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ASSESSMENT OF THE PHYSICOCHEMICAL SUITABILITY OF OILS AND FRYING FATS RESIDUALS FOR BIODIESEL PRODUCTION

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Abstract- *Frying food is a quick and tasty way of cooking, but generate a large amount of residual frying oils and fat (RFOF). However, these residuals can be used for making soap, animal food, and biodiesel. Companies have been increasingly interested in developing green solutions to waste management that control its generation, collection, recycling, and disposal. With the increase of biodiesel demand, RFOFs are increasingly used to produce biofuel instead of soap. However, unwanted residuals of RFOFs need to be cleaned and their physical-chemical characteristics need to be analyzed before they can be used for biodiesel production. RFOF was submitted to a Liquid-Liquid Extraction (LLE) to remove highly oxidized compounds and short-chain free fatty acids. Here, we evaluated the physicochemical characteristics and filtered RFOFs to remove waste to improve biodiesel production. We found that that the liquid-liquid extraction (LLE) and filtration using adsorbent decrease first-order oxidized compounds, like hydroxy-dienone, and decrease water content. Those are key parameters to assess quality of raw material for biodiesel production..*

Keywords *– Characterization. Gas Chromatography. Recycling. Waste.*
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1. Introduction

Residual frying oils and fats (RFOF) do not have a management policy to advise its proper disposal. They are a hazardous waste because they can damage the environment if dumped improperly, causing death of microorganisms and vegetation, and soil infertility. If dump into sewers, they can clog pipes and decrease oxygen concentration of rivers and lakes, impacting aquatic organisms (GOMES et al., 2013; GOUVEIA, 2012). Despite its impact, a small amount of RFOF is properly collected, stored, and recycled that need commitment from governments, business men, and people in general(Camargo & Carvalho (2014). However, this remains a challenge because in order to be successful, a management policy requires the population to be aware of the negative impacts of improper disposal of RFOF. But if accomplished, the reverse logistics of RFOF can benefit everyone.

Currently, many initiatives are trying to reverse this situation in Brazil. For example, the sewage treatment company of Goiânia (Saneamento de Goiás S.A.) has a program called de olho no óleo ("keeping an eye on oil") that offer a discount on the water bill for consumers who recycle RFOF. A company called Ecológica of Rio Grande do Sulcollects and recycles used vegetable oil. This company maintains containers around the city to store RFOF and also collects and transports RFOF to recycling plants. Resolution #275 of the Brazilian National Environmental Council (CONAMA) order that solid waste recycling should be encouraged, facilitated, and expanded through environmental education. Such initiatives would reduce consumption natural resources and raw materials.

RFOFs can be used to produce biodiesel, glycerin, dyes, putty, and to generate electricity by burning boiler. However, they are most commonly used to make soap and animal food (KAGAWA et al., 2013; YAAKOB et al., 2013). Conversely, the need to produce low-cost renewable fuels fostered the use of RFOFs for biodiesel production. Previous studies (KAGAWA et al., 2013; UDDIN et al., 2013; UZUN et al., 2012) showed that biodiesel from RFOFs meet the physical and chemical standards required by regulatory agencies. As a result, previous studies have tested

alternative materials in order to make biodiesel production energetically efficient and cheap. For biodiesel from RFOFs to reach the standards required by law, it is necessary to improve their quality. Here, we evaluated and provide a protocol for RFOFs treatment in order to adapt it for biodiesel production.

2. Materials and Methods

2.1 Collection and treatment of RFOFs

We obtained RFOFs from a restaurant of Anápolis, Goiás, central Brazil. Every week, the restaurant uses 32 L of refined soybean oil for frying foods and stored the RFOFs in 100-L metallic tanks. We removed20L from this tank for treatment and analysis, which were stored in high-density polyethylene plastic vials. Then, we filtered the RFOFs using glass funnel and quantitative filter paper (Whatman® 42, 240 mm diam.) to remove food remains and other particulates. Afterwards, RFOF was submitted to a Liquid-Liquid Extraction (LLE) to remove highly oxidized compounds and short-chain free fatty acids. LLE consisted in mixing RFOFs with distilled water (1:1 v/v) in an Erlenmeyer and then placing it in an incubator (Nova Ética, Model 430RD) for 1 h at room temperature and stirring at 180 rpm. Using water in LLE improves RFOFs quality, since it decreases both the acidity and peroxide indices (AZEREDO, 2012). This procedure has a low cost, since it does not require much energy or reagents. To remove the water, the mixture was placed in separatory funnel and then distilled in a round-bottom flask at 90 °C under reduced pressure for about 2 h, or until water droplets disappeared from the bottom.

