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Article

Biodiesel dry purification using unconventional bioadsorbents

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Abstract:

The dry washing method is an alternative to replace water washing, thereby reducing the negative impacts of contamination. However, commercial adsorbents come from industrial processes that, due to their composition, may not be such a sustainable resource in the global biodiesel production process. In this study, the use of organic residues, such as sawdust, coconut fiber, nutshell, rice husk and water hyacinth fiber, were proposed as bioadsorbents for the purification of biodiesel from waste cooking oil (WCO). Quality parameters such as the acid value, water content, and free and total glycerin content were evaluated and compared with those of commercial resins such as Magnesol® and Amberlite™. Promising results were obtained using sawdust during the purification process, achieving a 31.6% reduction in the acid value compared to that of unpurified biodiesel, the reduction was 31.3% more efficient than Amberlite™. Sawdust adsorbed free glycerin at 55.8%, being more efficient than Amberlite™. The total glycerin values were similar between commercial resins and sawdust. A water content values were similar than Amberlite™ and better than that with Magnesol®, at 4.3% and 39.81%, respectively. These results show that sawdust can be used as an alternative bioadsorbent in a dry purification method for biodiesel being a residue with less environmental impact.

Keywords: Bioadsorbents; biodiesel; waste cooking oil; purification

1. Introduction

Currently, approximately 80% of the world's energy consumption comes from fossil fuels [1]. The environmental problems associated with its use include air pollution and global warming [2]. The high dependence on fossil fuels for industrial, transportation and domestic purposes has led to research on alternative energy sources [3]. Biodiesel is an alternative to petrodiesel with the aim to mitigate the problem of fossil fuel depletion and environmental impact [4]. The main raw materials are vegetable oils or animal fats, which are transformed to Fatty Acid Methyl Ester (FAME) through the transesterification process [3, 4]; this method involves the conversion of triacylglycerides into methyl esters (methanol) or ethyl esters (ethanol) in the presence of a catalyst (KOH or NaOH) [4, 5].

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The use of biodiesel reduces greenhouse gas emissions in the production process, which is one of the most important advantages. Moreover, the biodegradability, low cost and high availability of biodiesel if it is produced from Waste Cooking Oil (WCO) are additional benefits [4, 6].

One of the challenges of biodiesel production is quality. Although the use of efficient heterogeneous catalyst and biocatalyst could help [7-9], purification remains one of the most important stages in the production of biodiesel. At this stage, the content of methanol, free glycerin, and soaps, among other impurities, may be decreased and thus comply with ASTM D6751 [10] and EN 14214 [11] specifications and with the official Mexican guidelines [12].

Among the quality parameters that interfere in the transesterification process is the acid value, on which the transformation of fatty acids depends [13], and this value is related to the degradation of the biofuel within the combustion chamber [14]. Another quality parameter is free glycerin, and at high concentrations, it can form deposits in storage tanks, filters and even injectors, reducing the useful life of engines [15]. Likewise, the water content is within biodiesel quality standards, as water is corrosive to the engine, and it is important to comply with the established limits [16]. These limits guarantee that the product obtained does not have negative effects on the engine or the environment; therefore, it is necessary that the biofuel meets quality standards.

The most used methods for biodiesel purification are water washing, ion exchange resins (Amberlite™) and adsorbents such as silica and Magnesol® [17]. Water washing is distinguished by the removal of a large portion of biodiesel impurities, such as glycerin, methanol, soaps and other hydrophilic compounds [18]. Unfortunately, the use of water increases the cost, production time, and liquid effluent contamination and causes significant product loss due to the formation of emulsions and soaps [19].

Magnesol® is a frequently used adsorbent due to its high efficiency in the removal of glycerin, methanol, soaps and water [17]. Another resin currently used for dry cleaning of biodiesel is Amberlite™ BD10DRY™, which is an ion exchange polymer for the removal of soaps and glycerin [20]. Among the disadvantages is the formation of free fatty acids, which increases the acid value [21].

