

Environmental Technology

Reuse of Magnesol® for Purification of Biodiesel

A Purificação de Biodiesel com reaproveitamento de Magnesol®

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ABSTRACT

Purification of biodiesel with dry adsorbents is an alternative method, as it uses compounds that eliminate the need for water in the process without generating liquid effluent. Currently, there is little discussion about the reuse of different types of adsorbents and in this context, the present work presents an efficient method of purification and investigates the performance of the reuse of Magnesol® in the purification of biodiesel in order to generate less waste to the environment. Therefore, the Magnesol® used was washed with several solvents with different polarity characteristics. Among the solvents tested, THF is the best solvent for the purification of Magnesol® and, after optimizing the Magnesol® purification process, it was concluded the best conditions are three washes in solvent / adsorbent ratio 30:1 at room temperature, resulting in only 3.89% of the residual value of contaminants and being an alternative for biodiesel companies.

Keywords: Magnesol®; Biodiesel; Purification; Reuse

RESUMO

A purificação do biodiesel com adsorventes secos é um método alternativo, pois utiliza compostos que eliminam a necessidade de água no processo sem gerar efluente líquido. Atualmente, pouco se discute sobre o reaproveitamento de diferentes tipos de adsorventes e, nesse contexto, o presente trabalho apresenta um método eficiente de purificação e investiga o desempenho do reaproveitamento do Magnesol® na purificação do biodiesel de forma a gerar menos resíduos para o ambiente. Portanto, o

Magnesol® utilizado foi lavado com diversos solventes com características de polaridade diferentes. Dentre os solventes testados, o THF é o melhor solvente para a purificação do Magnesol® e, após otimizar o processo de purificação do Magnesol®, concluiu-se que as melhores condições são três lavagens na relação solvente / adsorvente 30:1 à temperatura ambiente, resultando em apenas 3,89% do valor residual dos contaminantes e sendo uma alternativa para empresas de biodiesel.

Palavras-chave: Magnesol®; Biodiesel; Purificação; Reaproveitamento

1 INTRODUCTION

In recent decades, humanity has been concerned with the continued instability of the international oil market. In addition, there has been increasing concern about environmental issues, and this involves the need for sustainable alternatives for the production of clean and renewable energy. Among these technological alternatives, biofuels are studied worldwide, and from this perspective, biodiesel plays a key role in replacing petrodiesel because it is biodegradable and fully renewable if it is made with bioethanol (Kucek *et al.*, 2007; Aydin, 214).

Biodiesel is chemically defined as alkyl monoesters of long chain fatty acids derived from renewable raw materials, such as vegetable oils, animal fats and recycled cooking oil. The most well-known process is a chemical reaction in which the triacylglycerides (TAGs) found in these oily materials (e.g., soybean oil) combine with an alcohol (methanol or ethanol) in the presence of an alkali catalyst (usually sodium hydroxide, alkoxides) to produce alkyl (biodiesel) monoesters and glycerin. As a co-product, glycerol has little or no fuel value, but its various industrial applications are critical to supporting process economics (Kucek *et al.*, 2007; Ferrari *et al.*, 205; Lôbo *et al.*, 2009).

However, in the transesterification process some undesirable by-products are generated which must be removed, such as unreacted tri, di and monoacylglycerols; methanol; catalyst; soap; glycerin and water (Faccini *et al.*, 2011). The purification of biodiesel can occur in two ways: dry or wet. The wet method requires water or solvent for the removal of the by-products generated but produces large volumes of effluents due to the need to use a lot of water in the process. However, purification with dry

adsorbents is an alternative method, as it uses chemical adsorbents that act to remove by-products without generating liquid effluent (Gomide, 1988).

In the last few decades, new natural and synthetic adsorbents have been studied to purify oils of different origins as well as frying oil (oil containing higher acidity and other contaminants that influence biodiesel production) (Alves *et al.*, 2016). Among the various adsorbents used for the purification of biodiesel are some silicates and mixtures of silicates with magnesium and aluminum oxides as well as various silicates formed by the fusion of lime, magnesium and aluminum oxides with diatomaceous earth (Faccini *et al.*, 2011; Araujo *et al.*, 2010; Turan and Yalcuk, 2013).

Magnesol[®] is the most widely used commercial adsorbent in the biodiesel purification process. This salt is the amorphous form of hydrated magnesium silicate, $MgO.nSiO_2.xH_2O$, on the surface of which are active sites that adsorb the compounds based on their dielectric constant and acidic and basic properties (Alves *et al.*, 2016). In biodiesel purification, it acts by removing free or even bound molecules of glycerol, soap, potassium, sulfur, residual methanol and traces of the catalyst. There is also evidence that the use of Magnesol[®] increases the stability of biodiesel in the oxidation process. The disadvantage of this is the high cost of the product (Sundus *et al.*, 2017).

