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APPLICATION OF MICROALLOYED STEELS FOR HANDLING OF ACIDIC CHEMICALS IN THE BIODIESEL INDUSTRY

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ABSTRACT

Base-catalyzed transesterification of vegetable oils and fats is one of the most used technologies for biodiesel production globally. Several alternatives have been studied to enable the use of acidic and cheap raw materials for biodiesel production, but the initial investments are usually high due to the need for equipments made up of special steels. The present work was aimed at evaluating the corrosion resistance of three Brazilian steels in biodiesel with different acidity extents. Biodiesel was synthesized by the transesterification of soybean oil with methanol and further added oleic acid at specific contents. Gravimetric tests were carried out by the immersion of the steels in the biodiesel samples for up to 1344 h. The corrosion resistance of the steels was evaluated by corrosion rate determinations, whereas the biodiesel quality was monitored by acidity index and conductivity measurements. The best results were obtained with the microalloyedsteel, however, the quality of the biodiesel was reduced with the increasing oleic acid content, restricting the application of microalloyed steel to biodiesel with an acidity of up to 1%.

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INTRODUCTION

The processing technology of vegetable oils and fats has become highly valuable since they are renewable resources with several applications in the chemical, pharmaceutical and food industries, including the production of biofuels, such as biodiesel and bioethanol.Biodiesel was first included in the Brazilian energy matrix in 2005, which established the obligatory minimal biodiesel percentage (v/v) to be mixed with the commercial diesel sold throughout the country. Biodiesel integration was planned to occur gradually, starting with a mixture of 2 % (B2) by Law no. 11.097/05. In the subsequent 15 years, Brazilian National Council of Energy Policyauthorized the use of biodiesel at percentages larger. Currently the quality requirements of biodiesel are established by ANP Resolution No. 45/2014 and amended by Resolution No. 798/2019. In this context, transesterification of vegetable oils has become one of the most widely used technologies for biodiesel production. The transesterification process is advantageous in a broad perspective, but it still has several bottlenecks that affect its commercial viability, for instance, the cost of the raw materials, which accounts for 70 % of the operational costs. Vegetable oils are mainly comprised of triacylglycerols, which are chemically formed by a molecule of glycerol (1,2,3-trihydroxypropane) esterified with three fatty acid molecules. Any fatty acid not bonded to glycerol is referred to as free fatty acid and it is not converted to biodiesel during transesterification. The free fatty acid content in the raw material is limited to 0,5mg KOH/g as a biodiesel quality requirement.Several processes have been studied to prompt the use of cheaper, acidic raw materials for biodiesel production, such as catalytic and thermal cracking, esterification, hydroesterification, among others. These approaches reduce the operational cost significantly, however the initial investments are high due to the need for equipments built with special metal alloys (Tapaneset *al.*, 2013; Aranda *et al.*, 2008). The contact of the acidic raw material or biofuel with the metal surface is a critical aspect due to the typical susceptibility of metal alloys to corrosion (Fazal*et al.*, 2011a; Fazal *et al.*, 2011b; Fernandez *et al.*, 2013 and Konav *et al.*, 2015). Alloys used in the automobile industry can be likewise used for storage and transportation of biodiesel. In this context, microalloyed steels have been highly considered for such applications, which is also encouraged by their high mechanical strength and toughness that allow the design of lighter structures (Soeiro *et al*., 2013). These steels, also known as high-strength low-alloy (HSLA) steels, have almost the same composition as low carbon and manganese steels, with maximum C and Mn contents of 0.25% and 2%, respectively. HSLA steels also contain small amounts of alloying elements, including aluminium, vanadium, titanium, niobium, copper, molybdenum, nickel and chromium (Wollantset al., 2003). Microalloyed HSLA steel is a particular type of HSLA steel that is comprised of very tiny amounts of vanadium, niobium, nickel and/or titanium, not exceeding 2% which guarantees its high mechanical strength. The vanadium and titanium (up to 0.12%) improve mechanical resistance without reducing weldability, nickel (up to 0.7%) improves atmospheric corrosion resistance and benefits surface quality, while niobium (0.02%) increases the maximum mechanical strength and the yield point**Error! Bookmark not defined.**. Out of the microalloyed carbon steels, there are the classes API X70 and X80, which are produced by thermomechanical controlled rolling (TMCR) using small amounts of alloying elements, guaranteeing a minimum yield point of 275 MPa, higher mechanical strength, higher toughness and better corrosion resistance than conventional laminated carbon steels (Tariq, 2002).

