Comparative corrosive characteristics of petroleum diesel and palm biodiesel for automotive materials


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A B S T R A C T

Corrosive characteristics of biodiesel are important for long term durability of engine parts. The present study aims to compare the corrosion behavior of aluminum, copper and stainless steel in both petroleum diesel and palm biodiesel. Immersion tests in biodiesel (B100) and diesel (B0) were carried out at 80 °C for 1200 h. At the end of the test, corrosion characteristic was investigated by weight loss measurements and scanning electron microscopy with energy dispersive X-ray analysis (SEM/EDS). Fuels were analyzed by using TAN analyzer, FTIR, GCMS and ICP in order to investigate the acid concentration, oxidation level with water content, compositional characteristics and presence of metal species respectively. Results showed the extent of corrosion and change in fuel properties upon exposure to metals are more in biodiesel than that in diesel. Copper and aluminum were susceptible to attack by biodiesel whereas stainless steel was not.

1. Introduction

As a result of increasing environmental concern and diminishing petroleum reserves, there is a growing trend to substitute biodiesel for conventional diesel fuel. As an alternative fuel, though biodiesel have some technical advantages over diesel fuel, the former appears to be more corrosive than diesel. The corrosive nature of biodiesel can be more aggravated if free water and free fatty acid are present in it. As compared to diesel, biodiesel is more prone to absorb water which tends to condense on metal surface and may cause enhanced corrosion. Beside this, auto-oxidation of biodiesel can also enhance its corrosive characteristics and degradation of fuel properties.

There are only few studies available in the literature related to corrosion of different metals in biodiesel [1–5]. Most of these studies find corrosiveness of different biodiesel other than palm biodiesel. Kaul et al. [1] investigated the corrosiveness of different biodiesel (i.e. Jatropha curcas, Karanja, Mahua and Salvadora) as compared to that of diesel fuel. They found that biodiesel from Jatropha curcas and Salvadora were more aggressive for both ferrous and non-ferrous metal. Geller et al. [2] have reported that as compared to ferrous alloys, copper alloys are more prone to be attracted by corrosion into fat based biodiesel. In an another study, pitting corrosion was found on the bronze sintered filters integrated oil nozzle after 10 h operation with biodiesel at 70 °C [3]. Such effectiveness was also reported even for lower biodiesel (2%) blend [4]. Maleque et al. [6] and Haseeb et al. [7] found that wear rate in biodiesel was relatively increased due to its oxidative and corrosive nature. It has been suggested that copper, aluminum, zinc, brass and bronze are not compatible with biodiesel [8,9]. Besides, these metals even in small concentration exposed into biodiesel have been reported as catalyst to oxidize biodiesel [10,11]. According to Sarin et al. [10], during oxidation process, the fatty acid methyl ester usually forms a radical next to double bond and then quickly bond with the oxygen from air. After 25 h oxidation of rapeseed oil methyl ester at 200 °C, Niczke et al. [12] found different types of volatile products like acids, aldehydes, ketones, lactones, allylalcohols etc. According to Tsuchiya et al. [4], oxidation of biodiesel reconverts esters into different mono-carboxylic acids such as formic acid, acetic acid, propionic acid, caproic acid etc. which are responsible for enhanced corrosion. This process also increases the free water content. Free water is undesirable because it may promote microbial growth and corrode fuel system components [13,14].

Concerns arise from the fact that biodiesel degrades through auto-oxidation, moisture absorption, attack by microorganisms etc. during storage or use. These may give rise to potential problems such as interaction with metal surfaces and at the same time, degradation of fuel properties. Fuel degradation due to metal contact can also be different from metal to metal. What makes the situation more complicated is the fact that under the exposure of different metals into biodiesel, dissolved oxygen may aggravate its corrosive nature in different level. A full clarification of such observations is often quite complicated, as a number of different effects may be involved (changes in TAN value, increased water content, oxidation product, presence of metal species, changes in structural features of biodiesel component etc.). Irrespective of such effects, a limited but definite role

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is usually attributed to characterize the corrosion behavior of different metals into fuel.