In the literature, there are few studies on the reuse of different types of adsorbents, thus this work intends to demonstrate the reusability of Magnesol[®] and, consequently, the reduction of this adsorbent as residue. For this, biodeisels from frying soybean oil (possibility of using frying oil for energy purposes and reduction of environmental impacts) and virgin were used to verify the efficacy in both cases, as well as the cultivar soybeans because soy is responsible for more than 90% of biodiesel production in Brazil and the USA.

2 MATERIALS AND METHODS

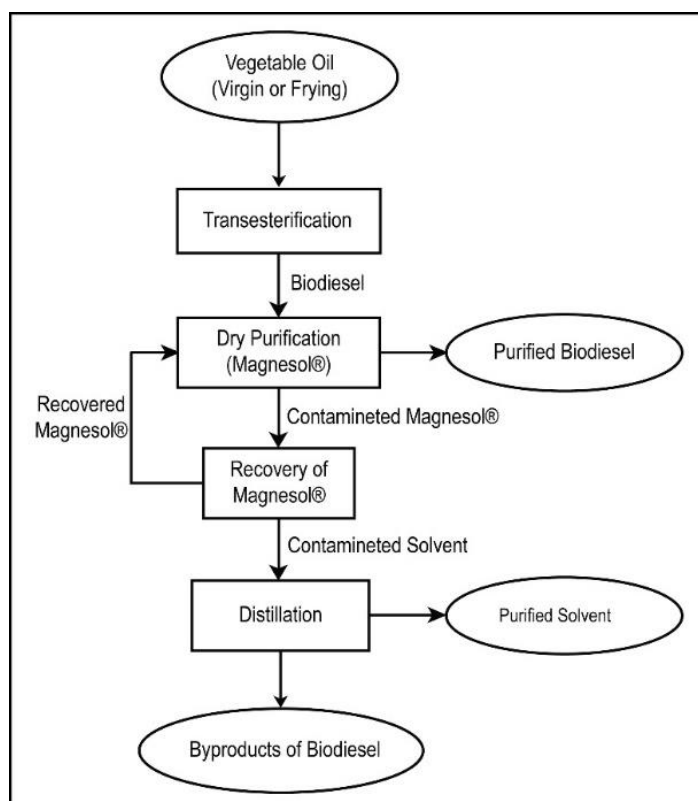
2.1 Materials

The materials used for the present work are virgin soy oil (SOYA), soy oil used in frying (donated by a local supplier) and Magnesol® (DALLAS GROUP). Reagents with analytical grade - 99.8% Methanol (ANIDROL), 85% Potassium hydroxide-KOH (VETEC), 99% Tetrahydrofuran-THF (VETEC), 99.18% Hexane (NEON), 99.5% Ethyl acetate (VETEC), 99.8% Dichloromethane (MERCK), 99.8% acetonitrile (VETEC) and 97% NaOH (VETEC) - were purchased from a local supplier and used without pre-treatment.

2.2 Methods

The methodology developed in this work is illustrated in Figure 1, which is described in two sections: biodiesel production and recovered of Magnesol®.

Figure 1 – Scheme of the Magnesol® purification process



2.2.1 Biodiesel

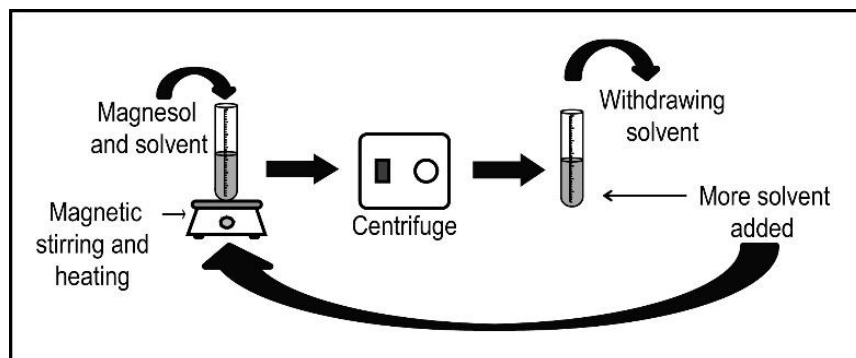
First, on a bench scale biodiesel plant, 10 L of biodiesel was produced through a batch process from soybean frying oil and virgin soybean oil. After, the oils (virgin and frying) had their acidity corrected with KOH, as they were not in compliance with the requirements of the National Agency of Petroleum, Natural Gas and Biofuels (ANP). Posteriorly, the two biodiesels were produced using the same conditions, through the alkaline transesterification process with KOH 1% (w/w). Thus, the conditions used in this process were: 6:1 methanol/oil ratio, 65 °C temperature and 1.5 h reaction time. The reactions occurred under constant agitation and constant oil pumping in the reactor for better homogenization. After that, the biodiesels sat for approximately 30 h before the separation of glycerin was carried out, and after which the excess methanol was removed by heating at 65 °C for 1 h (Kucek *et al.*, 2007).