The use of commercial resins causes a great economic impact, increasing the cost of biodiesel production, as well as an environmental impact due to their final disposition when become saturated. An alternative to this problem could be the use of bioadsorbents from industrial waste (wood and food) and water hyacinth (Eichornia crassipes), which is considered an invasive species that causes negative effects on aquatic ecosystems. Eichornia crassipes is now present on all continents except Antarctica and has invaded all tropical and subtropical countries, as well as some parts of the Mediterranean basin. It is considered one of the world's most invasive aquatic plants, and considerable effort is expended worldwide to manage E. crassipe and its impacts on agriculture, the environment and human activities [22]. Species has reached up to two million plants per hectare [23].

Another alternative source of bioadsorbents is industrial waste. In 2018, the worldwide forestry waste production was 0.234 km^3, consisting of sawdust, chips and bark [24]. In Mexico, large quantities of waste are produced, coming from the agricultural and forestry sector; the annual timber production generates approximately 2.8 million m^3 of waste, which is made of sawdust, chips and bark [25].

One crop that generates large amounts of waste is rice; this cereal is a staple food that forms the basis of the traditional diet of a large proportion of the human population. According to Zou et al (2019) the rice global production in 2014 was 741.48 million tons [26]. The national production of rice

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in Mexico was 0.25 MMt in 2017 [27]. Rice husk is used to produce feed for cattle, among other applications but could be an adsorbent to purify biodiesel [28].

Pecan husk is another alternative, the world pecan 2019/2020 crop was estimated at 139,739 metric tons. Production was led by Mexico and the USA with very similar shares, 47% and 43% respectively. In México the pecan nut industry production is 65,750 MT and considering the pecan shells represent 49% of the pecan, the production of pecan shells is 32,217 MT [29, 30].

Currently, resins from these wastes are used in different sectors and sawdust is used to produce pellets, substrate for plants and balanced food [25].

Coconut coir is applied in gardening, in production of ropes, mattress padding and in industrial processes [31, 32]. Regarding the coconut industry, 90% is in India and Sri Lanka, where the coconut fiber production is approximately 350,000 tons [33].

The absence of optimal management of these wastes represents an environmental problem, so it is necessary to develop alternatives for their use. One solution could be the use of these resources as bioadsorbents for biodiesel purification. This article aims to a) evaluate the efficiency of sawdust, coconut coir, nutshell, rice husk and water hyacinth fiber in the removal of free and total glycerin, water and Free Fatty Acids (FFA) from biodiesel derived from WCO and b) compare them with conventional purification methods, such as using Magnesol®, Amberlite™ BD10DRY™ and water washing.

2. Materials and Methods

2.1. Biodiesel production

The WCO was donated by an institutional restaurant and was characterized to obtain the acid value and thus know the amount of catalyst to use. Biodiesel production was carried out in a pilot plant with an effective volume of 150 L and water was removed from the oil by heating at 100 °C for 60 min. Forty liters of WCO were mixed with a methoxide solution (12.7 L of methanol and 360 g of KOH) in an 8:1 molar ratio. The transesterification process lasted four hours by recirculating the oil in the equipment pump; the separation phase lasted 24 hours. The methanol was removed by a rotary evaporator system (Sagaon V3) for 15 min at a pressure of 0.07 MPa and 80 rpm.

2.2. Purification

2.2.1. Conventional methods

2.2.1.1. Purification with Magnesol® and AmberliteTM BD10DRYTM

A total of 100 mL of unwashed biodiesel was measured and added to a 250 mL beaker. The purification was carried out in batch mode at room temperature (15 °C) and stirred at 700 rpm. The Magnesol® concentration used in the sample was 1% (w/w), and the purification time was 20 min. In the case of Amberlite™ BD10DRY™, the amount was 10 g. Subsequently, the sample was filtered on a 50 mL column and a 1 μm filter using a vacuum system. The samples were stored for later analysis. Purification was carried out in duplicate. Magnesol® and Amberlite™ BD10DRY™, were provided by The Dallas Group, Inc. and Dow Mexico, respectively.

