

## FFA Adsorption from Waste Oils or Non-Edible Oils onto an Anion-Exchange Resin as Alternative Method to Esterification Reaction Prior to Transesterification Reaction for Biodiesel Production

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### Abstract

Biodiesel is an alternative source of energy in diesel engines in its neat form or as a blend with conventional diesel fuel. The raw feedstock costs represent a lot of the overall biodiesel production cost. Therefore, the employment of waste oils or non-edible oils (which do not compete with edible oils) as feedstock for biodiesel production are presented as the best alternative. However, the presence of free fatty acids (FFA) in these oils is undesirable for biodiesel production due to the transesterification reaction performance is reduced. Hence, in this paper, the adsorption as treatment for reducing or removing FFA from oils prior to transesterification reaction for biodiesel production is examined using a strong anion-exchange resin in order to avoid esterification reaction employed commonly prior to transesterification reaction.

**Keywords:** Adsorption; Anion-exchange resin; Biodiesel; Deacidification; Free fatty acids

### Introduction

Due to the increasing energy demand and greenhouse gases problems caused by the use of fossil fuels, it has become necessary to develop alternative fuels as well as renewable sources of energy [1]. Biodiesel is a particularly attractive renewable fuel as it has significantly lower emissions and is less toxic than petroleum diesel when it is burned. It is biodegradable making it a more environmental benign fuel. Moreover, biodiesel can be used in conventional diesel engines without modification [2]. Biodiesel is obtained through transesterification reaction of vegetable oils or animal fats with an alcohol (methanol or ethanol, commonly) yielding a mixture of long chain esters and a valuable co-product, glycerol [3-4]. When methanol is used as reactant, reaction product will be a fatty acid methyl esters mixture (FAME), whereas if ethanol is used as reactant, a fatty acid ethyl esters mixture (FAEE) will be obtained.

Despite the advantages of the biodiesel use; it presents some disadvantages. One of its drawbacks is the high production cost, which is due to the high cost of the vegetable oils (edible-oils, in general). The raw feedstocks cost represent approximately 70 to 95% of the overall biodiesel production costs [5]. Besides, the biodiesel industry competes with the food industry for oil crops. In other words, biodiesel is competing limited land availability with food industry for plantation of oil crop. In fact, it needs large percentages of the current available arable land to obtain biofuel using crops such as rapeseed or sunflower. Therefore, it is inevitable to explore new raw materials but economically viable feedstocks as alternatives to edible vegetable oils in order to reduce the biodiesel price without competing with food production [6-8]. Attention has been focussed on the non-edible oils and on the used frying oils which are renewable as well as easily available [9-12].

However, these non-edible oils or low quality feedstocks present higher free fatty acid (FFA) content (or acid values) than edible oils which inhibit the transesterification reaction.

In the transesterification reaction for biodiesel production, the triglycerides (TG) are easily converted to biodiesel when the free fatty acid content from feedstock is relatively low. Expressly, many researchers reported that feedstocks containing a free fatty acids

concentration higher than 1% wt/wt would not be fully converted into biodiesel [13-16]. In fact, high FFA content could cause the soap formation through the undesirable saponification reaction, especially when homogeneous basic catalysts are employed in the transesterification reaction. Therefore, it is necessary to reduce or remove these FFA present in the alternatives feedstocks to edible oils in order carry out the transesterification reaction without a reduction in the performance of the same.

The general methods to reduce or remove FFA from oils are neutralization, extraction by alcohol, steam distillation and esterification reaction [17]. Neutralization for deacidification has many limitations due to glycerides can also be saponified by alkali and then the separation of glycerides from the formed soap stock is very difficult, which results in a large loss of glycerides. Extraction by alcohol for deacidification needs a great amount of solvent and many extraction stages because of the limited solubility of FFA in alcohol. Steam distillation for deacidification requires a high temperature and consumes a large amount of energy with a low efficiency. In bibliography, esterification reaction makes best use of FFA in oils because transforms FFA into biodiesel (FAME).