The present study aims to investigate the corrosion behavior of copper, aluminum, stainless steel in diesel and palm biodiesel, and in turn to evaluate their influence in degradation of fuel properties. The cause behind choosing these metals for investigation can be attributed to their broad range of usages in manufacturing automobile components. In automobile fuel system, the common parts made from aluminum are piston (100%), engine block (19%), cylinder head (70%) etc. [15]. Similarly, the metals used for manufacturing pump, injector, bearing are copper or its alloys [3,16]. Many components like nozzle, fuel filter, valve bodies, and pump ring are made from stainless steel [3,17]. All these parts from different materials come into contact with fuel and seem to undergo chemical interactions and thereby degrade fuel properties too. In a previous study [18], copper was found to be affected by pitting corrosion at 60 °C. In the present study, a higher temperature (80 °C) is used to investigate its effect on pit formation on copper as well as on other ferrous and non-ferrous metals. Results obtained are expected to help in understanding the corrosion of fuel system parts in biodiesel while its inlet temperature is 80 °C [19,20].

2. Experimental

The palm biodiesel used in this study was supplied by Weshchem Technology Sdn Bhd, Malaysia. The analysis report provided by the supplier is summarized elsewhere [18]. Corrosion characteristic of copper (99.99%), aluminum (99% commercially pure) and 316 stainless steel (18% Cr, 11% Ni, 2% Mn, 1% Si and 0.08% C) in both diesel and palm biodiesel was investigated by immersion test at 80 °C for 1200 h. During the test, fuels were continuously stirred by the magnetic stirrer at speed 250 rpm. The test coupons of copper (17.2 mm diameter × 2 mm thickness), aluminum (22.6 mm diameter × 2 mm thickness) and stainless steel (16 mm diameter × 2 mm thickness) were made from round bar by machining and grinding. For hanging the specimen into fuels, a hole of diameter 2 mm was drilled near the edge of the specimen. Before immersion, the coupons were treated as follows: polished with silicon carbide abrasive papers (from grade 400 to 1200), then washed and degreased with acetone. These were then dipped into 10% sulfuric acid at room temperature for several minutes followed by washing in deionized water. Similarly after exposure, for removing corrosion products, samples were scrubbed lightly in a stream of water with a polymer brush so as not to mechanically abrade the original surface. Before and after exposing the test coupons into different test fuels weight was measured by a balance with four decimal accuracy. Two duplicate coupons were immersed in each test fuel. For each coupon, weight loss was measured by subtracting the final weight (obtained after exposure) from its initial weight (before exposure). At the end of the test, corrosion behavior was investigated by measurement of corrosion rate and changes in surface morphology. The analysis report provided by the supplier is summarized elsewhere [18].

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\[
\text{Corrosion rate (mpy)} = \frac{W \times 534}{D \times T \times A}
\]

where corrosion rate “mpy” stands for mils (0.001 inch) per year, W is the weight loss (mg), D is the density (g/cm³), A is the exposed surface area (square inch) and T is the exposure time (h).

Changes in surface morphology were characterized by optical microscope (OM) and scanning electron microscopy with energy dispersive X-ray analysis (SEM/EDS). Fuels were analyzed by using gas chromatography mass spectroscopy (GCMS), Fourier transform infrared spectroscopy (FTIR), and inductively-couple plasma (ICP) in order to investigate the changes in fuel composition, oxidation level with water content and presence of metal species respectively.

Degradation of fuel properties were investigated by measuring total acid number (TAN), density and viscosity.

3. Results and discussions

3.1. Comparative corrosion rate

Fig. 1 shows the comparative corrosion rate for stainless steel, aluminum and copper upon exposure into diesel and biodiesel at 80 °C for 600 h and 1200 h. It is observed that the corrosion rate of copper in biodiesel increases with increasing time, while for aluminum, it slightly decreases. The corrosion rate of both copper and aluminum in biodiesel is much higher than that in diesel fuel. Stainless steel shows no significant corrosion even in biodiesel.