Purification of biodiesel was performed by the dry route with 1% w/w Magnesol[®]/biodiesel, heated at 50 °C under constant stirring for 30 minutes. After this time, the biodiesel was vacuum filtered and the adsorbent was taken for purification (Farag and El-Anany, 2006).

2.2.2 Recovered of Magnesol[®]

The magnesol extracted from the biodiesel purification process was purified according to the methodology presented in Figure 2, to which 1g of contaminated magnesol was placed in a conical flask and 11.2 ml THF was added (30:1 ratio (w/w)). The system was stirred for 30 min, subsequently being centrifuged for 3 minutes at 4000 rpm for decantation of Magnesol[®] and removal of the supernatant. This process was repeated three times, and at the end the product was dried at 70 °C for 24h.

Figure 2 – Magnesol® purification process



2.2.3 Characterizations

a) Characterization of Purified and Unpurified Biodiesel

Some parameters of the biodiesels from the virgin soybean oil and frying oil were analyzed in order to compare the efficiency of the purification with virgin and recovered Magnesol®. The main parameters were established in accordance with the standards described below.

Determination of acid value

Based on standard EN14104, 2.5 g of biodiesel was placed in a 250 ml erlenmeyer flask and 50 mL of ethyl ether/ethanol 2:1 was added, stirring until complete dissolution. Two drops of phenolphthalein solution - 1% in ethanol - was added, and it was titrated with a standardized solution of 0.1 M NaOH. The analyses were performed in triplicate and the results were expressed as mg KOH/g sample (Alves *et al.*, 2016).

Determination of the glycerol

For the determination of glycerol ester contents, about 250 mg of biodiesel was transferred into a glass balloon and 5 ml methyl heptadecanoate - internal standard solution (C₁₇; 10 mg/ml) - was added. Approximately 1.0 µl of the prepared solution was injected into a GC-14B Shimadzu gas chromatograph, equipped with a Flame Ionization Detector detector and a capillary column (DB-5MS, 30 m × 0.25 mm ID, THK 1µm, Agilent). Nitrogen gas was used as the mobile phase. The concentration of methyl esters

in biodiesel samples was calculated using the GC data by the method presented in EN14103:2003(E).

Determination of the flash point

The flash point was determined according to ASTM D 93 which limits its minimum value to 100 °C. The equipment used was a FLASHPOINT TESTER - HFP339 - Pensky Martens.

b) Analysis of the Purified and Unpurified Magnesol®

Elemental Analysis

The total carbon percentage (%TC) and total nitrogen percentage (%TN) was performed analysing the samples in the TruSpec CHN non-dispersive elemental analyser, mark LECO®, equipped with an infrared detector for carbon and hydrogen, and a thermal conductivity detector for nitrogen. For the determination, a small amount of the sample was used, which ranged from 50 mg to 100 mg (± 0.01). The samples were weighed into thin sheets of tin and taken directly to the equipment. The samples were burned under a 6.0 oxygen (99.9999 %) atmosphere with 10 lpm flow and 950 °C LECO® according to the ASTM D5291-16. The test was performed in triplicates (Ramos *et al.*, 2015).

Field Emission Scanning Electron Microscopy (FEG-SEM)

The surface morphology of pure and recovered Magnesol® was studied using a FEG-SEM, FEI Inspect F50 equipment, and the samples were coated with a thin layer of gold. The dimensions were calculated using the program Image J (n = 20).

Fourier Transform Infrared Spectroscopy (FTIR)

FTIR analysis was performed using a Perkin Elmer Instruments Spectrum One FTIR Spectrometer, and spectrum acquisition was performed with the Attenuated Total Reflectance (ATR) sample attachment in the 650 to 4000 cm^{-1} wave number range.

Thermogravimetry analysis (TGA)

The thermal stability was evaluated by thermogravimetric analyses using the SDT- Q600 model (TA Instruments) in the range 25 - 800°C with a heating rate of

20 °C.min⁻¹ under nitrogen. The weight of the sample was approximately 7.8 mg, and the analysis was carried out in triplicate.