The corrosion resistance- of microalloyed HSLA steels in contact with biofuels has been scarcely reported in the literature. AQUINO et al., 2012, evaluated the corrosive effect of biodiesel on carbon steel, stainless steel, aluminium, brass, copper, nickel, zinc and tin using gravimetric and electrochemical techniques. SERQUEIRAet al., 2014, examined the corrosive effect of soybean-based biodiesel by static immersion corrosion tests in presence of the antioxidant tert-butylhydroquinone (TBHQ) over 12 weeks. CANTERLE et al., 2016, evaluated the susceptibility of the AP X70 steel to stress corrosion in different ethanolic media by slow strain rate testing and mechanical fracture tests. The results indicated that API X70 steel was susceptible to stress corrosion, which caused a decrease in its toughness. Recently, FAZAL et al.16examined metals surfaces exposed to palm biodiesel. Immersion test confirmed the presence of pitting corrosion on the metal exposed to biodiesel. More pitting is observed on the copper surface. ALVES et al., 2019 evaluated the linkage between biodiesel degradation and the corrosion of stainless steel used to store the biodiesel. Were observedmicropits after the blend diesel-biodiesel (B7) immersion test. DEYAB et al., 2019, were studied the inhibition capacity of cardanol for the corrosion of AA 5052 H32 in biodiesel. Wasobserved that inhibition efficiency increases with increasing concentration of cardanol. Herein, the present work was aimed at evaluating the corrosion resistance of API X70 microalloyed steel in biodiesel with varying free fatty acid content. A comparative study was particularly conducted by evaluating the corrosion resistance behaviour of carbon steel and galvanized steel under similar experimental conditions.

MATERIALS AND METHODS

Materials

Three different steels were evaluated in the biodiesel corrosion tests: API X70 microalloyed steel, carbon steel and galvanized steel. These steels have many industrial applications, including in the diesel/biodiesel industry. Their chemical compositions are summarized in Table 1. The specimens used in the immersion tests consisted of bars prepared from the steel samples with a metallographic cutter (TECLAGO X60). The area and density values of these bars are given in Table 2. A 1mm width hole was made near the edge of each bar. Prior to the static immersion tests, the bars were sanded on bench polisher (AROTEC) using 100- and 600-µm grit SiC sandpaper, washed with water, degreased with ethanol, dried with a cold air jet and stored in a desiccator.

Table 1. Chemical composition of the metal alloys (Fe by balance)

Element	Microalloyed steel	Galvanized steel	Carbon steel
C	0.0600%	0.1400%	0.0564%
Si	0.2570%	0.1290%	0.0070%
Mn	1.5790%	0.6550%	0.2450%
P	0.0151%	0.0220%	0.0081%
S	0.0021%	0.0091%	0.0023%
Al-t	0.0340%	0.0001%	0.0540%
Cr	0.2170%	0.0070%	0.0120%
Ni	0.0110%	0.0030%	0.0040%
Mo	0.1110%	0.0020%	0.0020%
Cu	0.0050%	0.0050%	0.0160%
V	0.0010%	0.0009%	0.0018%
Nb	0.0620%	0.0005%	0.0007%
Ti	0.0170%	0.0003%	0.0003%
B	0.0005%	0.0001%	0.0002%
N	0.0050%	0.0055%	0.0135%
Ca	0.0028%	0.0011%	0.0001%
Sn	0.0011%	0.0010%	0.0010%
Co	0.0033%	0.0013%	0.0014%
Pb	0.0001%	0.0010%	0.0010%
As	0.0023%	0.0013%	0.0018%
Zr	0.0030%	0.0008%	0.0008%
Zn	0.0005%	0.0000%	0.0000%