Presence of free fatty acid, more oxygen moieties and water content, impurities remaining after processing seem to increase the corrosiveness of biodiesel as compared to diesel fuel. Kaul et al. [1] suggested that biodiesel was more corrosive due the presence of higher concentration of unsaturated acid components. They have also reported that the increasing of TAN number after test duration indicates oxidation of biodiesel due to contact with metal samples. This is in good agreement with the results obtained by Tsuchiya et al. [4] where it has been reported that oxidation of biodiesel increases TAN number and water content and thereby becomes more corrosive. As shown in Fig. 1(b), the obtained corrosion rates at 80 °C in palm biodiesel for copper, aluminum and stainless steel are 0.586, 0.202...
and 0.015 mpy respectively while stirring speed was 250 rpm. In our previous study [18], tests were carried out at 60 °C under static immersion in palm biodiesel. The corrosion rate obtained for copper was 0.053 mpy which is much less than that in the current study. Higher corrosion of copper in the present study can be attributed to higher test temperature as well as presence of relative motion between metal and the fuels. This is in good agreement with the literature where it has been reported that copper could be susceptible to enhanced corrosion as a result of higher temperature [22] as well as sufficient relative motion between the metal and fluids [23]. However, corrosion rate can also be varied depending on the feedstock of biodiesel. Upon exposure to fat based biodiesel blend (B80) for 10 months at 38 °C, Geller et al. [2] found that the corrosion rate for copper is 0.5576 mpy. In another immersion test conducted by Kaul et al. [1] in Jatropha curcas, Karanja, Mahua and Salvadora biodiesel for 300 days at room temperature, the obtained corrosion rates for aluminum were 0.0784, 0.0065, 0.1329 and 0.1988 mpy respectively. These results more likely demonstrate that palm biodiesel seems to be little bit more corrosive than fat biodiesel or biodiesel from Jatropha curcas, Karanja, Mahua and Salvadora.

3.2. Surface morphology

Fig. 2 shows that copper and aluminum in biodiesel were subjected to higher pitting corrosion than that in diesel. Upon exposure in biodiesel, the average linear densities (measured by using Eq. (2)) of the pits formed on copper and aluminum surfaces are 80% and 18% respectively while in diesel, 54% and 10% accordingly. No significant change is found for the surfaces of stainless steel exposed in diesel and biodiesel.

\[
\text{Pit Density} = \frac{\sum_{i=1}^{n} l_i}{L} \times 100\% 
\]  

(2)

![Optical photographs (100×) of the surfaces for copper (Cu), aluminum (Al) and stainless steel (SS) sample after exposing to diesel and palm biodiesel at 80 °C for 1200 h.](image-url)
where $n$ is the number of pits on a straight line drawn horizontally from one end to another over the optical photograph, $i$ is for $(1, 2, 3, ..., n)$, $l$ is the length of a pit that overlaps with the drawn straight line and $L$ is the total length of straight line.

For further investigation of the damaged surface exposed into biodiesel, SEM pictures (of 100X, 2000X magnification) were taken (Figs. 3 and 4). Upon exposure into biodiesel at 80 °C, the number of pits on copper surface is much higher than that on other metals. Size and distribution of the pits are also different as seen in Fig. 3(b). The pit morphology and the mechanism of pitting seem to depend on the relative concentrations of both positive and negative ions. According to Mankowski et al. [24], copper in oxygen atmosphere forms oxygen rich CuO/CuCO$_3$ (outer layer) followed by Cu$_2$O (inner layer). Similar things seem to happen in O$_2$ rich biodiesel. On biodiesel exposed copper surface, pits are more likely to form by replacing oxygen ions from CuO through destruction of CuO layer from copper surface. Higher magnification of the pit area as seen in Fig. 3(c) shows the pits that are available in the oxide layer. Results from EDS analysis of biodiesel exposed copper and aluminum surfaces (Fig. 5) show that higher percentage of carbon and oxygen are available on the oxide layer. It is pointed that the growth of oxide layer may occur by metal moving outwards or oxygen inwards [25].

