

Corrosion Behaviour of Biofuel

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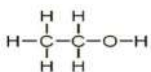
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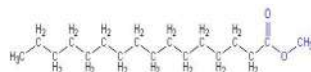
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Introduction

Biofuel are general name for liquid or gas fuel which is not petroleum-based fossils fuels. Also, biofuel is called nonconventional or alternative fuels. Biofuel can be classified to three categories which are first generation, second generation and third generation and this classification depends on the feedstock and the used conversion's techniques. First generation is produced from simple materials such as simple sugars, starch, fats and vegetable oils. Some examples for first generation biofuel are: “methanol, ethanol, propanol and butanol (bioalcohols), methyl-tertiary-butyl-ether or MTBE and ethyl-tertiary-butyl-ether or ETBE (bioethers), methane, carbondioxide, nitrogen, hydrogen, hydrogen sulfde and oxygen (biogas), carbonmonoxide and hydrogen (syngas) and cellulose, hemicelluloses and lignin(wood for solid biofuels)” [1]. While, second generation was produced from biomass such as lignocellulose, biohydrogen and wood diesel. Third generation is biofuel from algae. This report will focus on two types of biofuel which are bioethanol and biodiesel which can be used in automobiles [1]. There chemical structures are shown below:



Bioethanol chemical structure



Biodiesel chemical structure

Biofuel was used to reduce the dependence on fossil fuel and to produce fuel which reduces pollution and other environmental issues. The cost of biofuel is depending on the raw materials that were used to produce biofuel. Fossil fuel has higher cost if it is compared with biofuel [2]. Transesterification of vegetable oil or fat from the animal were used to give biodiesel. Biodiesel could be used as emulsifier because it is miscible with diesel and alcohol and this blend could be used in diesel engines. Biodiesel play important role in this blend because it prevents phase separation in the fuel blend contains ethanol and diesel. In addition, biodiesel can work as diesel fuel without producing nether aromatics nor sulfur. Bioethanol is another alternative fuel which can be extracted by using crops or biomass. When ethanol blended with fuel it can eliminate the emissions of nitrogen oxide if it compared with regular fuel. The problem which may occur when fuel content was changed was the material's issues such as corrosion in some parts of the automobile. An example of these parts which may contact the fuel are fuel injector, filters, piston, and fuel pump which will be most degraded part by fuel especially if it was made of copper alloy.

The corrosion of these parts may lead to another problem if it causes corrosion on the surface of metal then it may affect the properties of the fuel. Some metals which are not suitable to come in contact with bioethanol or biodiesel are bronze, copper, brass, aluminium and zinc [3].

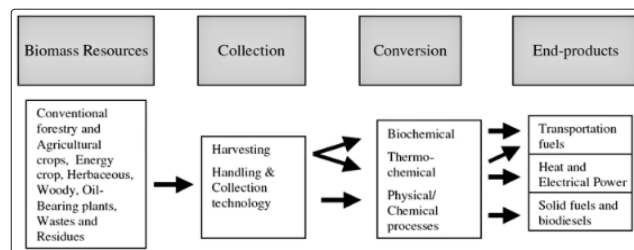


Figure 1: Typical sources and uses of biomass for energy purposes [1].

Bioethanol Corrosion Occur During Liquefaction or Pyrolysis Process for Biomass Fuel

Bio-fuel can be produced from biomass by four ways which are direct liquefaction, fast hydrolysis, hydrolysis and hydrothermal liquefaction. The acids, halide species and sulfur present in biofuel can cause corrosion damage in the metals in contact with fuel during fuel production from biomass. An example of this acid is carboxylic acids such as acetic acid and formic acid which can lead to form corrosion in low alloy of aluminium or steel during storage, transport or at usage of biofuel. Figure 1 shows the process of biofuel production from biomass by pyrolysis or liquefaction. It is very difficult to test corrosion behavior of biofuel during liquefaction or pyrolysis processes in laboratory because it take place in elevated temperature and high pressure and some parameter need to be controlled such as biomass feedstock, flow condition, catalyst and any other additives [4].

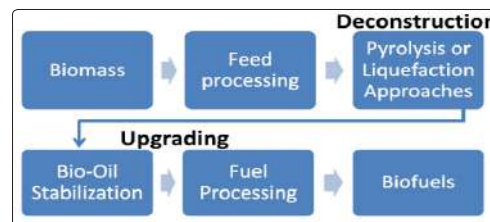


Figure 1: Biofuel production from biomass by pyrolysis or liquefaction [4].

Some techniques were used to determine qualitative and quantitative information about some elements such as Na, O, Cl, P, and S. These techniques were light microscopy, scanning electron microscopy with qualitative energy dispersive x-ray analysis (EDS), and electron probe microanalysis EPMA. Figure 2 shows the structure of cross section of the 316 stainless steel exposed for biomass liquefaction process [4].

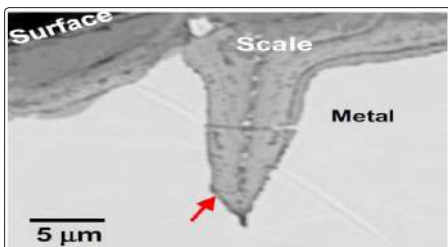


Figure 2: Stainless steel exposed for biomass liquefaction process [4].

The deposits or formed scale in 316 stainless steel which cause internal attack could continue growing during time then it is leading to cracking after short period of exposure (less than 1000h) which happened in different locations during liquefaction process for different biomass. Cracking was formed in inner surface of 316L tube then it was growing to the outer 6.4mm diameter as in figure3 [4].