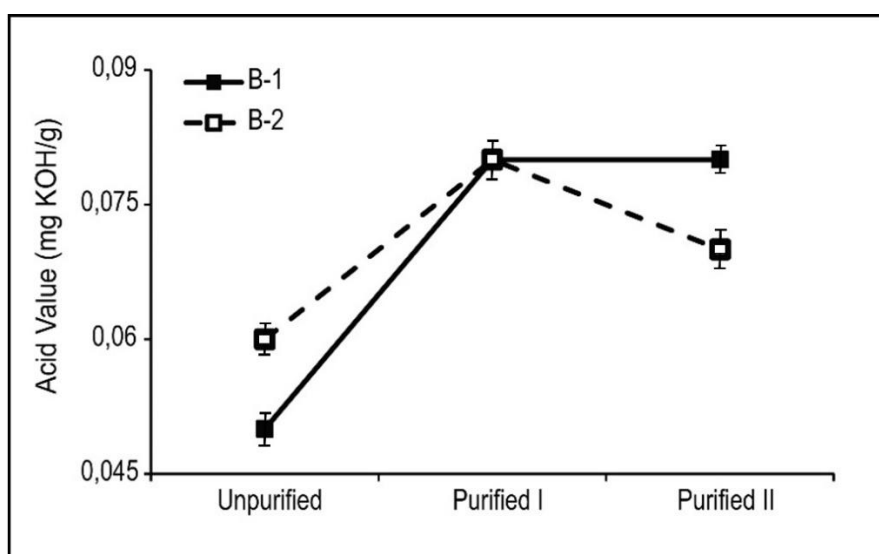
3 RESULTS AND DISCUSSION

3.1 Biodiesel Purification

1) Acid value

Even with the increase in the acidity index after the treatment of biodiesel with Magnesol®, all values of the acidity index are lower than that established by the ANP standard, where the document cites that only biodiesel with an acidity index less than 0.5 mg KOH/g sample can be marketed. Figure 3 shows the acid value of the unpurified biodiesels and biodiesels purified with virgin (purified I) and recovered Magnesol® (purified II), where occurs increase acids values due to the affinity of Magnesol® adsorbing basic compounds, such as soap and catalyst residue (KOH) (Faccini *et al.*, 2011).

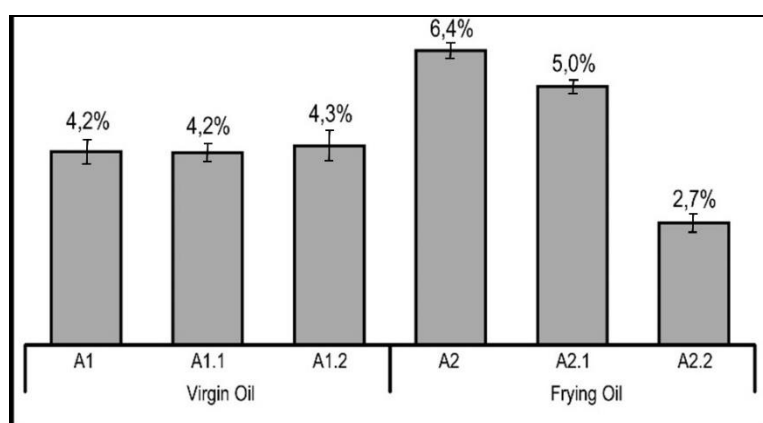
Figure 3 – Acid value of biodiesel: unpurified biodiesel, purified with virgin Magnesol® (purified I) and purified with recovered Magnesol® (purified II). B-1 and B-2 represents virgin oil and frying oil, respectively



2) Glycerol amount

Figure 4 shows the amount of glycerol in the biodiesel before and after the use of Magnesol[®], and it is observed that the used of Magnesol[®], and the reuse do Magnesol purified (originally used in the purification of biodiesel from virgin soybean oil), was not effective to purification of biodiesel, because the amount of glycerol found in the biodiesel is within the norms (below 5%). But the Magnesol[®] used in the purification of biodiesel from the frying oil, it can be reused with good efficiency, since the amounts of glycerol found in biodiesel A2.1 (5.03%) and A2.2 (2.7%) are within the values recommended by the legislation, showing a decrease in the amount of glycerol when compared to unpurified biodiesel (A2 - 6.41%). Probably, this decrease in glycerol observed in biodiesel after purification with recovered Magnesol[®] is due to the characteristics of the remaining compounds in the THF-purified Magnesol, because it is demonstrated in the literature that residual compounds with polar and nonpolar characteristics found in residues of the biodiesel and remaining in the Magnesol have greater affinity with glycerol, aiding in the purification of the biodiesel (Panagiotopoulou and Tsimidou, 2002; Wretensjo and Karlberg, 2002).

Figure 4 - Quantity of glycerols in unpurified and purified biodiesel: virgin oil (A1) and subsequently purified with virgin Magnesol[®] (A1.1) and recovered Magnesol[®] (A1.2); frying oil (A2) and subsequently purified with virgin Magnesol[®] (A2.1) and recovered Magnesol[®] (A2.2)



3) Flash point

In three fractions of biodiesels synthesized from virgin oil (A1) and subsequently purified with virgin Magnesol® (A1.1) and recovered Magnesol® (A1.2), flash point analyses were performed and the results obtained were 169°C, 165°C and 167°C, respectively. The results indicate that all fractions of purified biodiesel have values accepted by ANP, demonstrating good reusability after passing through the purification process. The same flash point analyses were performed with three fractions of biodiesel synthesized from frying oil (A2) and purified with virgin Magnesol® (A2.1) and recovered Magnesol® (A2.2), yielding results of 159 °C, 158°C and 160 °C, respectively. These results are in accordance with ANP 07/2008 standards that establish a flash point of 100°C as the minimum value to be used (Lôbo *et al.*, 2009).