### 3.3. Compositional changes

Fig. 6 shows the changes in color for as-received both diesel and biodiesel upon exposure with different metals for 1200 h at 80 °C. It is seen that only copper exposed biodiesel and diesel show large changes in color as compared to their as-received states. This may be attributed to presence of metal species or change in biodiesel composition. Such possibilities were investigated by GCMS and ICP test and their respective results are shown in Tables 1–3.

Results from GCMS analysis (Tables 1 and 2) show that the major component in palm biodiesel are palmitate (16:0; 44.272%), methyl oleate (18:1, 35.934%) esters. These components are highly reduced in abundance after heating with or without exposing copper into biodiesel. Table 1 shows the comparative changes in biodiesel components with respect to the components present in as-receive condition while Table 2 shows the newly produced acids, ketones, aldehydes etc. after heating with or without exposing copper. The newly produced acids due to heating of biodiesel without exposing metal are decanedioic acid, nanonoic acid, hydroxypentadecanoic acid, 15-hydroxypentadecanoic acid etc. In the presence of copper, different acids like 9-octadecenoic acid, octanoic acid, nonanoic acid, hexadecanoic acid, 9-octadecanoic acid are found. In addition to these acids, different types of other short chain esters, aldehydes, ketones are found to be produced in both conditions. Apart from increasing the corrosiveness of biodiesel, these compositional changes may also degrade the fuel properties.

Table 3 shows the result obtained from ICP test. It is seen that sulfur is only available in diesel fuel. Upon exposure of different metals into diesel and biodiesel, the obtained metal species is always higher in biodiesel as compared to that in diesel fuel. This suggests that biodiesel is more corrosive than diesel fuel.

Results from ICP test show that more copper ions are found in biodiesel than that in diesel. This indicates that copper is more reactive in biodiesel. According to Lu X et al. [26], existence of oxygen might accelerate the formation of metal oxide. Color variation of test fuel as shown in Fig. 7 indicates the possible type of formed copper oxide. The probable oxide of copper that is dominant in biodiesel is copper carbonate (CuCO$_3$) of pale green color while in diesel, it is cuprite oxide (Cu$_2$O) of red color. According to Huang and Tsai [27], color variation is an indicator of the transformation of copper species with reaction temperature and oxygen atmospheres. Like copper, more aluminum species are found in biodiesel than that in diesel. The metal species present in fuel may also degrade the fuel properties.

In addition to formation of metal oxide, biodiesel itself can also be oxidized while metal can act as a catalyst [9,10]. Fig. 7 shows the concentration of biodiesel oxidation product before and after exposing to different metals.

According to Yamane et al. [28] esters react with the oxygen in air to form a number of different chemical species including aldehydes, ketones, carboxylic acids etc. Absorption of these carbonyl groups measured by FTIR spectroscopy as per ASTM D7418 shows that biodiesel become highly oxidized due presence of copper.

### 3.4. Changes in fuel properties

The total acid numbers (TAN) of the diesel and biodiesel were measured before and after exposure of different metals as shown in Fig. 8. It is seen that TAN numbers for as-received both diesel and biodiesel are under the limit given by ASTM standard specifications. But biodiesel upon 1200 h exposure to different metals crosses this limit while for diesel, no significant changes are observed.

Changes in total acid number of aluminum and copper exposed biodiesel are almost similar irrespective to their respective corrosiveness (Fig. 8). In other words, upon exposure, the acid number is increased in similar trend for both metals while the corrosion rate for different metals is different. This indicates that metal itself has also its
own characteristics to determine the corrosiveness of the fuel. In addition, based on biodiesel feedstock, the change in TAN number can also be varied. According to Kaul et al. [1] the TAN numbers for aluminum exposed Jatropha curcas, Karanja, Mahua and Salvadora biodiesel were changed from 0.38, 0.42, 0.32, 0.45 (mg KOH/g) to 14.48, 14.39, 11.30, 2.38 (mg KOH/g) accordingly. Increased TAN

Fig. 4. SEM picture of aluminum (Al) and Stainless Steel (SS) surface before and after exposure into palm biodiesel at 80 °C for 1200 h.

