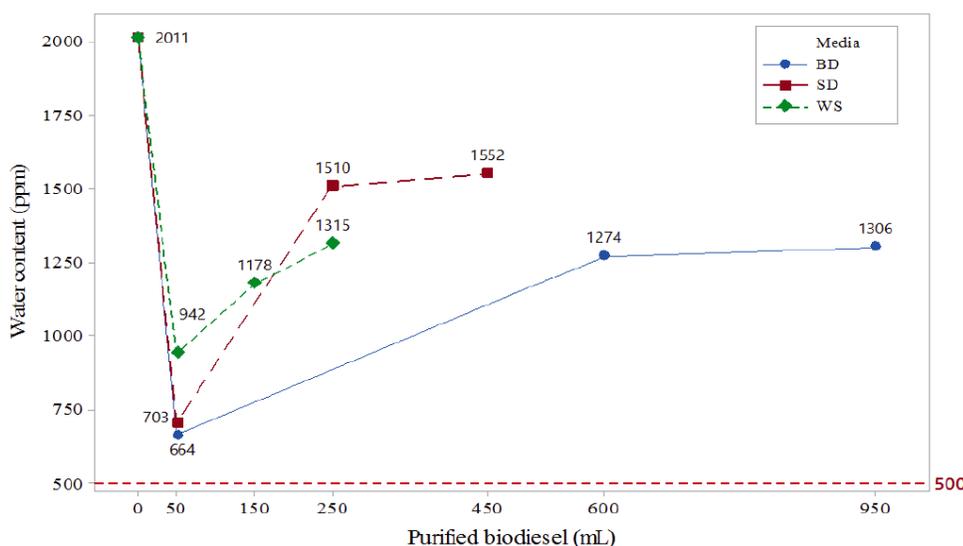


Figure 6: Comparison of the water content of the purified camelina biodiesel treated by dry washing media, BD Zorb (BD), Sawdust (SD) and Wood shavings (WS) (18 g each).

ppm to about 664-942 ppm when the first 50 mL of biodiesel was purified, and then gradually increased with increasing amounts of crude biodiesel fed to purification columns. The water content in the purified biodiesel was associated with the dryness of the three adsorbents. As this study mainly focused on the soap removal ability of sawdust and wood shavings, the moisture content of three adsorbents was not tested and recorded.

In summary, the soap removal capacity of the three fiber-based biosorbents was 9.4 mL/g, 24.4 mL/g and 51.1 mL/g for wood shavings, sawdust and

commercially available BD-Zorb respectively (soap content threshold set at 66 ppm). Table 3 also showed the water content and acid number of purified camelina biodiesel.

Based on these observations, it was found that the purification mechanism of sawdust and wood shavings was mainly due to physical filtration and adsorption. Porous fiber-based materials were able to absorb soap and moisture. Sawdust has a larger specific surface area than wood shavings, therefore showed a higher purification capacity. Ion exchange mechanisms may also be involved. Sawdust and wood shavings typically

Table 3: The Properties of Camelina Biodiesel Purified by Dry Wash Media and their Adsorption Capacities

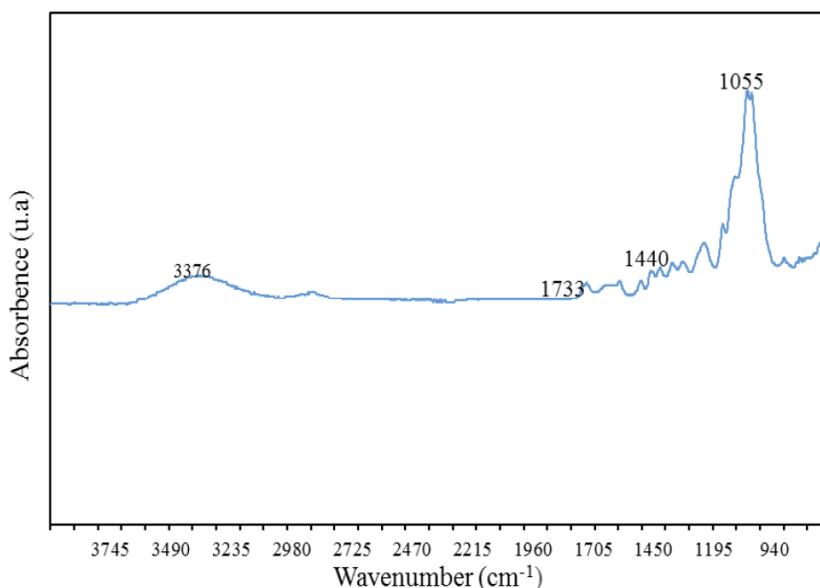
Adsorbent Used	Soap Content (ppm)	Acid Number (mg KOH/g)	Water Content (ppm)	Estimated Volume of Biodiesel Purified (mL)	Soap Removal Capacity (mL Biodiesel /g Adsorbent)
BD	66	>1.01	Nearly 1306	920	51.1
SD	66	<0.41	Nearly 1552	440	24.4
WS	66	<0.42	Nearly 1178	170	9.4

consist of 40-50% cellulose, 20-30% hemicellulose and 15-30% lignin [39, 40]. An FT-IR spectrum of sawdust was presented in Figure 7 (FTIR of wood shavings is similar to sawdust, not shown in this paper). There was a broad absorbance at around 3400 cm^{-1} , which corresponded to stretching bands of -OH (existing hydrogen bond) (17,18). The region between $1650\text{--}1750\text{ cm}^{-1}$ were attributed to the carbonyl and carboxyl C=O stretching [41]. The peak at 1440 cm^{-1} was responsible for the carboxylic functional group [42]. The relatively high peak at 1055 cm^{-1} corresponded to the C-O stretching [41]. Hence, sawdust and wood shavings were able to provide hydrogen ions for exchanging with cations K^+ in soap, therefore generated a very limited amount of FFA. This explained a slight increase in acid number of biodiesel purified using sawdust or wood shavings.

For BD Zorb, ion exchange played an important role in the dry washing process due to the presence of resins in wood chips. This can be seen from a much higher soap removal capacity (two times higher than sawdust) and dramatically increased acid number in the purified biodiesel.

4. CONCLUSIONS

Purifying crude biodiesel using dry washing media is an attractive and sustainable option. This study demonstrated that three fiber-based biosorbents (BD Zorb, SD and WS) were able to effectively remove soap from crude camelina biodiesel. The soap removal capacity was 9.4 mL/g , 24.4 mL/g and 51.1 mL/g for WS, SD and commercially available adsorbent, BD-Zorb respectively from an initial soap content of 9007 ppm . BD-Zorb exhibited the highest soap removal capacity, however, the acid number of BD-Zorb purified camelina biodiesel ($> 1\text{ mg KOH/g}$) was much higher than the limit of 0.5 mg KOH/g specified in the ASTM D6751 standard due to the existence of a small amount of resin. The mechanism of soap removal using sawdust and wood shavings was physical filtration and adsorption, while for BD-Zorb, ion exchange mechanism was involved in the soap removal. The water content of camelina biodiesel purified using the three biosorbents were all greater than the limit of 500 ppm stated in biodiesel quality references. These results indicate that additional steps are necessary to decrease the water content and acid number of biodiesel purified by fiber-based dry washing media.

**Figure 7: FT-IR spectrum of sawdust.**

Sawdust and wood shavings do have potential as economical adsorbents for the purification of crude biodiesel, however, compared to commercially available adsorbents, a lower purification capacity implies more frequent replacements of adsorbents packed in purification towers, which might lead to an increase in labor costs. A thorough evaluation of the economic viability of using wood wastes is essential in the future research.

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