2.2.1.2. Water purification

A total of 100 mL of unwashed biodiesel was measured, and a 1:1 (v/v) ratio of distilled water was added at a temperature of 40 °C and stirred gently for 10 min. Three washes were performed until the washings were clear. The biodiesel was washed at 15 °C and separated by gravity in a

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separatory funnel. Subsequently, the sample was dried at 100 °C for 30 min and 700 rpm on a magnetic stirrer.

2.2.2. Bioadsorbents

Sawdust, coconut coir, nutshell, rice husk and water hyacinth fiber, which were purchased by local producers, were used as bioadsorbents. All samples were dried in an electric oven at 100 °C for 3 h. The water hyacinth was collected from Lake Cuitzeo and was donated by a local university (Universidad Michoacana de San Nicolás de Hidalgo). Each of these samples was added to unwashed biodiesel at a concentration of 5% (w/w) and at 15 °C. The samples were stirred for 20 min at 700 rpm, filtered on a 50 mL column and a 1 μm filter using a vacuum system and stored for future analysis.

2.3. Characterization of biodiesel

The biodiesel (purified and unpurified) was analyzed following the ASTM D664 acid value standard by potentiometric titration [34] and the ASTM D4928 standard for moisture content by Karl Fischer coulometric titration [35]. Subsequently, Gas Chromatography–Mass Spectrometry (GC-MS) was performed to measure the content of free and total glycerin following ASTM D6584 [36].

The FTIR analysis was carried out in a Perkin Elmer FTIR system device equipped with a mercury-cadmium-tellurium detector. The spectrum was recorded in Attenuated Total Reflectance (ATR) mode in Spectrum GX software with a spectral range from 4000 - 650 cm^{-1} at a resolution of 4 cm^{-1}. Approximately 5 mg of each biodiesel sample was placed in the sampling accessory. ASTM biodiesel standard sample was donated by (LIBBA, CIATEJ, Guadalajara Mexico).

2.4. Characterization of the adsorbents

Samples of sawdust, coconut coir, nutshell, rice husk and water hyacinth fiber as well as Magnesol® and Amberlite™ BD10DRY™ were analyzed by scanning electron microscopy. SEM analyses were performed on a JEOL JSM-IT300 LV electron microscope. To avoid charge effects, the samples were observed in low vacuum mode between 20 and 50 Pa at an acceleration voltage of 20 keV. The elemental chemical composition on each sample was determined using Energy-Dispersive Spectroscopy (EDS) by means of two Oxford-X-ManN Silicon Drift Detectors (SDDs) with an active area of 20 mm^2 and energy resolution of 127 eV, developed by Oxford instruments; these detectors were coupled to the microscope. The EDS spectra were acquired with the parameters of 20 keV and 300 s live time. Aztec software was used to analyze the spectral data. It is important to note that by using two SDDs, the noise-signal relation is diminished, and studies with high precision can be performed.

3. Results

3.1. Biodiesel characterization

Table 1, shows the different purification methods used in this study, as well as the results for the most relevant reference parameters concerning biodiesel quality. Among the bioadsorbents that reduced the acid value to levels below those established in the ASTM standard are water hyacinth fiber, rice husk, sawdust and coconut coir. The use of some of the bioadsorbents even proved to be more efficient than conventional methods, demonstrating their ability to remove free fatty acids associated with a high acid value. The acid value is important since it is a reference used to relate the amount of impurities and free fatty acids within the fuel as well as the relationship that exists in terms of oxidative stability [37].

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The free and total glycerin content is a very important parameter since it is related to both the efficiency of the reaction in terms of conversion and the efficiency of the purification process. **Table 1** indicates the most efficient bioadsorbents for free glycerin removal, which is related to the glycerin remaining in the biodiesel after transesterification. The best materials for glycerin removal are coconut coir and sawdust. It is also observed that in the case of the commercial methods, the amount of glycerin increases with respect to that in the unpurified biodiesel, which could be due to the existence of residual methanol, as well as mono-, di-or triglycerides that continue reacting in the presence of the catalyst.