Synthesis of biodiesel from soybean oil: The biodiesel used in this work was synthesized by the transesterification of commercial refined soybean oil using an IKA RCT BASIC magnetic stirrer. In brief, 100 g of soybean oil were added to the reactor equilibrated at 45 °C under constant stirring of 500 rpm. Next, KOH solution in methanol (1 wt.%) was added to the reactor at a soybean oil to alcohol molar ratio equal to 6. The reactants were left to react for 60 min, then the reactor was shut down and cooled to room temperature. The mixture was transferred into a separating funnel and was allowed to settle for 12 h until separating into two phases, the top layer (~ 90%) mainly comprised of biodiesel and the bottom layer (~ 10%), also called as crude glycerin, which is formed by 30-50% glycerin, unreacted alcohol, catalyst and soap. The biodiesel phase was recovered for purification, while the crude glycerin was adequately discarded. The purification of the biodiesel was done by washing, settling and drying. The washing/ settling steps were performed in a separating funnel at 60 °C using 0.15% citric acid solution. The biodiesel was dried in a 1 L beaker at 130 °C under constant stirring at 500 rpm.

Acidification of biodiesel with oleic acid: The basic differences between fatty acids relates to the hydrocarbon chain length and the number and position of double bonds. Oleic acid is naturally present at very large amounts in triglyceride feedstocks, thus it has been used as a model molecule in studies with acidic raw materials intended for biodiesel production.To study the biodiesel acidity effect on the corrosion resistance of the steels, oleic acid was incorporated in the soybean-based biodiesel at 1 %, 3 % and 10 %. The acidity of the biodiesel was determined before and after acid addition following the ASTM D664. Acidity determinations were also performed after the static immersion tests.

Table 2. Electrical conductivity of biodiesel exposed to API X70 microalloyed and galvanized steels, h

Immersion Time	API X70 microalloyed steel			Galvanized steels	
	0 % oleic acid	1 % oleic acid	3 % oleic acid	0 % oleic acid	1 % oleic acid
0	0.0	0.0	0.0	0.0	0.0
168 h	0.0	0.0	0.0	0.0	1.2
336 h	0.0	0.2	1.84	0.0	1.1
672 h	0.3	0.3	3.3	0.6	1.2
1344 h	1.5	1.35	6.7	1.7	2.5

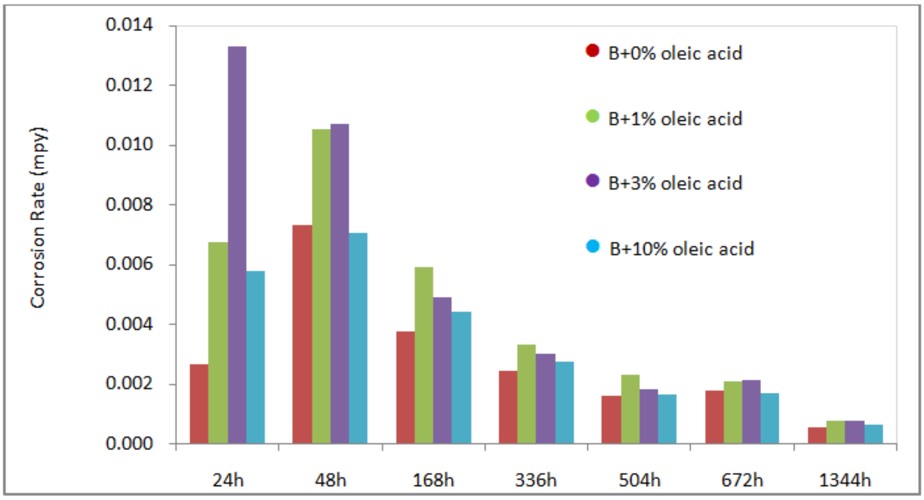

Figure 1. Corrosion rate of the API X70 microalloyed steel in biodiesel with different oleic acid contents