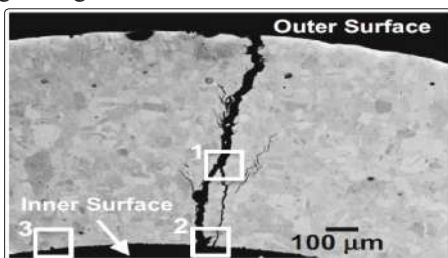


Figure 3: Cross-section backscatter mode SEM image of a cracked 316L stainless steel tube from a biomass liquefaction process system [4].

Moreover, figure 4 shows both EPMA maps and SEM image and they show the material of the crack was rich in iron, oxygen, sulfur, sodium, and chlorine species. Cl-attack and S-attack were the major issues [4].

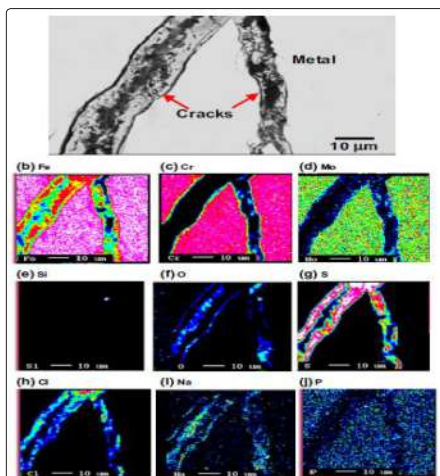


Figure 4: EPMA maps and SEM image show the material content of the crack was rich in iron, oxygen, sulfur, sodium, and chlorine species [4].

Corrosion of ethanol during production and distribution process

During the process of production of ethanol stainless steel is consumed in large amounts. An example of some parts which may be affected or corroded when they come in contact with ethanol are cookers, distillation columns, storage tanks and fermentation tanks. Automotive engine may also be affected by the purity and quality of the used fuel and it is very sensitive for different types of fuel. The oxygen is more soluble in ethanol by 10 times more than its solubility in water. Maximum amount of ethanol blends for automotive uses is allowed to be 10% unless if the cars were designed for E85. Therefore some studies are trying to reduce oxygen content to inhibit corrosion [5].

It is very important to choose suitable materials or stainless steel to construct production and storage vessels. One of the problems that ethanol may cause is stress corrosion cracking (SCC). Stainless steel can be used in constructions of most parts of ethanol plant, pipe fittings and in flat products because of its high resistance for corrosion due to the chromium oxide thin passive film that form in its surface. Higher corrosion resistance was found in higher chromium content and the lowest percentage required for corrosion resistance was 10.5% Cr. In addition, duplex stainless steel can be used for manufacturing equipment that more resistance for SCC. Moreover, stainless steel was used in constructing the vessel due to its high mechanical strength so that was reduced the wall thickness. Pitting corrosion was the most known corrosion form occurs at stainless steel and this problem increased with increasing in chloride ions and at high temperature [5].

Different Types of Corrosion Depending On Water Content in Bioethanol

The low water content in bioethanol and hydrocarbon mixture would affect the corrosion rate of carbon steel and sometime it can act as inhibitor. While 1 vol.% water content inhibited corrosion but when water content became 4% it was increased corrosion rate of steel [6]. So, water content in bioethanol fuel should be controlled during transportation and storage of alcohol fuel. Also, the increasing in of water content in ethanol led to increase the acidity which affected the corrosion rate and the mechanism of passive layer [6]. An electrochemical testing by using three electrode cells was effective way to study corrosion behavior of ethanol and hydrocarbon mixture [6].

Corrosion could be rapidly occurring if there was phase separation in ethanol fuel. 3% of water Presence in this fuel (or 9.5% at 20°C in bioethanol) was able to cause phase separation which then will lead to increase chlorides. This will dramatically corrode the tanks, reservoirs or lines that contain this fuel. Corrosion can be formed in horizontal or vertical surface and it will affect horizontal surfaces more than vertical surfaces due to different water and fuel density. Ethanol usually corroded the bottom surfaces because water was heavier in density [7].

There were different types of ethanol and gasoline blends and these types will lead to different degrees or forms of corrosion in different types of the metals or alloys. Some corrosion types can be general, dry or wet and they can happen in stainless steel, different aluminium alloys and brass alloys. Different types of corrosion depend on the impurities and the electrochemical damage it can cause. Also, these corrosion types depend in the water content in the blended fuel either separated or dissolved water. Some studies were done to test

electrochemical and alcoholate corrosion behavior of the metals and alloys in very high temperature and pressure [8].

General corrosion also known as uniform corrosion and it happen in storage tanks that contain biofuel. Water content in general corrosion can be 0.1% or above but, if it reach 0.5% it will cause phase separation which lead to wet corrosion. Third type of corrosion is dry corrosion which occurs if there is low water content in the blended fuel. Some elements such as Mg, Pd, and Al are sensitive to the ethanol in blended fuel. Aluminium is converted to alcoxides which is alcoholates. Then it can decompose easily and to avoid this type of corrosion low water 0.1% or 0.2% content can be used as inhibitor. The low water concentration that needed to inhibit corrosion rely on ethanol content of fuel blend, composition of the alloy and temperature. On the other hand, water content can has bad effect in wet and general corrosion if there are chlorides and organic acid dissolved in the fuel. There was test done to examine corrosion of the fuel containing water and it effect on some materials which usually used in the manufacturing of automotive engine. The test was for one year and the samples were subjected to direct sunlight during summer at 60 °C. Also, the samples were put in 150 °C and 130°C and the pressure was 10 bars. Different fuel content were used during the test. To prepare corrosion, chloride and acetic acid were added to E10 fuel. The fuel was changed every month during the test [8].