2.2 Physico-chemical characterization

Assessing the composition of fatty acids of RFOFs allows estimating key properties for biodiesel quality, such as oxidative stability, and cold filter plugging point(BORGES et al., 2014; SILVA, 2011).We carried out the physicochemical characterization of RFOFs using the following tests: Iodine Index (following EN 14111:2003), Peroxide Index (AOCS Cd 8-53:1993),Kinematic viscosity (NBR 10441:2007), Acidity Index (ASTM D664:1989), saponification value (AOCS Cd 3-25: 1993), Water content (ASTM D6304:2007), density (ASTM D 4052:2015), oxidative stability (EN 14112:2003), amount of chemicals (NBR 15553:2015 expanding for the analysis of other metals), fatty acid profile, presence of mono-, di-, and triacylglycerides, and oxidation products.

2.2.1 Fatty acid profile analysis

The fatty acid profile analysis is important to estimate biodiesel quality from any grease raw material and determine the efficiency of biodiesel production process. The percentage and type of fatty acid composition is related to plant species and its growing conditions. We used High Resolution Gas Chromatography (Shimadzu-2010 Plus) coupled to a Flame Ionization Detector(HRGC-FID)at 350 °C and DB wax capillary column (30 m x 0.25 mm x 0.25 μm). The carrier gas was Helium at linear velocity of 34.9 cm.s-1. The initial oven temperature was 70 °C, heated at 10ºC.min-1up to 240 °C and maintained at this temperature for 13 min. Then, the oven was heated again to 250 °C at 5ºC.min-1 and maintained at 250 °C for 20 min. The split injector was maintained at 350 °C under 10:1 for a injection volume of 2 μL.

2.2.2. Quantification of free fatty acids, mono-, di-, and triglycerides and oxidized products

We used a mobile phase A composed of 20% water and 80% acetonitrile and a mobile phase B composed of 50% acetonitrile, 37.5% isopropanol, and 12.5% hexane. The wavelengths monitored were 210, 235, and 270 nm, respectively for the analysis of intact acylglycerol and products from oxidative degradation, such as hydroxyperoxydiene and cetoperoxydiene. All analyzes were performed in triplicate in a HPLC (DAD Agilent 1220 Infinity LC). The column used was a C18 Zorbax Plus Ellipse (50 cm x 4.5 mm x 1.8 μm). Oven temperature was 40 °C and gradient from 0 to 8 minutes with 100% mobile phase A. Then, 8 to 10 minutes until a linear variation of the mobile phase reached 100% B, which was maintained isocratic up to 20 minutes. Then, the column was reconditioned with the mobile phase A for 5 minutes. The mobile phase flow rate was 0.8 mL.min-1and the injected volume was 20 μL, containing 5 mg of RFOFs diluted in 25 mL of acetonitrile.

Chromatographic analysis measures free fatty acids, mono-, di- and triacylglycerides, and alkyl esters of fatty acids. Their order of elution is dictated by their polarity, eluting first the polar compounds (free fatty acids and monoacylglycerides) during the first 10 minutes, followed by medium polarity compounds, from 10 to 12 minutes, which are ethyl esters fatty acids and diacylglycerides, and the less polar compounds after 12 minutes, which are triacylglycerides.

3. Results and discussion

3.1 3.1. Quantification of free fatty acids, mono-, di- and triacylglycerides, and oxidized products

The RFOF may contain unsaturated fatty acids prone to accelerated oxidation during storage and deep frying, producing polymerized compounds. The main oxidation products from RFOF are hydroxyperoxydiene, cetodiene, and hydroxydiene (ALBERICI et al., 2012; BEZERGIANNI; CHRYSIKOU, 2012; JAIN; SHARMA, 2013; LEME et al., 2012; MORALES et al., 2012a; NATARAJAN, 2012).