Static Immersion Tests: The corrosion resistance of the three steels in biodiesel was evaluated by gravimetric tests (static immersion tests) as per ASTM G1 and ASTM G31. As previously mentioned, the specimen bars were polished using 100-, 200-, 300-, 400- and 600-µm grit SiC sandpaper, starting by the smallest grit (100-µm) progressing the polishing process up to the 600-µm grit. Afterwards, the bars were cleaned by immersion in 20 % HCl solution for 30 s. They were then washed with neutral soap under running water, cleaned with ethanol to remove oil from their surface, washed again with distilled water and dried with a cold air jet. Next, the bars were weighed in an analytical scale (± 0.0001g) until at least 3 equal values were registered, which were defined as the initial mass (m0) of each bar for further mass loss and corrosion rate determinations. The static immersion tests were carried out in triplicate for all steel bars described in Table 2.
The immersion tests were conducted in a round-bottom glass flask containing approximately 250 ml of biodiesel added with oleic acid at different contents. The biodiesel amount used in each test was adjusted to 0.20 mL/mm² of sample according to the ASTM G31. Three polished bars of each steel were immersed in the biodiesel samples to ensure data reproducibility.The steel bars were left into the biodiesel for specific times (24, 48, 168, 336, 504, 672 and 1344 h).

Characterization of Biodiesel: The acidity and electrical conductivity of the biodiesel samples used in the static immersion tests were evaluated in parallel with the steel mass loss determinations. The acidity index was determined following the AOCS (American Oil Chemists' Society) Cd 3d-63 procedure. Electrical conductivity is a measure for the total concentration of metal ions dissolved in the biodiesel samples. Electrical conductivity measurements were performed on an Analyser® DDS-11C Benchtop conductometer (Laboratory of Environmental Biotechnology Research, LPBA/Uezo). Measurements were carried out with conductivity cell of K = 1 cm-1 calibrated with solution of 1413 mS/cm for the range 0.5 µS/cm - 200 mS/cm.

RESULTS AND DISCUSSION

Gravimetric Tests: The corrosion rates of the API X70 microalloyed, carbon and galvanized steels immersed in the soybean-based biodiesel with different oleic acid contents were calculated from the mass losses recorded in the static immersion tests. Figure 1 shows the evolution of the corrosion rate as a function of the immersion time for the microalloyed steel. It is clearly observed that the corrosion was more pronounced in the first hours of immersion, and that the corrosion rate decreased significantly over time, specifically after 168 h. This behaviour may be associated with the formation of corrosion products onto the API X70 steel surface. Fazal et al, 2012, observed a decrease in the copper corrosion after 1200 h of immersion. A similar behaviourwas also observed for the corrosion of aluminium immersed in palm oil-based biodiesel.

Chandran et al., 2016, also reported a reduction in the copper corrosion rate after 540 h of immersion in modified biodiesel. Furthermore, it was verified that corrosion extent increased with the increasing oleic acid content. The biodiesel samples included with 1% and 3% oleic acid led to the highest corrosion rates in the API X70 steel regardless of the immersion time. This confirms that the biodiesel corrosivity for this alloy is proportional to the free fatty acid content. The interaction between the galvanized steel and the biodiesel samples is depicted by the corrosion rate vs. immersion time plots in Figure 2. It can be seen a significant corrosion within the first hours of immersion, but the corrosion rates decreased with the increasing immersion time.

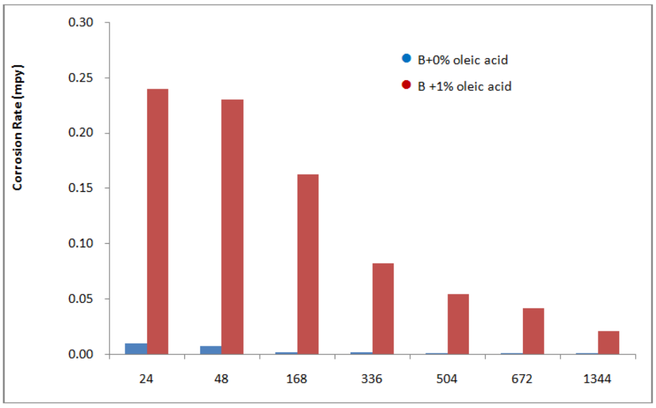

Figure 2. Corrosion rate of the galvanized steel in biodiesel with different oleic acid contents. Biodiesel+0% oleic acid (Blue) and Biodiesel +1% oleic acid (red)