The results of the test shows that localized corrosion was formed in the long term exposure samples in the E10 fuel containing water. Figure 5 shows the behaviour of different specimen. The damage in general occurs in the immersed area of the sample and corrosive attack formed in the liquid phase. Also, figure 5 was clearly showing that the conventional gasoline was the less corrosive fuel compared to other two blended fuels used in the test. While E10 ASTM fuel C which was contained acetic acid and chlorides impurities was the most corrosive fuel. But the fuel which has addition of chlorides showed pitting corrosion with less damage [8].

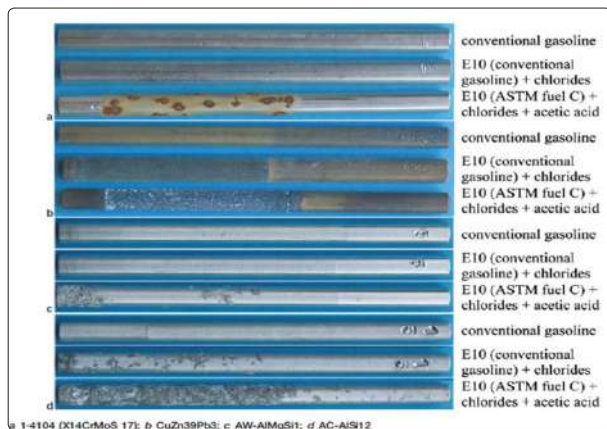


Figure 5: Appearance of steel, brass, wrought aluminium and cast aluminium after exposure for 1 year at ambient temperature in specified fuels [8].

Another result from the tested samples was that cast alloy AC-ASi 12 shows more pitting and corrosion than wrought aluminium alloy AW-AlMgSi1. For the brass alloy CuZn39Pb3, the Corrosion attack found more in the zinc rich phase. When aluminium alloys investigated in E10 gasoline blend and in approximately water free dry ethanol fuel, the temperature was very important factor affecting alcoholate corrosion. Above 130°C alcoholate corrosion

will occur but the composition of the alloy will not affecting this type of corrosion [8].

From that it is clearly observed that both material composition and fuel content were affecting the wet corrosion behavior. The higher dissolved impurities such as chlorides or acetic acid was the most corrosive fuel. While, alcoholate corrosion of the fuel that contain ethanol and removing water and impurities was mainly depending on the temperature [8].

Studying corrosion characteristics of metallic materials (by eis) in gasoline blended with ethanol and water:

Electrochemical impedance spectroscopy (EIS) testing was used to determine corrosion behaviour of the samples exposed to gasoline containing different percentage of ethanol 0%, 5%, 10%, and 15%. In addition, scanning electron microscope was used to determine the types of corrosion attack from the properties of specimens' surface. Addition of ethanol to gasoline leads to increase corrosion rate because of decreasing both solution resistance and polarization resistance value of the samples. From all of the tested samples, Al6061 was very suitable for corrosion resistance [9].

Five different samples were tested under EIS measurements. These samples were low and medium carbon steel, copper, Al 6061 and 304 stainless steel as in table (1). Different solutions used in the test were shown in table (2) [9].

Table (1): Different surface area, material and component for samples used in EIS measurement [9].

Solution Nos.	Composition
1	Gasonline + 0% ethanol
2	Gasonline + 5% ethanol
3	Gasonline + 10% ethanol
4	Gasonline + 15% ethanol
5	Gasonline + 0% ethanol + 1% water
6	Gasonline + 5% ethanol + 1% water
7	Gasonline + 10% ethanol + 1% water
8	Gasonline + 15% ethanol + 1% water

Table (2): Different solutions used in the test [9].

Specimen Nos	Component	Material	Surface area (cm ²)
1	Return fuel tube	Stainless steel 304	1.10
2	Fuel outlet tube	Low carbon steel	3.24
3	Transferring tube bolt	Medium-carbon steel	3.14
4	Main Fuel delivery tube	Al 6061	2.64
5	Washer	Copper	0.33
6	Brazed part	Brazing alloy	0.77

Addition of ethanol and water to gasoline as in solutions number 5, 6, 7 and 8 leads to decrease solution resistance (R_s) and polarization resistance (R_p). That means lower corrosion resistance. From EIS measurements showed that polarization resistance was depending on

the chemical composition of the samples. From figure 6 it was clearly shows different corrosion rate of different materials were dependent on ethanol content in gasoline blend. From that test Al 6061 and copper were the best two materials that can resist corrosion. While, low and medium carbon steel showed lower corrosion resistance because of high content of ferrite in them. Al 6061 showed lower corrosion rate and brazing alloy had lower corrosion attack. The materials which can be used with 10% ethanol or less were copper and stainless steel 304 [9].