Both refined soybean oil (Fig. 2A), RFOF untreated (Fig. 2B), and RFOF after treatment (Fig. 2C)are mostly composed of triacylglycerides, with a smaller amount of diacylglycerides. This indicates that this sample has the target components for biodiesel production

(BEZERGIANNI; CHRYSIKOU, 2012). There was also a small amount of fatty acids in untreated RFOF, and less so after treatment. This result proves the effectiveness of the procedure. We did not detect free fatty acids in refined soybean oil.

The analysis at 235 nm (Fig. 3) shows that the primary oxidation products, i.e., hydroxyperoxydiene and hydroxydiene are absent in refined soybean oil (Fig. 3A), but present in large amounts in untreated RFOF (Fig. 3B), and lower concentration after treatment (Fig.3C), demonstrating the effectiveness of oxidized components removal.

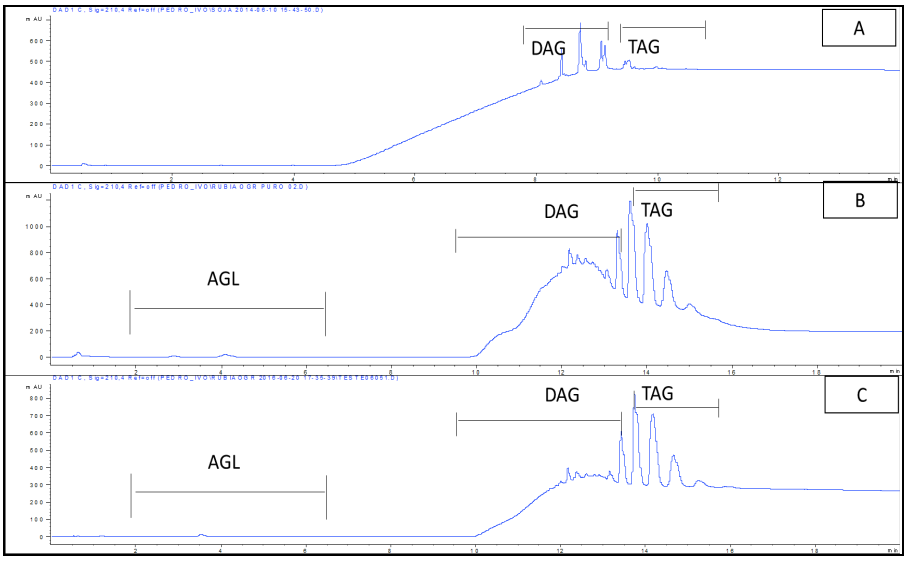

Figure 2. Chromatograms of refined soybean oil (A), untreated (B), and RFOF after treatment (C) obtained by HPLC-UV/DAD at 210 nm