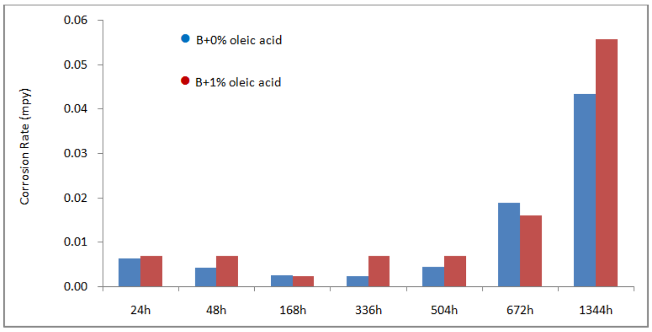

Figure 3. Corrosion rate of the carbon steel in biodiesel with different oleic acid contents

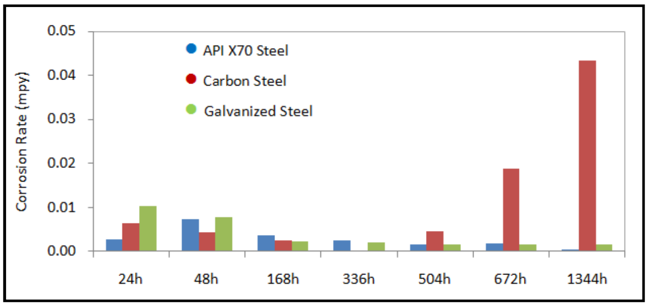

Figure 4. Corrosion rate of the steel samples in pure biodiesel (0 % oleic acid added)

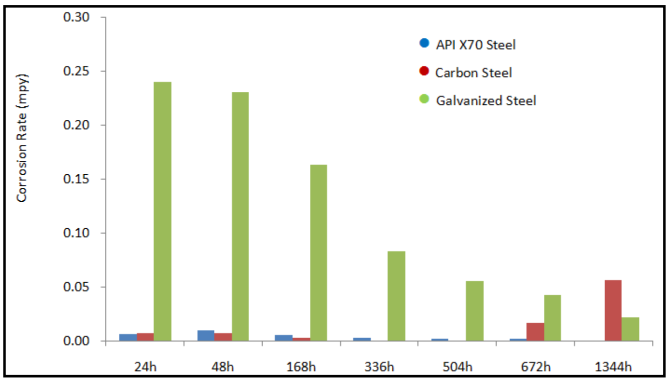

Figure 5. Corrosion rate of the steels samples in biodiesel added with 1 % oleic acid

The addition of 1% oleic acid to the biodiesel considerably increased the corrosion rate of the galvanized steel. Overall, the corrosion resistance of the API X70 microalloyed steel was superior to that of the galvanized steel. For instance, their corrosion rates in the first 24 h of immersion in biodiesel were 0.067 mm/year and 0.2398 mm/year, respectively. Such a difference was also large even after 1300 h of immersion, 0.008 mm/year vs. 0.214 mm/year. The lower corrosion resistance of the galvanized steel may be explained by the presence of a zinc layer on its surface, which is more likely to corrode because zinc is a very reactive metal. In contrast, the API X70 steel contains alloying elements such as nickel, molybdenum, niobium and titanium, whichare known to increase the corrosion resistance of metal alloys. The corrosion behaviour of the carbon steel immersed in biodiesel differed from those of the other steel samples, as can be seen in Figure 3. The corrosion rate was practically constant until 504 h of immersion and further increased with the increasing immersion time. FAZAL et al., 2010, reported a similar behaviour with respect to the increasing corrosion of copper and stainless steel in palm oil-based biodiesel over time. However, it is verified that, likewise the API X70 and galvanized steels, the corrosion rate of the carbon steel was higher when the sample was immersed in the biodiesel added with oleic acid, meaning that the biodiesel corrosivity for the carbon steel is also determined by the free fatty acid content.

Altogether, the microalloyed steel presented the highest corrosion resistance. This can be concluded from the comparison between the corrosion rates of the steel samples immersed in pure biodiesel (Figures 4) and biodiesel with 1 % oleic acid (Figure 6).