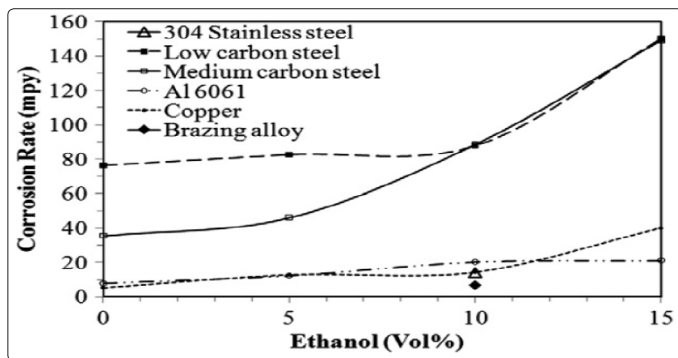
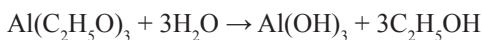
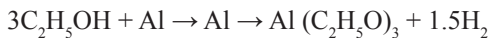


Figure 6: Corrosion rate of different materials in different blends of ethanol and gasoline [9].

Corrosion of Aluminium Alloys In Ethanol Fuel

Corrosion of different aluminium alloys immersed in ethanol was studied with different temperatures, various water content and ethanol content. Aluminium alloys which tested were the following AlSi17-Cu4Mg, AlSi8Cu3 and AlSi7Mg. These alloys were tested because of their mechanical properties and many new engines were made from these alloys [10]. The following equations represent the dry corrosion occur in free water media:



The results of the test done in dry corrosion showed that water content and temperature both had big effect on corrosion while, ethanol has minor effect. But when ethanol content increased the number of pits in the metal increased which may lead to failure. Another observed result was that nickel form protective passive layer that reduced corrosion rate. Also, the study [10] found that the critical temperature of corrosion was the boiling point of ethanol which is 78.4 °C and near to this temperature the corrosion will increase.

Corrosion behaviour of aluminium alloy tested with ethanol solutions:

From previous studies, they found that aluminium was not compatible with ethanol. Although, in North America they used fuel containing 85 vol.% ethanol (E85) in auto industry. The electrochemical mechanisms of corrosion in organic solvent such as ethanol were the same as the mechanism in aqueous solutions. Although, the most ethanol corrosion studies were done for aluminium at high temperatures (dry corrosion) which were higher than boiling temperature of ethanol (>78 °C). But automotive parts usually exposed to bioethanol in longer time compared to running time. For example, parking time and bioethanol in the pipeline which occurs

at room temperature. So, it is very important to study corrosion and the effect of bioethanol and aluminium alloy at room temperature or temperature lower than boiling point of ethanol. Aluminium usually comes in contact with ethanol because fuel pump, engine blocks and pipe to deliver ethanol are made from Al [11].

Corrosion of aluminium AlSi8Cu3Fe(Zn) alloy was tested with ethanol solutions. One of the Solutions had 10 vol.% water and the other solution had 10 vol. % acetic acid. Water and acetic acid were blended or added to anhydrous ethanol to prepare these solutions at volume ratios of ethanol:water=9:1 and ethanol:acetic acid=9:1. The test was done in temperature below boiling temperature of ethanol. EIS is the famous technique or instrument that is used to determine corrosion of the metal in aqueous medium such as Al corroded by ethanol especially when corrosion resistance is high. When aluminium exposed to bioethanol it formed protective oxide or hydroxide layer on Al surface. This thin protective film play very important role in controlling corrosion and corrosion resistance in Al. Another result from EIS was that corrosion was formed in film defects areas like in the pores or cracks in the film. Corrosion bath was same for both aqueous solution and Al in bioethanol [11].

Figure 7 shows the logarithmic current density graphs. The graphs show that both solutions of ethanol and water blended ethanol were having close corrosion potential. However, acetic acid blended ethanol had more positive corrosion potential. Also, from Figure 7 it can be observed that water addition to ethanol leads to enhance anodic polarization current density. For acetic acid addition it enhanced both cathodic and anodic reactions [11].

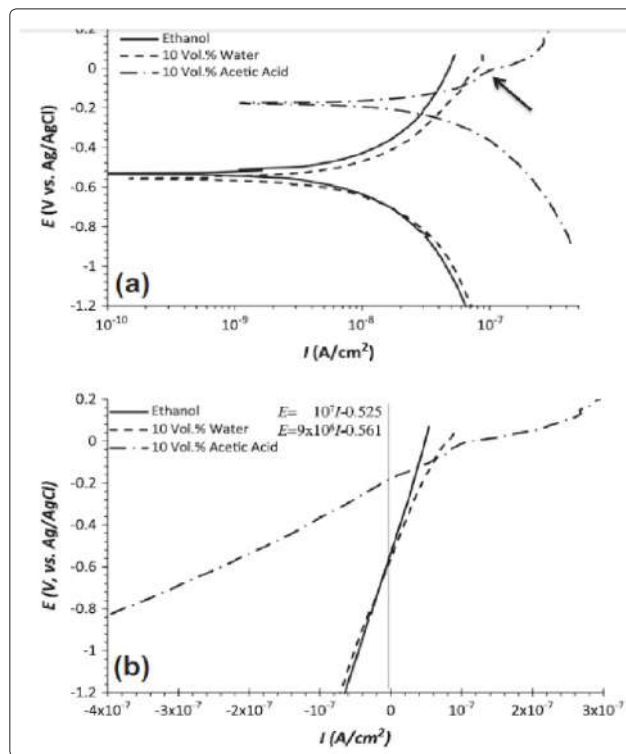
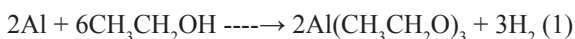
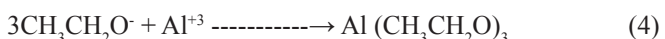


Figure 7: a) logarithmic current density graphs b) linear current density graphs for aluminium tested with ethanol, ethanol containing 10 vol.% acetic acid and ethanol containing 10 vol.% water [11].