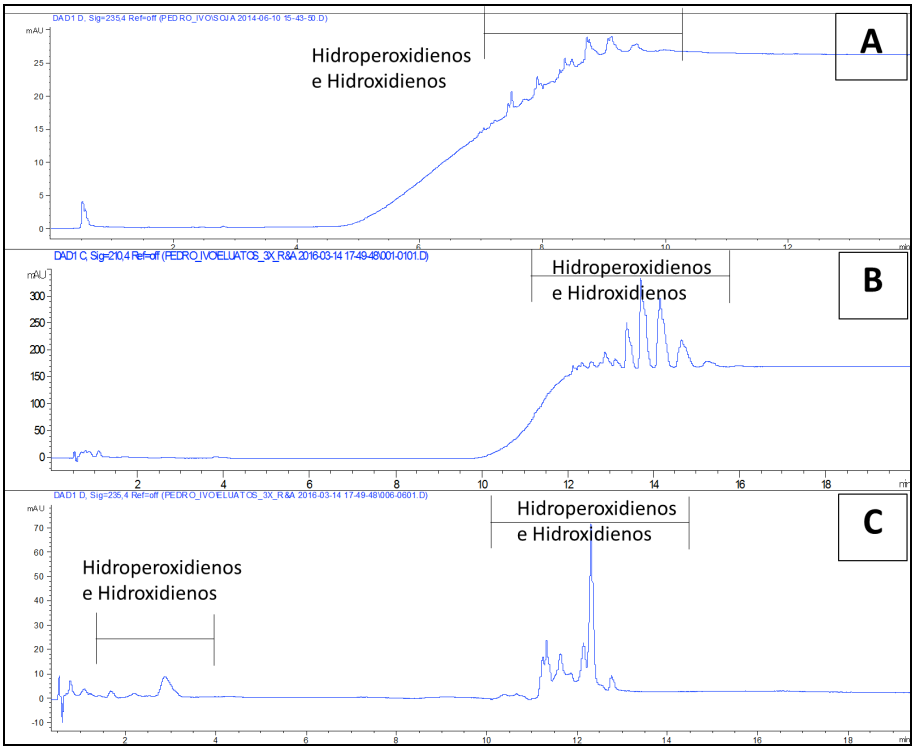

Figure 3. Chromatogram of refined soybean oil (A), untreated (B), and treated RFOF (C) obtained by HPLC-UV/DAD at 235 nm

This decrease in the concentration of oxidized species via liquid-liquid extraction followed by distillation is also shown in Fig. 4, demonstrating that the treatment was effective in reducing second-order oxidized compounds, the cetoperoxydiens (MORALES et al., 2012b, 2012c).

3.2 Physico-chemical characterization

The physicochemical characterization of refined soybean oil (RSO) and oil and treated RFOF showed properties that influence quality, performance, and cost of biodiesel (Table 1). Deep frying food releases a large amount of water, forming free fatty acids (Botelho, 2012), mono- and diglycerides, increasing acidity index and saponification (ALBERICI et al., 2012; CHARPE; RATHOD, 2011). Moreover, because part of the unsaturation are oxidized or have a broken link during frying, the RFOF has a higher peroxide index and lower iodine index (SILVA et al., 2011) than RSO. The oxidation products also increase the kinematic viscosity RFOF (CHAKRABORTY; GUPTA; CHOWDHURY, 2014; IGLESIAS et al., 2012; PHAN; PHAN, 2008).

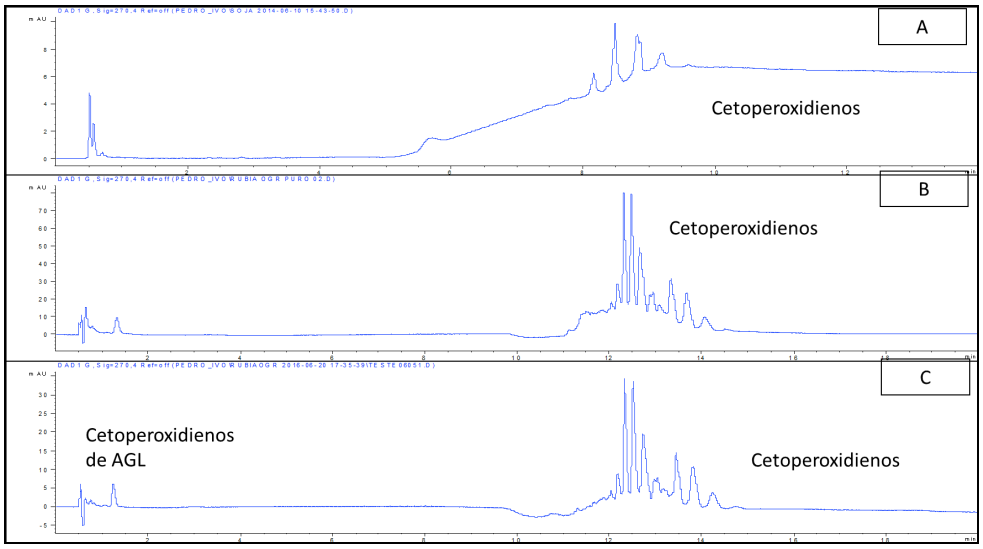

Figure 4. Chromatogram of refined soybean oil (A), untreated (B), and treated RFOF (C) obtained by HPLC-UV/DAD at 270 nm