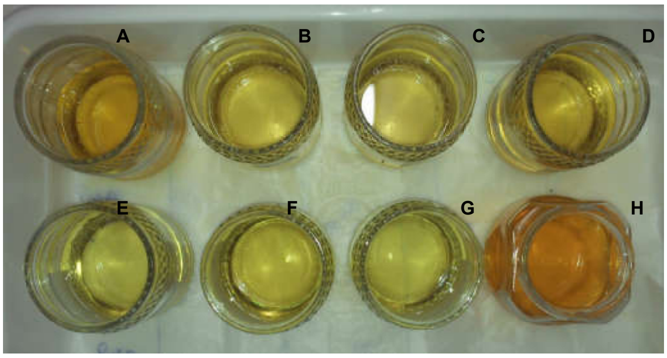

Figure 6. Colour changes in biodiesel samples with 0 %, 1 %, 3 % and 10 % oleic acid exposed to API X70 microalloyed steel (A, B, C and D, respectively), biodiesel samples with 0 % and 1 % oleic acid exposed to carbon steel (E and F, respectively) and biodiesel

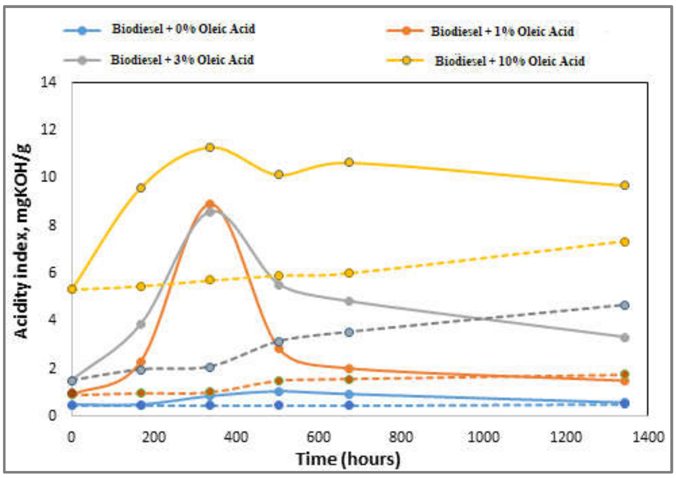

Figure 7. Acidity index as a function of time of biodiesel samples added with oleic acid (0, 1 %, 3 % and 10%) without steel contact (—●—) and exposed to API X70 microalloyed steel (- - ● - -).

The corrosion rate of the galvanized steel was significantly larger than those of the API X70 and carbon steels regarding the first 24 h of immersion in pure biodiesel. The differences between the corrosion rates became more significant with the increasing immersion time, especially after 300 h. It is observed that the API X70 steel exhibited the lowest corrosion rates, whereas the corrosion of the carbon steel was highly pronounced for immersion times longer than 300 h. These results may be due to the faster formation of corrosion products onto the galvanized steel surface, thus forming a passive layer that hinders the corrosion progress. Overall, the oleic acid addition increased the corrosion rates of all steels, markedly for the galvanized steel, Figure 5. This may be directly attributed to the chemical composition of the samples. As previously mentioned, the API X70 steel contains more noble alloying elements than the other steels, thus it exhibited the highest corrosion resistance in pure and acidified biodiesel. galvanized steel displayed the most evident colour changes, once the largest corrosion rates were observed for this alloy. The higher the corrosion rate the higher the migration of metallic ions to the biodiesel and the larger the colour difference. The colour changes in the biodiesel samples exposed to the API X70 and carbon steels were also significantly different.

Characterization of Biodiesel: The acidity index of the soybean-based biodiesel with different oleic acid contents was determined before and after the static immersion tests. Acidity indexes were also determined considering biodiesel samples without and with metal contact after a period of 60 days (1400 hours), as given in Figure 7. The total acidity of the pure biodiesel (without oleic acid addition and metal contact) was about 0.5 mg KOH/g. This value did not increase over the experimental time, standing below 1.0 mg KOH/g.