When Al tested with ethanol contain no water alkoxidation reaction take place as in equation (1):



This reaction was accelerated at high temperature when it occurs in temperature more than boiling point of ethanol (dry corrosion/alkoxidation). While, in low temperature from room temperature to 70°C another electrochemical mechanism was take place (wet corrosion/alkoxidation) as in equations (2), (3) and (4):



When water and acetic acid were blended with ethanol corrosion mechanisms changed and additional reaction occurred. Water and acetic acid were reacted with aluminium oxide on the film formed on Al surface as in equations (5) and (6):



The effect of water content in ethanol in dry corrosion/alkoxidation was different from it is effect on wet corrosion/alkoxidation. Another factor can affect corrosion was oxygen content. The effect of oxygen from environment was worse than the effect of water in corrosion. In addition, alloying elements such as Cu, Fe, Mn and Mg can play important role in corrosion. Because they can cause corrosion damage which were distributed irregularly. Figure 8 shows aluminium sample before and after corrosion. After corrosion, there was black area of corrosion products which was precipitate of Al-Cu-Si-Mg. Then these precipitate spreads on the surface of the metal and it go deeper in the metal [11].

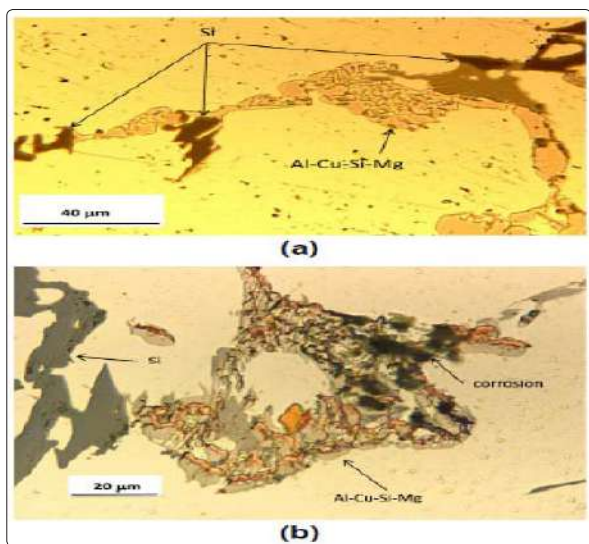


Figure 8: Microstructure of Al alloy (a) before and (b) after immersion test [11].

Higher corrosion was take place at higher temperature which led to more material loss in the areas affected by heat. Moreover,

Corrosion can be accelerated by increasing carbon and oxygen content in the fuel as in bioethanol. From the experimental test it was observed that adding 10 vol. % of acetic acid to ethanol was more corrosive than adding 10 vol.% of water. Also, the precipitates which contain copper were more corrosive because of the microgalvanic effect. In general water content in ethanol was increased with time during storage [11].

Corrosion Behaviour of Aluminium Tested At High Temperature in Bio ethanol Blended Gasoline

Ethanol was used to reduce the emission of greenhouse gas. Ethanol was blended with gasoline to enhance octane number, because ethanol used to replace methyl tertiary butyl ether (MTBE). MTBE is prohibited because it is harmful for human health. It is important to study the effect of bioethanol on the material used to make automotive parts such as stainless steel and aluminium [12].

Corrosion behavior of cast alloy Al (A384) was tested with different ethanol contents blended with gasoline. The contents of ethanol were 10%, 15% and 20% which were tested at 60, 80 and 100°C. At high temperature (100°C) there was more localized pitting. Corrosion was increased by increasing both temperature and ethanol content. Increasing temperature led to change in the structure of the hydroxide protective film. This protective film was formed at specific temperature and ethanol content [12].

Figure 9 showed the surface morphology of the corroded aluminium sample which was immersed for 24h at 100°C in gasoline contain bioethanol. The white color was corrosion product which was $\text{Al}(\text{OH})_3$. There was no corrosion damage below 100°C, but there was localized pitting corrosion occur at 100°C at all gasoline-ethanol blend containing different ethanol content [12].

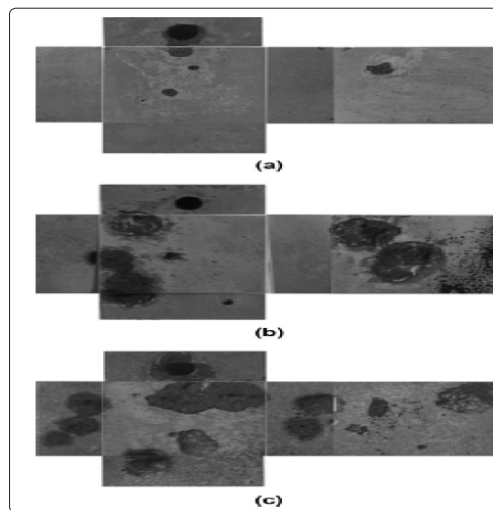


Figure 9: Surface morphology of the corroded aluminium samples tested at 100 °C in (a) 10%, (b) 15% and (c) 20% ethanol/gasoline fuel [12].