Table 1. Physico-chemical characterization of RSO and RFOF

Proprieties	RSO	RFOF	RFOF Filtered in Na_2SO_4
Water content ($mg.kg^{-1}$)	157.55	1,190.16	357.00
Acidity index (mg $KOH.g^{-1}$)	0.23	1.18	1.18
Saponification index (mg KOH g^{-1}ofoil)	121.19	134.26	134.26
Peroxyde index ($meq.kg^{-1}$)	6.64	14.47	14.47
Iodine index ($g.I_2^{-1}$)	123.74	73.01	73.01
Kinematic viscosity ($mm^2\ s^{-1}$)	32.42	42.68	42.68
Oxidative stability (h)	6.55	2.93	2.93

The presence of peroxides in RFOF can also form sludge and sediment that, by having higher polarity than biodiesel, separate from alkyl esters of fatty acids and diesel hydrocarbons,which can clog nozzles (ALBERICI et al., 2012; DA SILVA et al., 2011).The increase in kinematic viscosity is due to the formation of polymers, observed as lumps. The breaking of hydrocarbon chains and release of free fatty acids after frying increased the amount of volatile species that reduce the oxidative stability of RFOF compared to OSR, besides color change and foaming.

The acidity index above 1 mg.KOH.g-1 suggests the need of a conjugate process in order of RFOF to be converted to biodiesel, such as esterification followedby transesterification (CAI et al., 2015; ENCARNAÇÃO, 2008; SUAREZ; MENEGHETTI, SIMONI, 2015). The high water content in RFOF is also a problem, which cancompromisebiodiesel production, yield, and quality. Keeping water in the RFOF would requires a higher proportion of alcohol then oil in biodiesel production to favor the formation of esters in the reaction (CASTRO, 2009; SILVA, 2011). As this has a high cost and is energetically infeasible, it is necessary to add sodium sulphate in the filtration to reduce the water content, which was reduced to 70%, from the previous 1190.16 to 357 mg.kg-1.

3.2.1. Chemical elements in RFOF

Some chemical elements, especially metals, are added to the oil during frying. These metals are usually derived from foods or utensils (CHARPE; RATHOD, 2011; GUPTA, 2005). However, we detect neither iron nor aluminum (Table 2), suggesting that the metallic container used for frying barely influenced metal contamination. We found none chemical elements monitored in the RSO, and the RFOF only contained magnesium and phosphorus, possibly from potatoes and fish that are rich in magnesium, and meat that is rich in phosphorous (e.g., phospholipids).

Phosphorus and magnesium are monitored in biodiesel and can compromise biodiesel quality. Magnesium can be deposit in the nozzle and phosphorus generates emissions with a large amount of particulate matter that influence automotive catalytic converters, forming gums (JAIN; SHARMA, 2013; KNOTHE, 2007; LÔBO; FERREIRA; CRUZ, 2009).

Table 2. Content of chemical elements in RSO and RFOF.

Chemical elements		Quantification threshold	Content ($mg.kg^{-1}$)	
			RSO	RFOF
Ag	Silver	0.24	<LQ	<LQ
Al	Aluminum	0.02	<LQ	<LQ
B	Boron	0.37	<LQ	<LQ
Ba	Barium	0.10	<LQ	<LQ
Ca	Calcium	0.24	<LQ	<LQ
Cd	Cadmium	0.02	<LQ	<LQ
Cr	Chrome	0.04	<LQ	<LQ
Cu	Copper	0.26	<LQ	<LQ
Fe	Iron	0.02	<LQ	<LQ
K	Potassium	0.03	<LQ	<LQ
Mg	Magnesium	0.01	<LQ	0.60±0.03
Mn	Manganese	0.05	<LQ	<LQ
Mo	Molybdenum	0.04	<LQ	<LQ
Na	Sodium	0.29	<LQ	<LQ
Ni	Nickel	0.07	<LQ	<LQ
P	Phosphor	0.25	<LQ	7.20±0.09
Cu	Lead	0.39	<LQ	<LQ
Si	Silicon	0.10	<LQ	<LQ
S	Tin	0.02	<LQ	<LQ
V	Vanadium	0.01	<LQ	<LQ
Zn	Zinc	0.02	<LQ	<LQ

3.2.2. Chromatographic analysis of fatty acids

Unsaturated fatty acids chain play a key role in determining the properties of biodiesel (LEÃO.; ARANDA 2009). For example, the greater the amount of unsaturated fatty acids, the better the performance, because the unsaturation increase their polarity, allowing a charge instability which facilitates bond breaking. The RFOF analyzed has about 80% of unsaturated fatty acids (TABELA 3), which is favorable for biodiesel production. The majority of it is composed of oleic acid, which improves flow properties without damaging oxidative stability, since it is a monounsaturated.