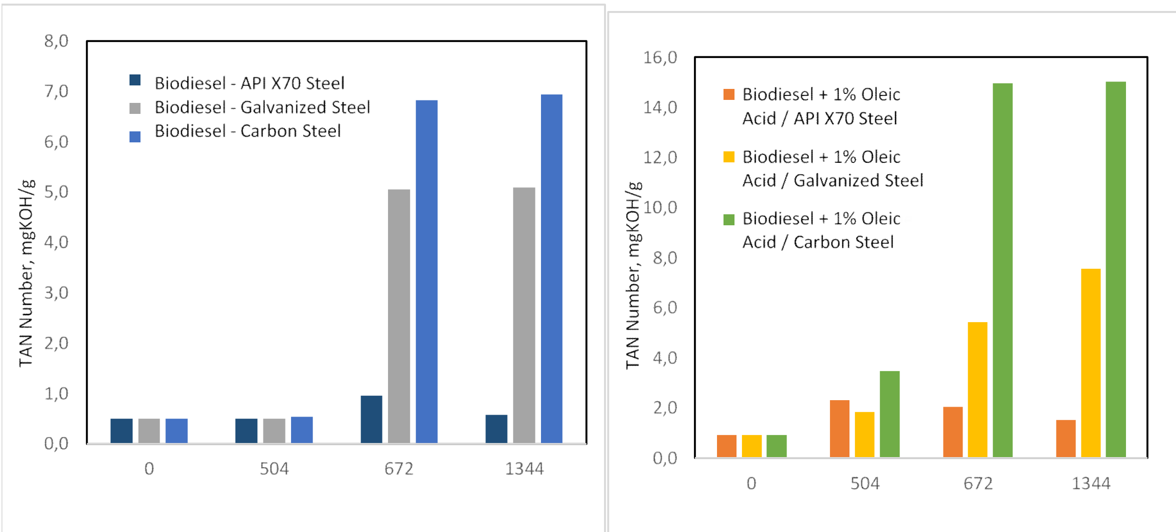

Figure 8: Comparison between the acidity indexes of the biodiesel exposed to the steel samples a) 0 % oleic acid b) 1 % oleic acid

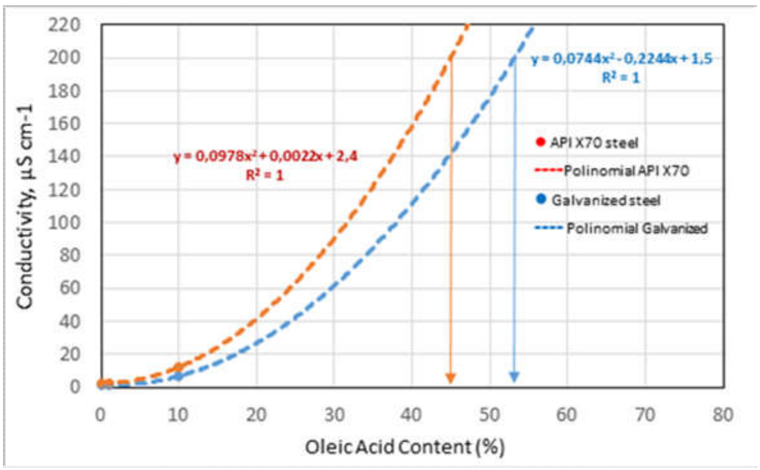

Figure 9. Electrical conductivity (µS cm -1) as a function of oleic acid content for the soybean-based biodiesel exposed to the steel samples for 1344 h