Esterification reaction is normally carried out in a homogeneous phase in the presence of acid catalysts such as  $H_2SO_4$ , HF,  $H_3PO_4$ , HCl and p-toluene sulfonic acid [18,19]. However, the use of these catalysts is dangerous because they are hazardous and corrosive liquid acids. Therefore, acid heterogeneous catalysts can be considered as an alternative to minimize environmental damage and reduce biodiesel cost [20]. However, in general high reaction temperatures are needed

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to carry out the FFA esterification to FAME using heterogeneous catalysis [21].

Adsorption can be an alternative procedure to reduce or remove FFA from oils. Adsorbent treatment has the advantages of reduced oil losses and soap contamination [22]. However, there are a few studies investigating the adsorption of FFA on adsorbents such as different types of clay: montmorillonite, sepiolite, kaolinite cristobalite and bentonite [23-28]. Recently, there are some studies on the adsorption of free fatty acids using anion resins [29-31]. The present work reports the use of a different anion-exchange resin (Dowex 550A) for adsorbing FFA from different oils as previous step to transesterification reaction in order to replace the esterification reaction.

A lot of research efforts have been carried out in relation to cation-exchange resins as catalysts for the esterification reaction due to in acidic ion-exchange resin, FFA removal is accomplished by esterification of FFA at the acidic functional site in the presence of methanol or ethanol [32-36]. However, we propose the use of an anion-exchange resin as adsorbent of FFA instead of catalyst due to adsorption is an easier method because it can carry out to low temperature and it not requires an alcohol such as occurs in the esterification reaction; so costs of raw materials can be reduced.

## Experimental

### Materials

Dowex monosphere 550A resin provided by Sigma-Aldrich was used as adsorbent and as catalyst for the esterification reaction. Physical properties of the resin are shown in Table 1. Pumice granules purchased from Panreac were used as catalytic support for the transesterification reaction.

Three types of oils with different acid values (AV) or FFA contents were used as raw material: a commercial edible-grade sunflower oil (AV=0.17 mg KOH/g oil) purchased from the market, a waste oil (AV=2.13 mg KOH/g oil) from frying in a canteen (filtered to remove suspended solids) and a *Jatropha curcas* oil (AV=6.62 mg KOH/g oil) extracted from its seeds. Besides, acid oil was made by adding different amounts of oleic acid to sunflower oil in order to achieve the desired FFA content. Commercial grade oleic acid was purchased from Merck. Methanol (p.a. 99.8%) used as reactive for esterification and transesterification reaction was supplied by Sigma-Aldrich.

### Procedures

In a typical adsorption experiment (Figure 1a), certain amount of resin, between 10-20% wt. related to oil weight, was added to 100 g of acid oil (20 mg KOH/g oil) in a 250 ml jacketed glass container and the mixture was shaken at 200 rpm at 45°C for 2 h. Then, the resin was separated from oil by filtration and the amount of remaining FFA was determined by acid value titration. In this method, a weighed amount

Structure	Gel
Matrix	Styrene, divinyl-benzene
Functional group	Quaternary amine
Counter ion	OH-
Moisture content	44-50%
Total exchange capacity	≥ 1.1 equiv/L
Particle average diameter	590±50 µm
Density	1.08 g/mL
Maximun operating temperature	60°C

Table 1: Physical properties of Dowex monosphere 550A resin.

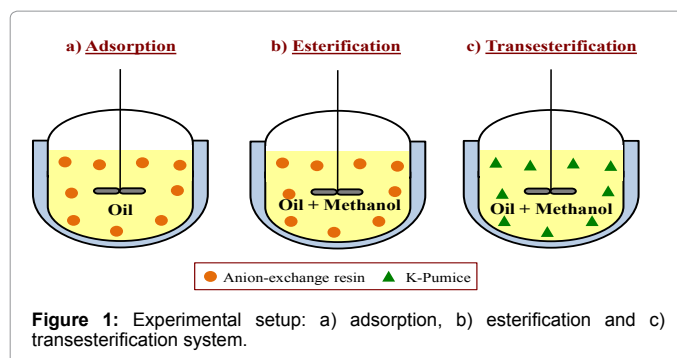


Figure 1: Experimental setup: a) adsorption, b) esterification and c) transesterification system.

of the sample was added into a flask and it was dissolved in a diethyl ether and ethanol mixture; phenolphthalein was added as an indicator. KOH were used as titrant solution and the acid value was calculated using the next equation:

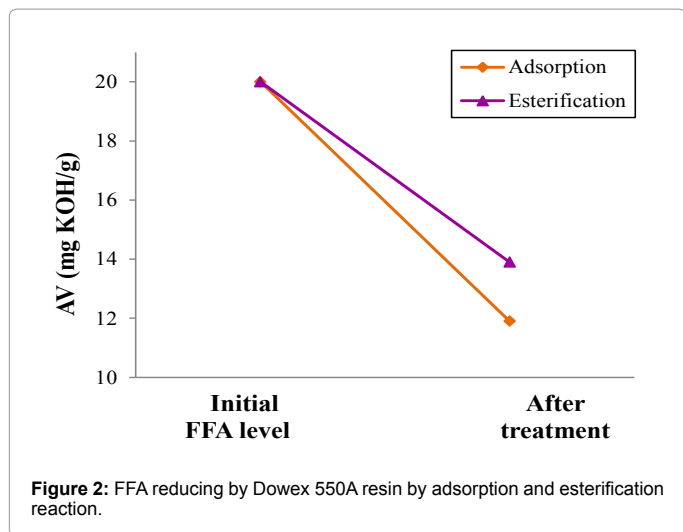
$$\text{Acid value} \left( \frac{\text{mg KOH}}{\text{g}} \right) = \frac{56.1 V 0.1}{W}$$

Where 56.1 is the molecular weight of the solution employed for titration (mg mmol<sup>-1</sup>), V is the volume of the titrant solution (ml), 0.1 mmol ml<sup>-1</sup> is its concentration and w is the weight (mg) of the analyzed sample.

Esterification reaction was performed in a 250 ml jacketed glass reactor equipped with a reflux condenser and a mechanical stirring (Figure 1b). The reaction system was heated to 45°C and 100 g of acid oil (20 mg KOH/g oil) was added. When the oil reached selected temperature, methanol (6:1 methanol/oil molar ratio) and the resin as catalyst (20% wt. related to oil weight) were added with continuous stirring for 2h. After stipulated reaction time, the catalytic solid was separated from the reaction mixture by filtration. The liquid product obtained was rotary evaporated to remove methanol in excess. The acid value of main product obtained was determined by titration. Besides, the main product was analyzed by <sup>1</sup>H Nuclear Magnetic Resonance (NMR) spectrometry (Bruker Avance 400) in order to estimate the fatty acid methyl ester yield and the reaction yields by the method exposed by Gelbard et al. [37].

In addition, a comprehensive study on the production of biodiesel from two acid oils (waste oil and *Jatropha curcas* oil) was carried out: first, oil deacidification by adsorption of free fatty acids onto the resin was performed; subsequently, the deacidified oil was used as feedstock, together with methanol, for transesterification reaction in order to produce biodiesel. The FFA adsorption was carried out under the same conditions as described above (20% wt. resin, 45°C and 2 h).

Heterogeneous catalysed transesterification reaction (Figure 1c) was carried out using sieved pumice particles (size range: 1.4-3.0 mm), submitted to a potassium interchanging treatment (K-Pumice) as described by Borges et al. [38]. This catalyst showed good activity acting as heterogeneous catalyst on biodiesel production process by transesterification reaction. The transesterification reaction was carried out in the same reaction system as that employed for the esterification reaction and under the optimum operation conditions obtained by Borges et al. [38] when employed K-Pumice as heterogeneous catalyst for the sunflower oil transesterification reaction (55°C, 2 h, 18:1 methanol/oil molar ratio, 20%wt. catalyst amount). After 2 h reaction time, the catalytic solid was separated from the reaction mixture by filtration and the liquid product obtained was rotary evaporated to



remove methanol in excess, and then settled in a separating funnel. The upper phase consisted of fatty acid methyl esters (biodiesel product, FAME) and the lower phase contained the glycerol by-product. Moreover, the biodiesel product obtained was analyzed by NMR spectrometry.

Infrared spectra of the pure Dowex 550A resin and there's after adsorption and after esterification reaction were collected using a Thermo Nicolet, Avatar 360 FT-IR.