Table 3. Fatty acids profile of RFOF

Fatty acids		Normalized Percentage (%)	
		OSR	RFOF
C14:0	Myristic	0.1	0.2
C16:0	Palmitic	13.8	8.0
C16:1 *cis9*	Palmitoleic	1.1	4.4
C18:0	Stearic	3.3	4.6
C18:1 *cis9*	Oleic	22.2	48.4
C18:1 *cis11*	Vaccenic	1.4	0.1
C18:2 *cis9, cis12*	Linoleic	55.6	32.0
C18:3 *cis9, cis12, cis15*	Linolenic	1.4	0.9
C20:0	Arachidic	0.4	0.6
C20:1 *cis9*	Gadoleic	0.1	0.1
C22:0	Behenic	0.4	0.0
C22:1 *cis13*	Erucic	0.1	0.5
C24:0	Lignoceric	0.1	0.2

Frying decreased the content of more unsaturated fatty acids, such as linoleic and linolenic in both RSO and RFOF. Thus, the diunsaturated linoleic acid comprehends 55.6% of the chemical composition of fatty acids of the RSO and 32.0% of RFOF, indicating that the frying decreased 58% of its percentage. The tri-unsaturated linolenic acid is more susceptible to thermal degradation and oxidative had a more pronounced decrease, reducing its percentage in 64%, ranging from 1.4% in RSO to 0.9% in RFOF.

Previous studies showed that the percentage of linoleic acid generally decreases with frying and high temperatures. Other studies showed that the soybean oil has on average 50% linoleic acid, but reduces to 13%after frying (FREIRE; MANCINI FILHO; FERREIRA, 2013; SANIBAL; MANCINI FILHO, 2004). Decreasing the di- and tri-unsaturated acids increased the proportion of most monounsaturated and saturated fatty acids. Thus, the content of oleic acid increased from 22.2% to 48.4%, which is an increase of 218%.

The lower the amount of saturated fatty acids in RFOF, the better the performance in biodiesel production (ChhetrI, Watts e Islam (2008) e Knothe, Gerpen e Krahl (2005)). Furthermore, a high content of monounsaturated acids (e.g., oleic and palmitoleic) is important to provide both good oxidative stability and fluidity. We found the same situation in RFOF, indicating that its fatty acid composition is suitable for biodiesel production (BEZERGIANNI; CHRYSIKOU, 2012; KNOTHE, 2007; MITTELBACH; GANGL, 2001). However, the change in fatty acids composition is not only due to oxidation and thermocatalysis, but also to absorption of part of the soybean oil fatty acids subjected to frying, as well as the release of fatty acids from food. It is noteworthy that this second pathway is less common than the first, because the OGFR had different fatty acids from RSO. Furthermore, the temperature and time of frying, the continuous or discontinuous heating, and oil replacement can change the levels of fatty acids. These vary in restaurants and cafeterias, because they usually use several heating and cooling cycles, such as before and after food addition, and during and in the end of frying. These factors partly explain because frying can produce oils with the same physicochemical behaviour as obtained here comparing RSO and RFOF, but at different intensity levels (GUPTA, 2005). Thus, it is possible to obtain frying oils with different contents of water and fatty acids, and index of acidity, peroxide, and iodine, but always with the physico-chemical and structural modifications shown here.

4. Conclusions

Here, we demonstrated that the treatment of RFOF with Liquid-Liquid Extraction, distillation, and filtration in adsorbent decreased the water and free fatty acids content, which can compromise the efficiency of conversion to biodiesel, and the content of oxidized components, which promote sludge and sediment formation, especially if biodiesel is mixed with diesel. Moreover, the high residual acidity, common in RFOF, indicates that biodiesel production directly from it should use hydroesterification or acid esterification followed by alkaline transesterification. The latter would involve mixing small amounts of RFOF with high quality oil, so only alkaline transesterification is needed. This pathway is already used by biodiesel plants.

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