Magnesol® Purification

In Table 1, processes 1 and 3 correspond to the solvents used to purify the Magnesol® used in the purification of biodiesel derived from virgin oil, while processes 2 and 4 correspond to the solvents used to purify the Magnesol® used in the purification of biodiesel derived from frying oil. Results of the elemental analysis corresponding to the related process showed the best results for the Magnesol® used in the purification of biodiesel from virgin oil. Consequently, after process 4, it was decided to optimize the adsorbent purification process only for the Magnesol® used in the purification of biodiesel derived from virgin oil, and later, to apply this to the Magnesol® used in the purification of biodiesel derived from frying oil (Table 1).

The elemental analysis of virgin Magnesol® did not show any percentage of carbon, thus all carbon found after the purification of the adsorbent is an impurity derived from biodiesels. In the Magnesol® recovery process, the parameters optimized were reaction time, solvent type, solvent ratio and temperature. Above is Table 1 with the 19 Magnesol® recoveries carried out in this work. As the goal of this analysis was to determine the amount of carbon, the reactions that were demonstrated to be more efficient were reactions 9, 10, 14, 15 and 16.

Table 1 – Optimization of the Magnesol® purification process and amount of carbon found in samples

R.	Solvent	Tem p (°C)	Proportio n Solv. (g:g)	Stage *3x (g)	Reaction Time (min)			Carbon Amount (%)*
					t ₁	t ₂	t ₃	
1	NaOH	r.t	20:1		90	-	-	21.03
2	NaOH	r.t	20:1		90	-	-	18.18
3	Hexane	r.t	20:1		90	-	-	8.69
4	Hexane	r.t	20:1		90	-	-	11.72
5	Hexane	r.t	15:1	*3x 5	30	30	30	9.74
6	Hexane	50	15:1	*3x 5	30	30	30	9.20
7	Ethyl acetate	r.t.	15:1	*3x 5	30	30	30	5.72
8	Ethyl acetate	50	15:1	*3x 5	30	30	30	5.66
9	THF	r.t	15:1	*3x 5	30	30	30	4.31
10	THF	50	15:1	*3x 5	30	30	30	4.40
11	Ethanol	r.t	15:1	*3x 5	30	30	30	6.13
12	Dichloromethane	r.t	15:1	*3x 5	30	30	30	9.15
13	Acetonitrile	r.t	15:1	*3x 5	30	30	30	9.79
14	THF	r.t	30:1	*3x 10	30	30	30	3.89
15	THF	r.t	45:1	*3x 15	30	30	30	3.83
16	THF	r.t	60:1	*3x 20	30	30	30	3.75
17	THF	r.t	15:1	*3x 5	20	20	20	8.93
18	THF	r.t	15:1	*3x 5	10	10	10	8.14
19	THF	r.t	15:1	*3x 5	5	5	5	10.92

R.: Optimization process; r.t: Room Temperature; *3: number of times

Magnesol® purification was performed using several solvents of different polarities in order to verify the influence of the dielectric constant in the purification of biodiesels, because it is known that the by-products of biodiesel synthesis have polar and nonpolar characteristics. Knowing the dielectric constant (μ) of the solvents, it was expected that the solvents with a higher degree of polarity, such as H₂O (used to make the solution of NaOH), $\mu = 80$; CH₃CN, $\mu = 37$ and ethanol, $\mu = 30$, would be the least effective. The CH₂Cl₂, although showing $\mu = 9.1$, very close to μ of THF ($\mu = 7.5$), also does not provide satisfactory results, perhaps because it did not show hydrogen interactions with the by-products generated in biodiesel synthesis. Ethyl acetate ($\mu = 6.02$) is observed to have purification rates very close to THF because ethyl acetate has a μ slightly smaller than THF and it has the possibility of having hydrogen interactions with compounds withdrawn from the process of biodiesel purification.

In relation to the effect of the temperature during the reaction, it is observed that an increase in temperature (25 to 50°C) in the purification process of Magnesol® does not contribute significantly to a better result. This can be seen in the reactions using hexane (reactions 5 and 6) and ethyl acetate (reactions 07 and 08) in which a small decrease in impurities (amount of C, 0.54% and 0.06%, respectively) occurred. In addition, when comparing the THF solvent at 25 and 50 °C a slight variation in the amount of carbon (4.31 and 4.44%, respectively) is seen.

Another parameter analyzed in the reaction optimization was the number of extractions. When comparing reactions 04 and 05, it is evident that when three extractions are used (reaction 05) instead of a single extraction (batch), the purification of Magnesol® is more effective, even using 25% less solvent. Subsequently, by comparing the amount of solvent, it was observed that when increasing the amount of THF in the proportions of solvent/adsorbent from 15:1 (reaction 09) to 30:1 (reaction 14), Magnesol® was obtained with a C quantity 0.42% lower, and when testing the purification of Magnesol® by increasing the proportion of solvent/adsorbent to 45:1 and 60:1 (reactions 15 and 16, respectively), a very small decrease in impurity was observed, 0.06 and 0.08%, respectively, showing that above the solvent/adsorbent ratio

30:1, there is no considerable gain in impurities reduction. And finally, when the contact time between the solvent and Magnesol[®] was decreased, a decrease in the purification efficiency of the solvent was observed, indicating that the solvent and the Magnesol[®] are required to remain in contact for 30 minutes at each step during the washing process.