## Results and Discussion

This study investigated the deacidification capacity of Dowex 550 A resin. In Figure 2 the FFA reduction from an acid oil (20 mg KOH/g oil) is observed when Dowex 550 A resin is employed as adsorbent (FFA adsorption) or catalyst for esterification reaction (conversion FFA to FAME).

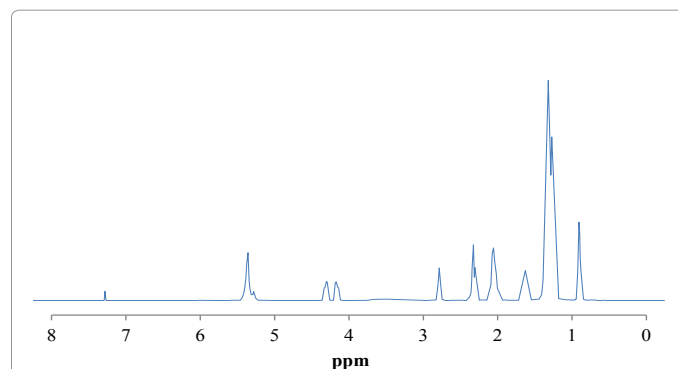
Both treatments produced a significant reduction in acid value; however the FFA reduction by adsorption appears to be more effective. Though, we must to confirm what the reason for the acid value decrease is. For this, FAME content in the product obtained from the esterification reaction was determined by <sup>1</sup>H NMR in order to verify the conversion of FFA to FAME. Figure 3 shows <sup>1</sup>H NMR spectra of the product obtained from the esterification reaction using the resin as catalyst. FAME was not produced during the esterification reaction because no peak appears at 3.7 ppm characteristic of methoxy groups in the methyl esters [37]. Therefore, the FFA reduction observed may be due to the FFA adsorption on the resin and no to the conversion of FFA to FAME. FT-IR spectra of the resin after being subjected to the adsorption treatment of FFA and to the esterification reaction are shown in Figure 4. These spectra are compared with the spectrum of pure resin. In the spectra of the resin subjected to the FFA removal treatments, peaks characteristics of oleic acid were observed. Two peaks appear at 2933 cm<sup>-1</sup> and 2856 cm<sup>-1</sup>, which were attributed to the asymmetric CH<sub>2</sub> stretch and the symmetric CH<sub>2</sub> stretch, respectively. The peak at 1754 cm<sup>-1</sup> was derived from the existence of the C=O stretch [39].

Therefore in both treatments the adsorption of free fatty acids on the resin is occurring as shown in Figure 5. Dowex 550A resin is an anion-exchange resin with quaternary amine as functional group and an OH<sup>-</sup> as counter ion. Hence, OH<sup>-</sup> group from resin could be exchanged by carboxylic group from acid oleic; then carboxylic group

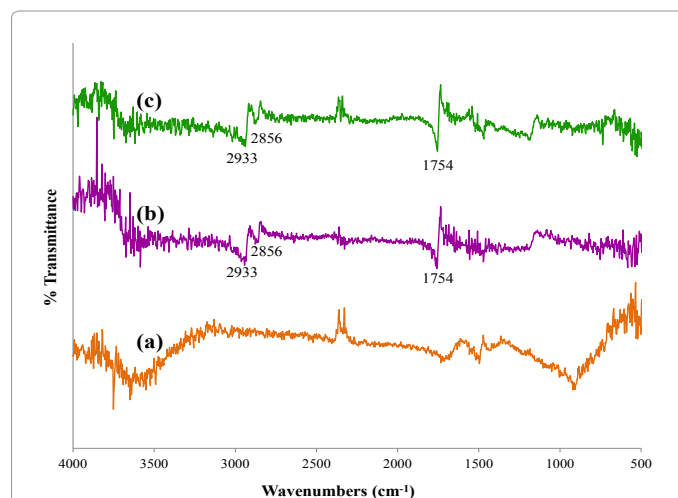
will be adsorbed onto N<sup>+</sup>R<sub>3</sub> group. In this process, water molecules could be generated.