Fig. 5. Elemental analysis of copper and aluminum surface upon exposure into palm biodiesel at 80 °C for 1200 h.
number is the indicator of oxidation [1,4]. From Fig. 8, it is seen that the TAN number for copper exposed biodiesel is relatively higher than that of others. This suggests that copper acts as a strong catalyst to oxidize biodiesel. It is noted that the difference in TAN number between stainless steel and copper exposed biodiesel is very little but the difference in their respective corrosion rate is many times (Fig. 1). This suggests no strong correlation between change in acid number and corrosiveness of the fuel. Similar agreement was also found by Jakab et al. [29].

Fig. 9 shows the changes in density and viscosity for as-received both diesel and biodiesel as compared to metal exposed fuels at 80 °C for 1200 h. It is noticeable that only copper exposed biodiesel crosses the given limit for density whereas for viscosity, each fuel shows their value within the limit given by ASTM standard.

Change in density for copper exposed biodiesel is relatively larger than other fuels. Unlike density, values of viscosity for different metal exposed biodiesel are almost similar and also very close to the viscosity of its as-received state. However, density may change a lot due to presence of higher metal species in biodiesel as evidenced by the results obtained from ICP test (Table 3). It is seen from Fig. 10 that percentage of water in both diesel and biodiesel increases upon exposure to different metals. Results from Fig. 10 demonstrate that biodiesel absorbs more water than diesel fuel.

Water is the major ingredient to make the fuel more aggressive. From Fig. 10, it is seen that as-received both diesel and biodiesel have no water while after immersion test, water content is increased. Though as-received fuels are virtually water-free, during storage or use, due to its hygroscopic nature it can absorb moisture from atmosphere. A far more striking fact, however, is that the water content of biodiesel exposed with different metals is almost similar. This can indicate that increasing water content is not depended on the types of metal exposed in biodiesel. Upon exposure of metals into biodiesel, water may condense on metal surface and thereby enhance the corrosion process. According to Kaminski and Kurzydlowski [13], water can promote microbial growth and corrode fuel system components. Besides, water particularly at high temperature can hydrolyze esters as well as triglycerides and thereby produce different types of fatty acids which are more corrosive [30].

Results obtained from the investigation of compositional changes in fuel as well as degradation of fuel properties indicate that biodiesel is more aggressive than diesel fuel by bearing more acid compounds due to higher oxidation. This is in general agreement with the literature where it has been reported that biodiesel is far more prone to oxidation than petrodiesel [11,31]. The higher degradation of metal surface when in contact with biodiesel is probably related to its fatty acid components, water content, oxygen moieties etc. For diesel fuel, hydrocarbons themselves do not cause corrosion but the presence of water content, sulfur molecule and acid compounds can cause chemical attack [32]. However, these phenomena did not affect for changing surface morphology of stainless steel. Fig. 2 shows that both copper and aluminum in biodiesel were subjected to pit corrosion while stainless steel was unaffected. Such effectiveness of diesel and biodiesel for stainless steel cannot be expected with reference of their electrical conductivities since if they are the determinant, galvanic corrosion may happen preferably with the pair of biodiesel and stainless steel [29].

4. Conclusions

This study suggests the following conclusions:

1. As compared to diesel, biodiesel is more corrosive for copper and aluminum as indicated by weight loss and corrosion rate measurement, density of pits, and results from ICP test.
2. Copper acts as strong catalyst to oxidize palm biodiesel.
3. Higher moisture absorption, presence of oxygen moieties, and fatty acids produced from auto-oxidation seem to act as major factors for enhanced corrosiveness of biodiesel.
4. Biodiesel in contact with metals shows significant degradation in fuel properties as evidenced by increasing TAN number, viscosity and density.
5. Though stainless steel is compatible with biodiesel, it can change the fuel properties as well.