Characterization of Purified and Unpurified Magnesol[®]

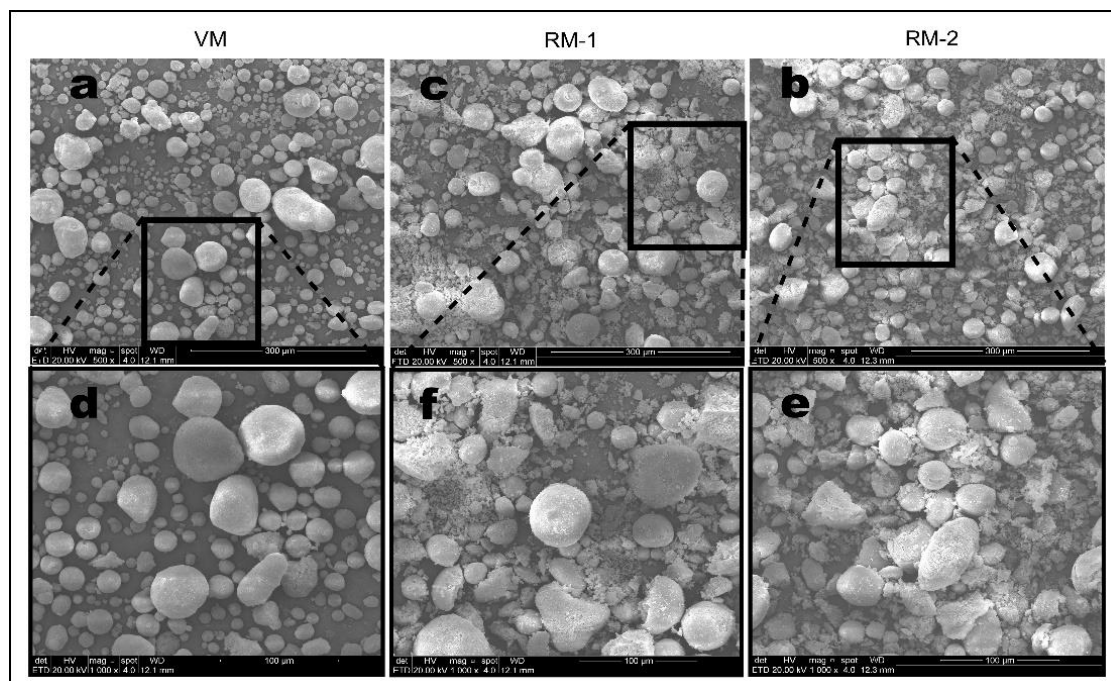
In order to justify the efficiency of the recovery of Magnesol[®] (purified), SEM-FEG (Scanning Electron Microscopy-Field Emission Gun) analyses were performed, in which it was possible to compare the morphology and particle diameter. In addition, through the FTIR analysis it was possible to compare which chemical groups the Magnesol[®] can retain in its structure as well as evaluate the efficiency of the recovery process. Also, thermogravimetric analysis was performed, in which the different degradation temperatures of the virgin and recovered Magnesol[®] can be compared.

1) SEM-FEG

Figure 5 shows the SEM-FEG of virgin Magnesol[®] and Magnesol[®] recovered from the purification of biodiesel derived from virgin and frying soybean oils. From the micrographs obtained by SEM-FEG, the particle diameters of **(a and d)** Pure Magnesol[®], **(b and e)** Magnesol[®] used to purify biodiesel derived from frying oil and **(c and f)** Magnesol[®] used to purify biodiesel derived from virgin oil were calculated. A predominant microporous morphology was observed in the analyzed materials as it was also highlighted by Facicini *et al.*, 2011.

It is noted that the process of purification and recovery causes breaking of part of the Magnesol[®] structure, probably due to the agitation and temperature of these processes. This corroborates with previous results regarding the reuse of Magnesol[®] because the structure remains porous and the breaking of part of the spherical structures increases the contact surface, compensating for the probable decrease in Magnesol[®] efficiency that can occur with the small amount of contamination that remains after the recovery process.