Cren and Meirelles [40] studied the oleic acid adsorption from ethanol + water solutions using a strong anion-exchange resin (Amberlyst A26 OH) and they proposed that adsorption of fatty acids in anionic resins involves an ion exchange equilibrium in which the hydroxyl ions present initially in the resin are partially replaced by counterions generated from the dissociated fatty acids. However, when a weak anion-exchange resin is employed as adsorbent, the adsorption is due to the formation of weak hydrogen bond complex between the lone pair electron on nitrogen of the amino group present on the resin matrix and acidic hydrogen of fatty acid. The interaction which is expected between the lone pair of electrons on nitrogen of secondary and tertiary amino groups and acidic hydrogen can be treated as a Lewis acid-base interaction [41,42].

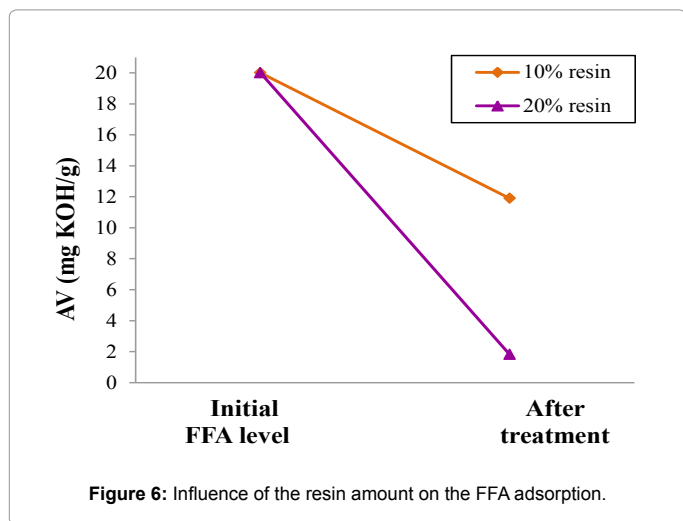
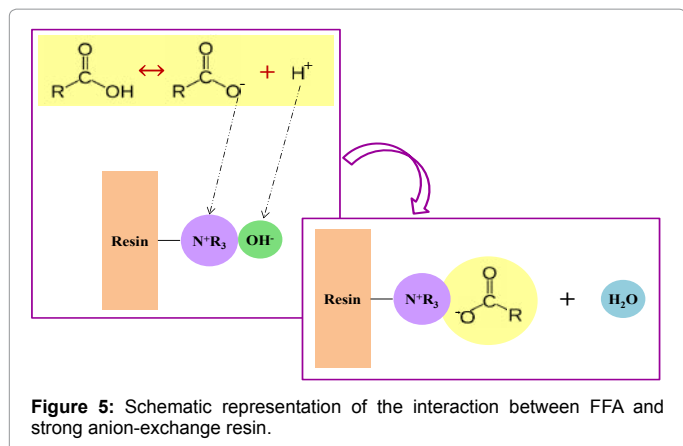
Marchetti et al. [43] used this anion resin (Dowex 550 A) as heterogeneous catalyst to carry out the esterification reaction of free fatty acid from frying oil. They concluded that this catalyst appears as a great alternative to purify frying oil; achieving a final conversion of FFA around 80%. However, they determined the conversion of FFA by titration and gasses chromatography, but they did not analyze the FAME content. Therefore, it is likely that the FFA decrease observed by them is due to adsorption of free fatty acids on the resin as demonstrated



**Figure 3:** <sup>1</sup>H NMR spectrum of the product obtained from esterification reaction using Dowex 550A resin as catalyst.



**Figure 4:** FTIR pattern spectra from (a) pure Dowex 550A resin, (b) resin after adsorption, (c) resin after esterification reaction.



earlier in our study. Besides, some researchers agree in anion-exchange resin (basic resin) the mechanism of FFA removal appears to be adsorption to the basic site; while in cation-exchange resin (acidic resin), FFA removal is accomplished by the esterification of FFA at the acidic functional site in the presence of methanol or ethanol [44].