From the figure, localized pitting corrosion was formed on the surface of the sample and the number of pitting was increased when ethanol content was increased. Also, corrosion was increased with increasing ethanol content and temperatures as in bar chart in figure 10 [12].

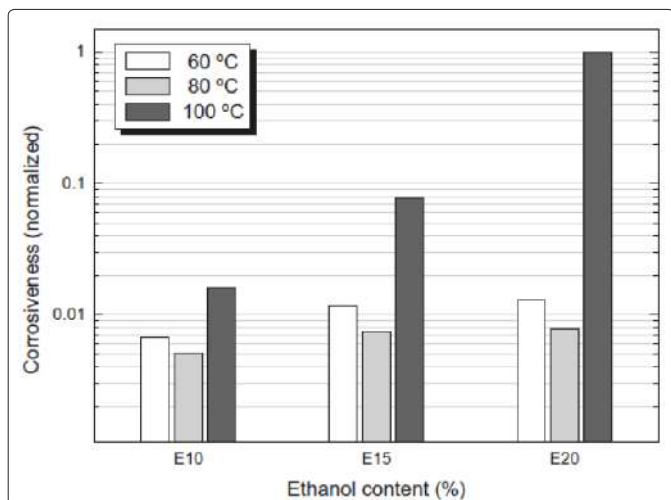


Figure 10: The corrosiveness of Al in bioethanol/gasoline blend with different ethanol content (E10, E15 and E20) at 60, 80 and 100°C [12].

Corrosion of aluminium in bioethanol blended with gasoline was happened due to phase separation theory. Ethanol has hydrophilic property which lead to form two phases of water-ethanol and gasoline which leads to form corrosion. When aluminium reacts with ethanol the product from the reaction will be aluminium alkoxide and hydrogen gas. This hydrogen atom that came from OH of ethanol will replace Al atom in the metal or oxide film. So, Al will not be protected [12].

The effect of temperature change in the weight loss is shown in the Figure 11. It showed the effect of E20 on Al immersed for 12 h at different temperatures. There was slight increase in weight loss from 60 to 80°C, but after 80°C there was significant increase in the weight loss. That means corrosion increased with increasing temperature and in the test below 80°C corrosion reactions was slow. Also, the corrosion of protective film was depending on temperature [12].

The Effect of Bioethanol Produced In Misiones on the Corrosion Resistance of Aluminium Alloys

It is essential to study the effect of bioethanol on aluminium during manipulation, storage and transportation. Because aluminium has good mechanical properties and it has low density comparing with pure aluminium, aluminium which alloyed with silicon has better resistance and aluminium which alloyed with magnesium has better hardness. Aluminium alloyed with silicon and which alloyed with magnesium was tested with bioethanol from fermentation of sugar cane produced in Misiones to study their corrosion behaviour. Aluminium was not compatible with bioethanol. Aluminium alloys were used as structural material in many industries related to bioethanol due to its mechanical properties [13].

ESI was used to study the corrosion in aluminium exposed to bioethanol. The material which were used in the test were aluminium 99.9%, aluminium alloyed with silicon (Al-10wt.%Si and Al-12wt.%Si) and aluminium alloyed with magnesium (Al-12wt.%Mg and Al-4wt.%Mg). The bioethanol which was used as electrolyte in the test produced in Misiones. Potentiodynamic polarization methods were used to analyze corrosion resistance in the tested aluminium alloys. Immersion test of the samples in the electrolyte was done

for 16 h then these samples were analyzed by EIS technique [13].

Figure 11 shows the corrosion potentials which was differ for different alloys. For aluminium silicon alloy, corrosion potential increased with increasing silicon content. While, for magnesium alloyed with aluminium there was no change with increasing magnesium content [13].

Another result for the test was that the potential for the entire sample was increased then it stabilized. That indicates the presence of protective porous oxide layer on the surface of the sample in corrosive bio ethanol. Also, the analyzed results showed that Mg-Al alloy (Al_3Mg_2) was more active compared to pure aluminium. Mg-Al alloy (Al_3Mg_2) can react with O_2 which was diffused to the interface between metal and solution. So, cathodic reactions was controlled by O_2 diffusion. Also, thick layer cause difficulties for active species during diffusion [13].

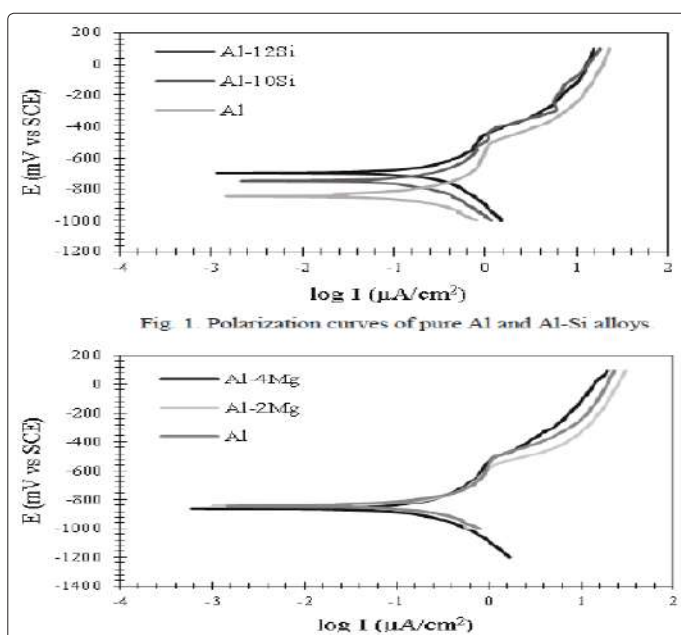


Figure 11: Comparing the results of potentiodynamic polarization for pure aluminium, Si-Al alloy, and Mg-Al alloy [13].