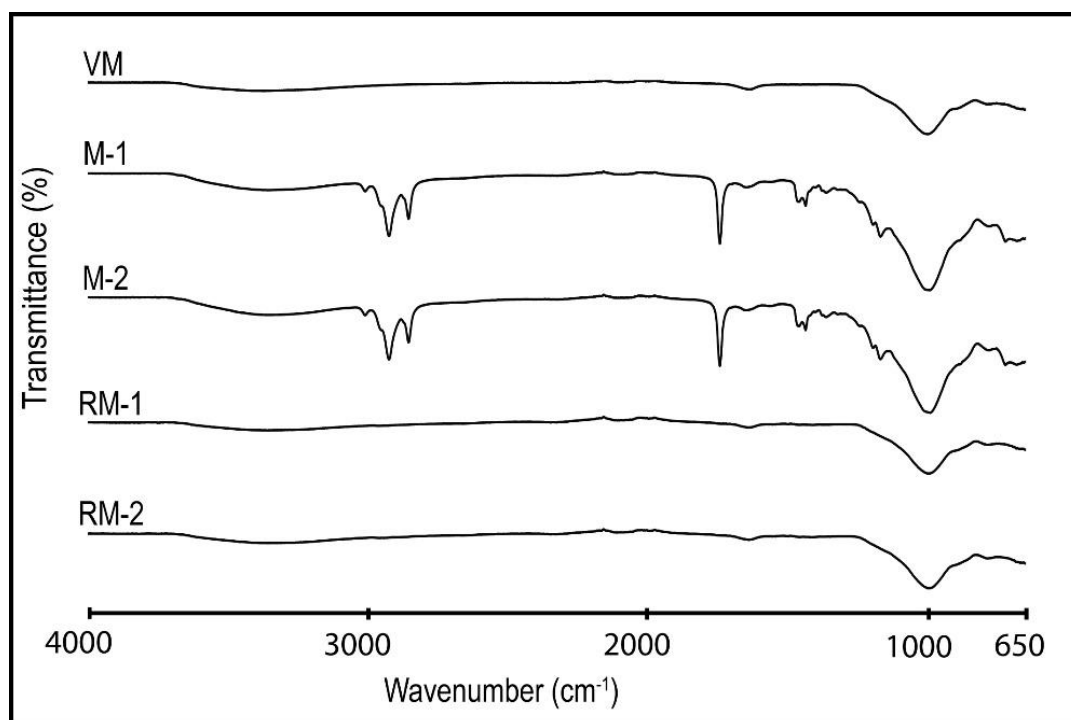
Figure 5 – Micrographs, magnification of 600x: (a) Virgin Magnesol®, (b) Recovered Magnesol® used in the purification of biodiesel from frying oil and (c) Recovered Magnesol® used in the purification of biodiesel from virgin oil. The images in (d), (e) and (f) show micrographs with magnification of 1000x for the Magnesol® shown in (a), (b) and (c), respectively



2) FTIR

Comparing the first spectrum with the RM-1 and RM-2 spectra, not difference is noted, specifically, the recovered Magnesol® (RM-1 and RM-2 spectra) demonstrates compatibility with the virgin Magnesol®. It may be further noted that the M-1 and M-2 spectra show some characteristic bands having absorptions between 3000 and 2800 cm^{-1} (C-H), compatible with the presence of carbon sp^3 (carbon with just sigma bonds (σ)). The bands between 1820 and 1630 cm^{-1} show carbonyl functions of C=O and the region 1599 to 1500 cm^{-1} represents sigma (σ) and pi (π) bonds, i.e., unsaturated bonds between C=C (Lopes and Fascio, 2004; Silverstein and Bassler, 1962). With this, it can be observed that the recovered Magnesol® spectra (RM-1 and RM-2) do not contain the infrared absorption bands found in the M-1 and M-2 spectra, and thus, resemble the virgin Magnesol® FTIR spectrum (Figure 6).

Figure 6 – FTIR Spectra of virgin and recovered Magnesol[®]. Virgin Magnesol (VM); Magnesol[®] used in the biodiesel purification: virgin soybean oil (M-1) and frying soybean oils (M-2); Orecovered Magnesol[®]: virgin soybean oil (RM-1) and frying oil (RM-2)