In the case of total glycerin, which reflects the amount of oil that is unconverted or partially converted to biodiesel [38], the best bioadsorbents were coconut coir and sawdust, although they were not efficient enough to decrease the percentage of glycerol below the ASTM standard. The increase in total glycerin for both commercial adsorbents and some bioadsorbents may be due to the reversibility of the reaction process between esters (fatty and methyl).

Table 1, shows that sawdust is the only adsorbent that could remove water from the final biodiesel, reducing levels below the limit of the ASTM standard. In contrast, the other adsorbents increased the final water content. This result may be due to the humidity of the environment [39], as well as the inefficiency of the drying process of the bioadsorbents. As a result of this analysis, it can be verified that sawdust can remove impurities at low concentrations, such as glycerol, free fatty acids and water, in the same way as or even more efficiently than commercial adsorbents.

Table 1. Biodiesel quality analysis under different purification methods.

	Acid number (mg KOH g^{-1})	**Free glycerin (%)**	**Total glycerin (%)**	**Water content (%)**
Unpurified	0.671±0.001	0.0310±0.0001	0.0436±0.0008	0.0465±0.0000
Magnesol®	0.444±0.002	0.0067±0.000	0.1591±0.0012	0.0663±0.000
Amberlite™ BD10DRY™	0.668±0.001	0.0223±0.0001	0.1421±0.0317	0.0417±0.001
Water washing	0.465±0.000	0.001±.0.000	0.0086±0.0001	0.0313±0.001
Sawdust	0.459±0.001	0.0137±0.0001	0.1608±0.0006	0.0399±0.000
Coconut coir	0.483±0.001	0.0207±0.000	0.1808±0.0007	0.0551±0.000
Nutshell	0.528±0.001	0.0136±0.0001	0.1723±0.0002	0.0718±0.000
Rice husk	0.381±0.001	0.0211±0.0001	0.1765±0.0003	0.0694±0.000
Water hyacinth fiber	0.352±0.000	0.0642±0.0002	0.2175±0.0008	0.0927±0.000

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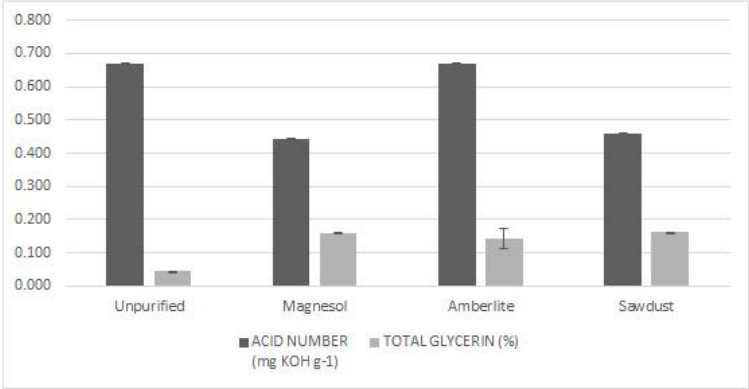
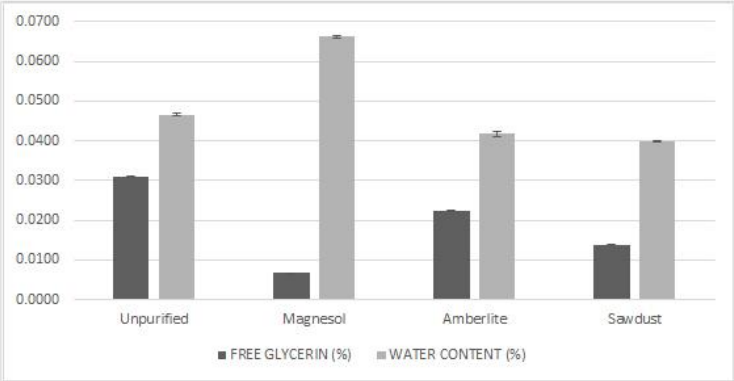
Figure 1a, shows the comparison between sawdust and the dry purification methods of biodiesel. There is a significant difference in the acid value reduction with respect to unpurified biodiesel for sawdust. The reduction was similar to that with Magnesol® but more efficient than that with Amberlite™ BD10DRY™. The total glycerin reduction is slightly significant between the commercial resins and sawdust. In Figure 1b, it is shown that sawdust was able to remove water from the unpurified biodiesel, being more efficient than Magnesol® but with a behavior similar to that of Amberlite™ BD10DRY™. For free glycerin removal, sawdust achieved a low reduction compared to that with unpurified biodiesel and Amberlite™ BD10DRY™ but was not as efficient as Magnesol®.