Later, Marchetti and Errazu [45] performed a similar study which the resin was also employed as a catalyst for the transesterification reaction and, in this case, the FAEE content was analyzed by gasses chromatography. These authors observed that excessive reaction times (3-8 days) were necessary to achieve high FAEE content. As a result, they propose that the reaction time needs to be optimized. In order to reduce the reaction time, the same anion resin could be used as FFA adsorbent and, then a second step could be carry out, so the transesterification reaction with base catalyst. This alternative could be reduced the production process of biodiesel from 3-8 days to 2-4 hours, approximately.

In Figure 6, a higher FFA reduction is observed when the resin amount is increased for adsorption process; this is because when the amount of resin is increased, more functional groups will be available for adsorption of the free fatty acids. When the resin amount was increased from 10 to 20% wt., the acid value of oil decreased from 11.9 mg KOH/g oil to 1.82 mg KOH/g oil. Therefore, the amount of

resin used for the adsorption process is important for achieving the reduction of free fatty acid required.

Due to the adsorption of FFA in oil onto anion-exchange resin has been studied as previous step to transesterification reaction; two oils which present high FFA content (waste oil, 2.13 mg KOH/g oil and *Jatropha curcas* oil, 6.62 mg KOH/g oil) were subjected to the complete process (FFA adsorption + transesterification reaction from deacidified oil) in order to obtain biodiesel. Acid values of both oils before and after the adsorption treatment of the free fatty acids on the resin and after the transesterification reaction using K-Pumice as heterogeneous catalyst are shown in Table 2. Moreover, the FAME yield of the transesterification reaction is also shown.

FAME yield of 96.4% and 95.6% oil were obtained from waste oil and *Jatropha curcas* oil, respectively. Although during adsorption of free fatty acids onto the resin some water molecules could be generated; these apparently do not affect or poison the active sites of the K-pumice used as a heterogeneous catalyst in transesterification reaction, as was demonstrated in a previous study [47].

However, if high water amounts were generated during the adsorption process and other homogeneous or heterogeneous catalyst (no water tolerant) would be used in the transesterification reaction; a water adsorption step, prior to the transesterification reaction and after the FFA adsorption process, may be carried out using eg. Hydrophilic silica gel, sieve molecular. The Figure 7 shows a diagram of the proposed process.

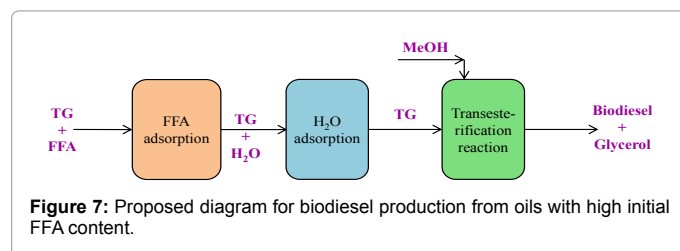
## Conclusions

Recent investigations have focused on the development of potential deacidification processes to remove FFA effectively without deteriorating the quality of oils. In this work, the FFA adsorption using an anion-exchange resin appears to be a viable solution for reducing or removing FFA from oils. The adsorption of free fatty acids onto Dowex monosphere 550 A resin is a pre-treatment for decreasing the initial acid content of waste oil or non-edible oil with high initial free fatty acids content, obtaining a suitable raw material for biodiesel production.

When the objective is to carry out the esterification reaction to reduce free fatty acids from oil, a cationic resin should be used as catalyst, whereas when the objective is to carry out the reduction of free

	Waste oil	<i>Jatropha curcas</i> oil
Initial AV*	2.13	6.62
AV after adsorption*	0.25	0.28
AV after transesterification*	0.14	0.17
FAME yield (%)	96.4	95.6
* mg KOH/g		

**Table 2:** Characteristics of oil before and after adsorption process and of the product obtained after transesterification reaction.



fatty acids from oil by adsorption treatment, an anionic resin should be used as adsorbent. Anion-exchange resins appear not to be suitable to carry out the esterification reaction because the FFA is not converted to FAME.

FFA adsorption is an easier method than esterification reaction because it can carry out to low temperature and it not requires an alcohol such as occurs in the esterification reaction; so costs of raw materials can be reduced.

Due to the high effectiveness of the Dowex 550A resin for the removal of FFA; effects of contact time, resin amount (adsorbent dose), initial FFA concentration will be studied in future works. Besides, adsorption isotherms and kinetics will also evaluated.

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