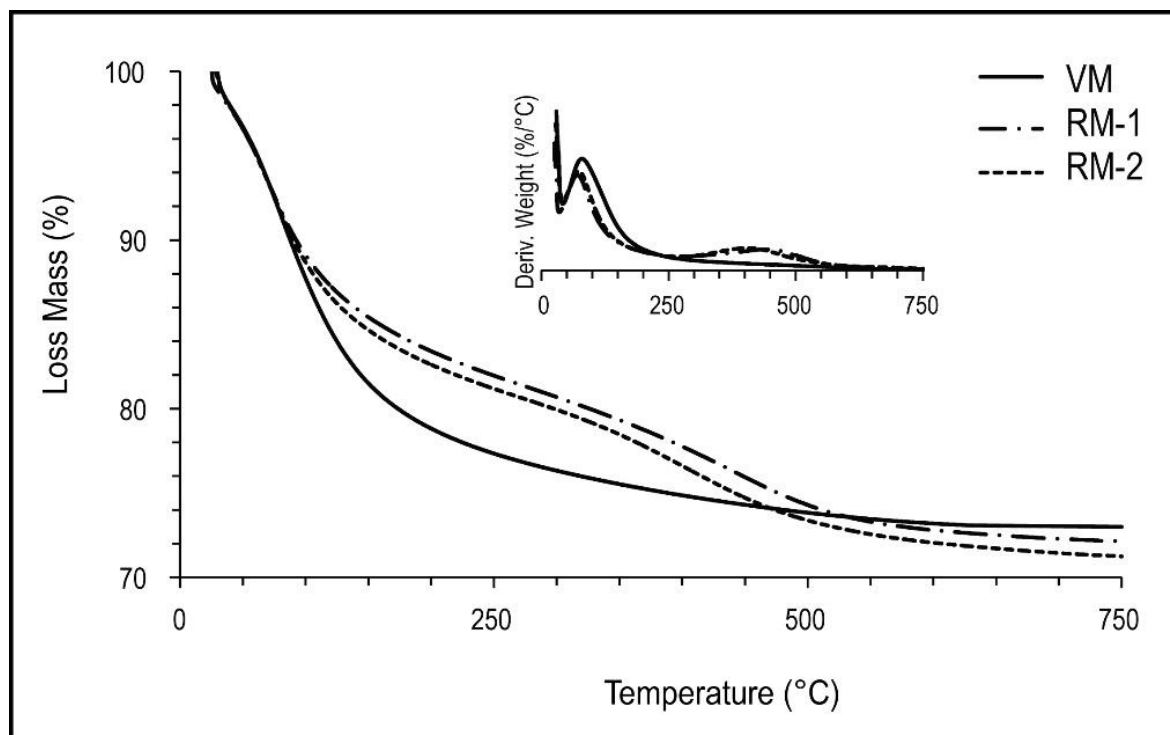


3) Thermal gravimetric analysis

From the thermograms in Figure 7, it is observed that the degradation of virgin Magnesol[®] (VM) occurs in a single step. However, the degradation of the recovered Magnesol[®] (RM-1 and RM-2) occurs in two steps, because contaminants are present in the purified biodiesel which the THF solvent was not capable of removing. This result was proven by the presence of carbon in the elemental analysis.

As shown in Figure 7, VM, RM-1 and RM-2 experience maximum mass loss with temperature at 83 °C, 76 °C and 74 °C, respectively. This result is expected due to the volatile components and water steam beginning to be released in this first range of temperature starting at 42 °C, 42 °C and 35 °C and finishing at 230 °C, 266 °C and 266 °C, respectively. In addition, a second maximum mass loss temperature was observed for RM-1 and RM-2 (408 °C and 446 °C, respectively); mass loss in this temperature range was expected due to the small amount of carbon found in the products.

Figure 7 – Thermal gravimetric analysis (TGA) and differential thermal gravimetric analysis DTGA graphs of virgin Magnesol® (VM), recovered Magnesol® used in the purification of biodiesel from virgin soybean oil (RM-1) and recovered Magnesol® from the purification of biodiesel from frying oil (RM-2)



4 CONCLUSIONS

Through the analysis of total carbon, it can be observed that the methodology used in the purification of Magnesol® is satisfactory, as approximately 3.90% carbon was found after the washing process with THF. This was proven through infrared analyses which showed the absence of signals around 2900 and 2700 cm^{-1} in the purified Magnesol® as well as a decrease in the signal at 1000 cm^{-1} , resembling virgin Magnesol®.

After the Magnesol® was purified with THF (r.t., 30:1 solv./Magnesol®; 3 stages, duration of 10 min. for each wash), we observed different behaviors depending on whether it was used with virgin oil or frying oil. For the Magnesol® used in the purification of biodiesel from the virgin soybean oil, we found it can be used at most

two times without losing its efficiency, whereas the Magnesol® used in the purification of biodiesel from soybean oil used in frying can be used at least 2 times, as no loss of efficiency was observed. These results were verified in the glycerol analyses presented in this study.

After the acidity, glycerol, flash point and density tests, it was found that biodiesel after being purified with Magnesol® is in good condition to be used in industry with less glycerol, the main impurity.

ACKNOWLEDGMENT

LOR - Laboratory Organometallic and Resins – PUCRS; LASPI - Systems Laboratory and Industrial Processes; PET-Chemistry / Program of Education Tutorial in Chemistry – PUCRS and Braskem SA.

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How to quote this article

COELHO, B. N.; NOGUEIRA, B. M.; MARTINS, F. L.; ROCHA, P. R. da; CARDOSO, V. de M.; RAMOS, A. da S.; PIRES, J. P.; SANTOS, C. A. B. dos; SEFERIN, M.; LOUREGA, R. V. Reuse of Magnesol® for Purification of Biodiesel. **Revista Eletrônica em Gestão, Educação e Tecnologia Ambiental**, Santa Maria, v. 26, e7, 2022. Available from: <https://doi.org/10.5902/2236117065590>