(a)



(b)

Figure. 1. Comparison between sawdust and conventional purification methods of biodiesel from WCO. (a) Acid Number (mg KOH g^{-1}) and Total Glycerin(%), (b) Free Glycerin(%) and Water content(%)

Table 2, lists different organic adsorbents or bioadsorbents that have been used by other authors as biodiesel purification methods. In this table, removal percentages have also been added for each of the quality parameters, such as the acid value, water content, total glycerol and free glycerol. In the case of the acid value, it is observed that the results obtained in this study are similar to those obtained by Gomes et al [40], who used different types of starches. Compared to other studies where the acid value increased after purification, in this study, the value decreased below 0.5 mg KOH g-1, the ASTM standard value. The water content results were similar to those reported by Gomes et al (2015) [40], who also observed an increase in the water content after purification due to the humidity of the environment, which could be transferred to the biodiesel during the purification process. It is observed that the results of total and free glycerin removal are not as significant as those

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reported in other studies, demonstrating that the bioadsorbents used may not be suitably effective in total glycerol removal.

Table 2. Comparison of various studies that used (bio)adsorbents in biodiesel purification.

Adsorbent	**Acid Number (mg KOH g^{-1})**	**Acid number reduction (%)**	**Water content (mg/kg)**	**Water removal (%)**	**Total glycerol (%)**	**Total glycerol removal (%)**	**Free glycerol (%)**	**Free Glycerol removal (%)**	Reference
Magnesol®	0.17± 0.01	48.4	500 ± 6	61.5	0.28	60.5	0.02	92.3	[17]
Silica	0.15 ± 0.01	54.5	700 ± 6	46	-	-	-		
Rice husk ash	0.13 ± 0.02	60.61	1292.13	42.97	0.46 ± 0.01	33.33	0.00420 ± 0.00003	46.97	[41]
Sugarcane bagasse	0.3 ± 0.021	14.3	677 ± 4.97	4.57* times	-	-	0.0053 ± 0.0000	82.3	[39]
Potato starch	0.106 ± 0.004	38.37	-	-	-	-	0 ± 0.001	100	[40]
Corn starch	0.104± 0.008	39.53	-	-	-	-	0.006 ± 0.003	95.34	
Rice starch	0.100 ± 0.001	41.86	-	-	-	-	0 ± 0.001	100	
Cassava starch	0.098 ±	43.02	-	-	-	-	0 ±	100	

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	0.002						0.001		
Eggshell	0.1375 ± 0.016	30.55	-	-	-	-	0.03	83.6	[42]
Oil palm empty fruit bunch	0.19 ± 0.02	36.6	120 ± 6	66.7	-	-	0.02 ± 0.008	13	[43]
Bentonite	0.1 ± 0.03	66.6	181 ± 5	72.4	-	-	0.015 ± 0.003	34.7	
Silica gel		66.6	138 ± 7	78.9	-	-	0.015 ± 0.010	34.7	
Banana peel	1.132	3*	1250	54	0.2	93	0.0068	99	[44]
Mushroom powder	1.27	4*	2000	27	-	-	-	-	
Purolite PD 206	0.99	2.5*	1000	63	0.18	93	0.0065	91	
Amberlite ™ BD10 DRY ™ and TULSION T-45 BD	1.132	3*	750	72	-	-	-	-	
Bentonite	0.22	24.14	809	5*	-	-	0.008	20.00	[45]
Coconut coir	0.319 ± 0.005	4.9*	--	--	--	--	--	--	[46]
Sawdust	0.459	31.5	-	11.2	0.322	11.29	0.015	11.76	This