The biodiesel corrosivitywas further evaluated by the colour changes resulting from the contact with the alloy samples. Corrosion phenomena typically change the colour of the liquid medium due to the leaching of metallic ions from the metal surface. Figure 6 illustrates the appearance of the pure biodiesel and biodiesel acidified with 1 % oleic acid after 1344 h of contact with the API X70, galvanized and carbon steels. As expected, the biodiesel samples in contact with the It is also possible to notice that the total biodiesel acidity showed a direct correlation with the oleic acid content. The acidity of the oleic acid-added biodiesel was also time-dependent, that is, the higher the oleic acid content and the storage time the larger the total acidity index. The contact between the soybean-based biodiesel and the API X70 steel also resulted in an increased acidity index. This effect was perceived even for the pure biodiesel sample. Figure 7 reveals a general trend in which the acidity of the biodiesel in contact with the API X70 steel reached a maximum value followed by a decline. The largest acidity index values were observed at 336 h of immersion time. The most pronounced biodiesel acidity declines were observed for the oleic acid contents of 1 % and 3 %, while the most gradual one was observed at 10 % oleic acid. The acidity index peaks seen in Figure 7 are directly proportional to the metal ion amount leached from the API X70 steel surface. A comparison with the corrosion rates of the API X70 steel (Figure 1) reveals that the highest rates occurred prior to 336 h of immersion, thus a considerable amount of metal ions has already migrated to the biodiesel. Figure 8 reveals a drastic increase in the biodiesel acidity after contact with the carbon and galvanized steels, reaching 15 mg KOH/g for the biodiesel sample added with 1 % oleic acid in contact with the carbon steel. These results agree with the larger corrosion rates presented by these alloys not only in pure biodiesel, but also in biodiesel acidified with 1 % oleic acid. Since the corrosion was more intense in these alloys in comparison with the API X70 steel, a higher amount of metal ions migrated to the biodiesel intensifying its degradation. It is further observed that the increase in the total acidity was more significant with the increasing immersion time. The largest acidity value was observed for an immersion time of 672 h. For longer times, the acidity remained constant, similarly to the biodiesel/API X70 microalloyed steel system.At the end of the immersion tests (1344 h), the acidity values increased 7-fold and 14-fold for the biodiesel without and with 1 % oleic acid, respectively. These results were expected, especially for the carbon steel, which is known to suffer general corrosion, forming corrosion products throughout its surface rapidly, whereas the galvanized and API X70 steels tend to suffer localized corrosion. According to Gentil, 2003, the carbon steel corrosion products exhibit a high contamination potential, which is harmful for the biodiesel/alloy compatibility.

Electrical Conductivity: Electrical conductivity measurements were performed on the biodiesel samples in contact with the galvanized and API X70 steels to provide further description of the role played by the oleic acid content and steel type on the biodiesel corrosion behaviour. The conductivity values shown in Table 3are considered low (< 200 μS/cm). However, these results indicate a possible correlation between the oleic acid content and the release of metal ions from the steels, the latter occurring at a lower extent for the API X70 steel. Assuming a second-order polynomial model between the biodiesel electrical conductivity and the oleic acid content (Figure 9), one can estimate that the inferior oleic acid contents able to reduce the corrosion resistances of the galvanized and API X70 steels are 53 % and 45 %, respectively.

Conclusions

Immersion tests performed on API X70 microalloyed, galvanized and carbon steels allowed to examine the corrosivity of soybean-based biodiesel, as well as to evaluate the oleic acid (free fatty acid) effect on their corrosion resistance. Considering the corrosion rate data, it can be concluded that the addition of oleic acid clearly increased the acidity of the biodiesel, which, in turns, increased the corrosion rate of the steels. The corrosion rates were higher for oleic acid contents of 1 % and 3%. The microalloyed and galvanized steels exhibited similar corrosion behaviours, that is, their corrosion rate decreased with the increasing immersion time, being the microalloyed steel more resistant to corrosion. This relates to the presence of more noble alloying elements, such as molybdenum and niobium, in its chemical composition. The quality of the biodiesel in contact with the microalloyed steel reduced with the increasing oleic acid content, until reaching unacceptable levels for fuel applications. In contact with microalloyed steel, acidity indexes of the biodiesel samples added with oleic acid displayed polynomial-type changes, reaching values higher than the acceptable level. More specifically, the acidity index of the biodiesel with 1 % and 3 % oleic acid suffered a markedly decline after reaching a maximum level at 336 h. This effect must be further evaluated considering the possible chemical reactions taking place at the alloy surface and in the biodiesel. Further, the contact of the biodiesel with the carbon and galvanized steels caused a strong increase in acidity index, including in the biodiesel sample without oleic acid addition.The electrical conductivity tests allowed to conclude that the conductivity of the biodiesel samples was also influenced by the oleic acid content, but, in general, they were low and not significant, especially in relation to the contact of the biodiesel with the microalloyed steel. In conclusion, the use of the API X70 microalloyed steel in the biodiesel industry is recommended when the raw material acidity lies below 1 %. The inclusion of additives in the biodiesel to extend the applications of this alloy should be evaluated carefully, considering both technical and economic aspects.

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