### Table 3

<table>
<thead>
<tr>
<th>Chemical name</th>
<th>Commercial name</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>As-received B100</td>
<td>B100 (heated)</td>
</tr>
<tr>
<td>Dodecanoic acid methyl ester</td>
<td>Methyl laurate (12:0)</td>
<td>1.07</td>
</tr>
<tr>
<td>Tetradecanoic acid methyl ester</td>
<td>Methyl myristate (14:0)</td>
<td>3.70</td>
</tr>
<tr>
<td>9-Hexadecanoic acid methyl ester</td>
<td>Palmitoleate (16:1)</td>
<td>0.49</td>
</tr>
<tr>
<td>Hexadecanoic acid methyl ester</td>
<td>Palminate (16:0)</td>
<td>44.27</td>
</tr>
<tr>
<td>Heptadecanoic acid methyl ester</td>
<td>Methyl heptadecanoate</td>
<td>0.19</td>
</tr>
<tr>
<td>8-Octadecanoic acid methyl ester</td>
<td>Methyl oleate (18:1)</td>
<td>35.93</td>
</tr>
<tr>
<td>Octadecanoic acid methyl ester</td>
<td>Methyl stearate (18:0)</td>
<td>4.13</td>
</tr>
<tr>
<td>Eicosanoic acid methyl ester</td>
<td>Arachidic (20:0)</td>
<td>0.24</td>
</tr>
<tr>
<td>9-12-Octadecatrienoic acid methyl ester</td>
<td>Linoleate (18:2)</td>
<td>0</td>
</tr>
</tbody>
</table>
Acknowledgement

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Table 2
Newly produced components in heated biodiesel with and without exposing copper.

<table>
<thead>
<tr>
<th>Heated B100 without exposing copper; B100 (heated)</th>
<th>Heated B100 upon exposure of copper; B100 (Heated/Cu)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acids (%)</td>
<td>Others (%)</td>
</tr>
<tr>
<td>Decanedioic acid (0.578%)</td>
<td>Oxiranoeanoic acid 3-octyl methyl ester (5.301%)</td>
</tr>
<tr>
<td>Nannoic acid (1.069%)</td>
<td>Methyl 10-oxohexadecanoate (7.263%)</td>
</tr>
<tr>
<td>15-Hydroxypentadecanoic acid (0.228%)</td>
<td>Nanonoic acid mono methyl ester (4.772%)</td>
</tr>
<tr>
<td>Hexadecanoic acid (1.874%)</td>
<td>Suberic acid methyl ester (2.592%)</td>
</tr>
<tr>
<td>15-Hydroxypentadecanoic acid (0.22%)</td>
<td>Butanal, ethylhydrazone (0.9%)</td>
</tr>
</tbody>
</table>

Table 3
Concentration of metal species obtained from ICP test of diesel (B0) and biodiesel (B100) before and after immersion test at 80 °C for 1200 h.

<table>
<thead>
<tr>
<th>Element</th>
<th>B100 (ppm)</th>
<th>B0 (Cu) (ppm)</th>
<th>B100(Cu) (ppm)</th>
<th>B0(Al) (ppm)</th>
<th>B100(Al) (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu</td>
<td>0</td>
<td>15.5</td>
<td>30.1</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Al</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>13.92</td>
<td>15.83</td>
</tr>
<tr>
<td>S</td>
<td>0</td>
<td>1.25</td>
<td>0</td>
<td>1.21</td>
<td>0</td>
</tr>
</tbody>
</table>

Fig. 7. Variation of oxidation product rate (absorbance per 0.1 mm path length: A/0.1 mm) of biodiesel before and after exposing or without exposing to different metals at 80 °C for 1200 h.

Fig. 8. Total acid number (TAN) measured in diesel and palm biodiesel before and after exposure to different metals at 80 °C for 1200 h.

Fig. 9. Changes in (a) density and (b) viscosity for both diesel and palm biodiesel before and after exposure to different metals at 80 °C for 1200 h.
immersion test by exposing different metals at 80 °C for 1200 h.

Fig. 10.

References