After analyzing some results from immersion test was that the alloying element such as Si and Mg cause change in corrosion behavior. Also, when the sample exposed for longer time they form resistive layer. From EIS results, the formed layer on the samples surface was protective, porous and semipermeable. Al-10wt. %Si and pure Al have better resistance behavior compared to other samples [13].

Characteristic of martensitic stainless steel when it corroded by ethanol blended with Gasoline and has another contaminations

Ethanol containing gasoline and other contaminants such as acetic acid, water and chloride was studied to know its effect on the stainless steel DIN 1.4035 (Fe-13%Cr). Chloride and water contents both were affecting corrosion rate and there was critical water content that caused pitting corrosion. Critical water content was depending in ethanol/ gasoline ratio. While, acetic acid has minor effect on corrosion rate. Higher acidity and pitting propagation were increase when chloride concentration was increasing. But when acetic acid

concentration increased that was affecting attack area of corrosion which were in proportional relationship [14].

Water content was increased with increase ethanol content in ethanol/gasoline blend. That was accelerating corrosion rate because of phase separation between water and gasoline which cause increasing of hydrophilic impurities like chloride. In ethanol/gasoline (v/v%) blend (E20), there were some alloys or material that corroded faster such as copper and carbon steel. While there were some material that were more resistance to corrosion like aluminium alloys and stainless steel. At low concentration of H₂O and higher temperatures passive film was formed on the surface of aluminium alloy and that worked as protective layer. So, there is specific water content for passivation and it exceed critical concentration that will lead to breakdown the passive film and form corrosion [14].

Figure 12 showed corrosion of two samples immersed at room temperature in E24 ethanol/gasoline blend contained 20 ppm chloride (Cl⁻) and 30 ppm acetic acid for four weeks. Water content in fuel blend was different for these two samples. For first sample, water content was low (12,000) and it did not formed phase separation. There was single phase solution which leads to homogenous pitting on sample surface. While, in the presence of high water content (50,000 ppm) in the fuel there were two phase solution and that formed different corrosion pitting and morphology on the sample surface [14].

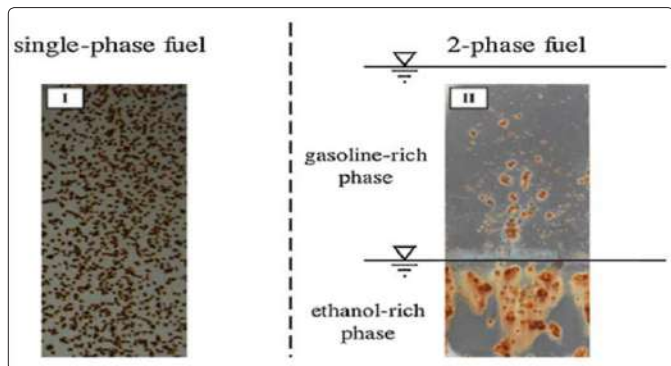


Figure 12: Corrosion morphology of two samples immersed at room temperature for four weeks in E24 ethanol/gasoline blend contained 20 ppm Cl⁻ and 30 ppm acetic acid. Water content in fuel blend was different for these two samples in (I) H₂O: 12,000 ppm and in (II) H₂O: 50,000 ppm [14].

Another test was done to study corrosion behavior of stainless steel immersed for 28 days in three different solutions which were water, E24 (ethanol/gasoline blend + 12,000 ppm water), and ethanol (E100) as in figure 13. These three solutions were contained same concentrations of acetic acid (30 ppm) and of chloride (20 ppm). The concentration of acetic acid and chloride were low and not enough to breakdown passivity of steel in both E100 and in H₂O solutions. From that it was clearly that there should be critical concentration for chloride to be able to cause pitting corrosion in stainless steel. In figure 14 there were six samples of stainless steel immersed for 28 days in in blended fuel contained 30 ppm acetic acid and 20 ppm chloride with different ethanol content 10%, 24%,40%, 55%, 85% and 100%. In 10% there was phase separation. At 24% ethanol content there was pitting corrosion. On the other hand, there were no corrosion in both samples immersed in E85 and E100. The effect of chloride and acetic acid concentrations contents in the ethanol

blend on corrosion of stainless steel was shown in figure 15 and 16 respectively [14].

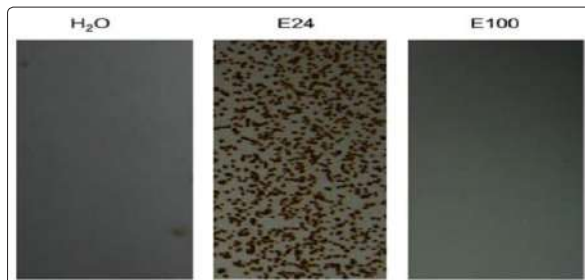


Figure 13: Corrosion behavior of stainless steel immersed for 28 days in water, E24 and (E100) (containing 30 ppm acetic acid and 20 ppm chloride) [14].