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									study
Coconut coir	0.534	20.4	-	10*	0.342	5.78	0.017	0	This study
Nutshell powder	0.531	20.9	-	13.72*	0.358	1.38	0.0385	2.26* times	This study
rice husk	0.394	41.3	-	49.21*	0.367	1.1*	0.0425	2.5* times	This study
Water hyacinth fiber	0.352	47.5	-	77.45*	0.373	2.75*	0.043	2.53* times	This study

**Increased the value*

3.1.1. Biodiesel FTIR analysis

In Figure 2, the FTIR spectra from 650 – 4000 cm^{-1} are shown, corresponding to the samples of unpurified and purified sawdust biodiesel, as well as the ASTM biodiesel standard sample. This figure shows in the region from 1750 cm^{-1} to 1730 cm^{-1}, which indicate the existence of functional groups assigned to the carbonyl groups (C=O) typical of methyl esters. In the region of 700 – 800 cm^{-1}, bands corresponding to the methylene groups =CH and $–CH_2$ are observed [47]. In the fingerprint region, a band between 1200 and 1300 cm^{-1} appears; this band is attributed to $O\text{-}CH_2$ groups related to glycerol, which means there are triacylglycerides, diacylglycerides and monoacylglycerides present in the final biodiesel [48, 49]. For each spectrum, the absorption band from 2950 to 3000 cm^{-1} corresponds to the stretching vibrations of CH_3, CH_2, and CH bonds. The bands between 3200 and 3600 cm^{-1} correspond to OH groups, indicating the presence of glycerol and water [49, 50].

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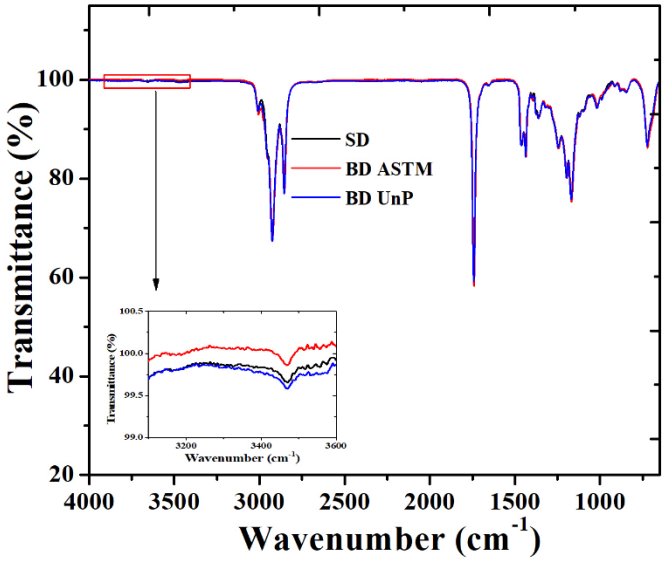

Figure 2. FTIR spectra of sawdust purified biodiesel, unpurified and ASTM sample.

3.2. Adsorbent characterization

3.2.1. Morphology of adsorbents

Figure 3 shows the surface morphology of sawdust, Magnesol® and Amberlite™ BD10DRY™, as revealed by SEM images. Analyzing Figure 3(A-B), an irregular surface and rough shape are observed, as well as small cavities in the woody structure [51-53] that can influence the adsorption of compounds such as glycerin, soaps and water from the transesterification process. The spherical shape of Magnesol® [41] is shown in Figure 3(C-D), revealing spaces between each adsorbent particle, which could diminish the surface contact with the biodiesel impurities. Figures 3E and 3F present the surface morphology of Amberlite™ BD10DRY™, which only shows a spherical and refined surface. In contrast to sawdust, the free spaces are more visible due to the size of the spheres, which affect the retention time between the resin and biodiesel.

The EDS results obtained during SEM analysis are shown in Figure 3(G); the elements with the highest content are C (61%) and O (39%) by weight of sawdust [52-54], in addition to the presence in a smaller proportion of Ca, K, Cu and Al. The appearance of Al may be because the samples were in contact with aluminum foil for storage, and Cu was part of the fixation support tape. Figure 3 (H, I) shows the characteristic composition of Magnesol® and Amberlite™ BD10DRY™, containing elements such as Si (32%) and Mg (10.79%) as well as C (64.3%) and S (13.43%), respectively.

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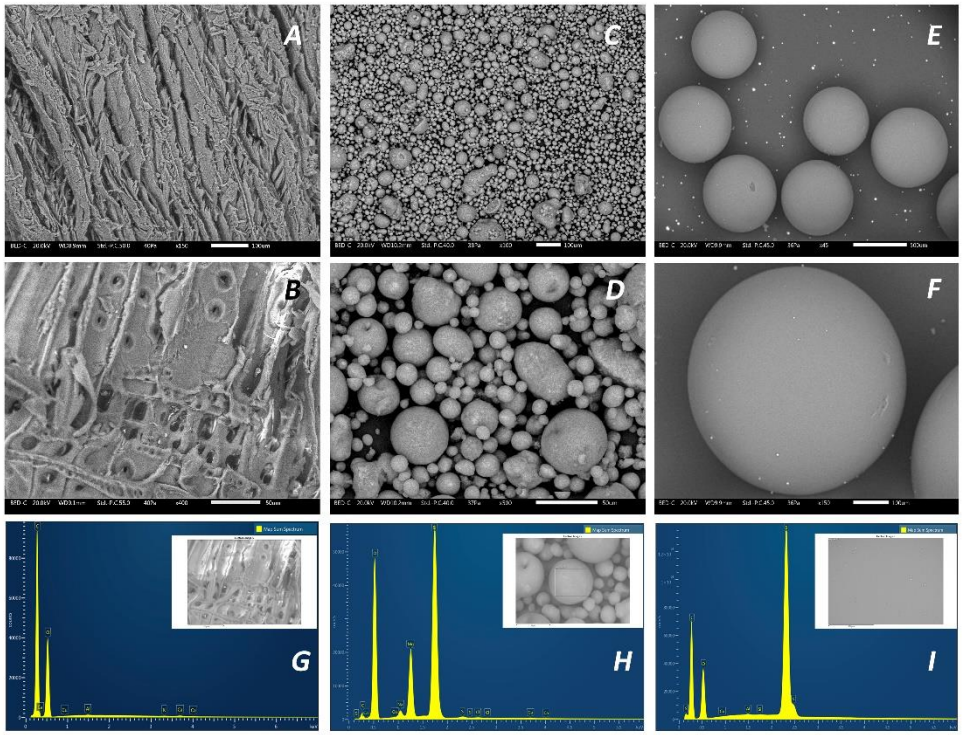

Figure 3. SEM Images and EDS analysis for Sawdust (A x150, Bx400, G), Magnesol® (C x100, Dx500, H) and Amberlite BD10DRY (E x45, Fx150, I)

4. Conclusions

Sawdust was the most effective adsorbent in purifying WCO biodiesel, statistically showing considerable adsorption of impurities. The efficiency of sawdust in removing impurities was similar to and, in some cases, better than that of conventional purification methods. The FTIR analysis shows that sawdust improves reductions in the proportions of OH functional group corresponding to glycerol. The more porous morphology of sawdust can facilitate diffusion of impurities as well as improve liquid-solid surface contact. Sawdust has the capacity to decrease the biodiesel acid value, as well as the water content and free glycerin content, with values below the ASTM standard. In the case of total glycerin, the results were not significant compared with those using Magnesol® and Amberlite™ BD10DRY™. We should not discard the use of the other bioadsorbents analyzed in this study. One of the main advantages of using sawdust as an adsorbent is that it is biodegradable and organic waste, ensuring that the biodiesel production system has a lower environmental footprint. While the results were promising in some cases, more research is needed to assess their potential as bioadsorbents.

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