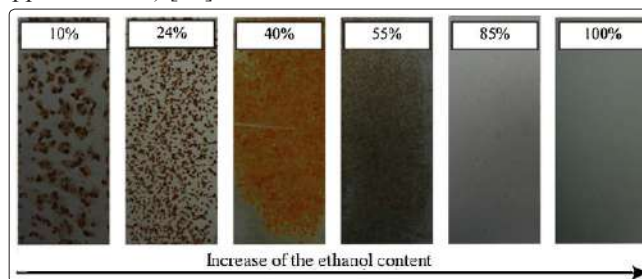


Figure 14: Samples of stainless steel immersed for 28 days in in blended fuel contained 30 ppm acetic acid and 20 ppm chloride with different ethanol content 10%, 24%,40%, 55%, 85% and 100% [14].

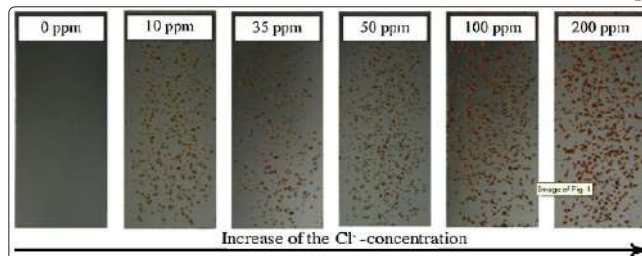


Figure 15: Samples of stainless steel immersed for 28 days in ethanol/gasoline blend contained different concentrations of chloride [14].

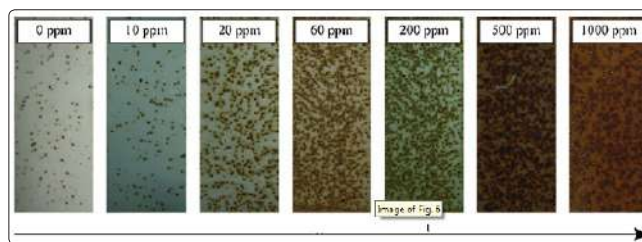


Figure 16: Corrosion behavior of samples of stainless steel immersed for 28 days in ethanol/gasoline blend contained different concentrations of acetic acid [14].

Corrosion Behaviour of Steel in Presence of Carbon Dioxide and In Different Bacterial Communities

Anaerobic was suitable environment for microbial communities' growth. Especially in the presence of sources of carbon, electron source such as steel and electron acceptor as carbon dioxide. Corrosion rate was increased by 45.5% in culture more than in sterile controls for 2 months. Pyrosequencing indicate that all of sulphur reducing bacteria (S⁰RB), sulphate reducing bacteria (SRB)

and acetogens were growing in a syntrophic relationship. SRB can grow in both aerobic and anaerobic mediums. Electron can be extracted from iron by sulphate reducing bacteria. Then acetate was produced as result when acetogens accept electrons and reduce CO_2 to acetate. Microbially influenced corrosion (MIC) is one of corrosion problems that happened in steel infrastructure and that lead to high cost in industrial or petroleum sectors. Many different types of microorganisms can increase corrosion rate of steel such as archaea, bacteria and fungi. These microorganisms could lead to galvanic formation and different aeration which increase the acidity, form enzymes and consuming electrons from steel. Also, these microorganisms can form biofilm which accelerate corrosion rate. Bacteria need carbon source, electron source and acceptor [15].

An example of cultured microbial communities was *broe* (BR) and *soil* (SL). The results from ion-exchange chromatograph showed that the nitrate and sulphate were more in abiotic controls when compared with culture. The effect of the communities was studied for the elements that were cycled between species when phosphate was added. In BR culture there was recycling of carbon and recycling of iron between species. In natural communities, microbial communities can extract electron from steel and that was contribute to recycle element and syntrophic growth. Some example for metals and alloys that help to increase growing these communities and increase corrosion rate were 316 stainless steel, low carbon steel, pure iron and ferrous iron. Comparing to abiotic control, these communities can increase corrosion rate of low carbon steel by 45.5%. This type of study was focusing in the causes and conditions that accelerate corrosion rate or MIC. Then there were some effort to find ways to prevent it [15].

Biodiesel

Corrosion Behaviour of Copper In (Bde) Blended Fuel

The corrosion behavior of the copper samples was tested to know the effect of fuel blend contains biodiesel-diesel-bioethanol (BDE). B45D35E20 is representing the percentage content of the component of the fuel blend as following 45% biodiesel, 35% diesel and 20% bioethanol. The duration of the test was for 408 hours and the test was done in temperature range from room temperature around 25°C to 50°C . The sample that used in the test was pure flat plat of copper sample (99.99%) as in Figure 17. Immersion test was done to compare some properties of the copper sample before and after the test such as chemical, mechanical, and physical properties. Also, after immersion test some other changes were tested such as the changes that may happen in mass and volume, chemical structure and morphological changes [3].



Figure 17: Copper specimen and blended fuel of (BDE) [3].

The results of the test done to study the corrosion behavior of copper sample immersed in B45D35E20 fuel blend showed that the corrosion rate in pure diesel (B100) is higher than the corrosion rate of B45D35E20. Immersing the copper sample in blended fuel of BDE caused pitting on the sample at 25°C to 30°C which caused

by mono-carboxylic acid and the presence of water. This means high oxygen content that lead to formation of cupric oxide CuO/CuCO_3 in outer layer and Cu_2O in inner layer. The pitting become black because of the oxide layer formed on the copper at room temperature which was blacker than pitting formed on copper at higher temperatures. At higher temperature 50°C the oxide layer become thinner than at 25°C because the protective layer of oxide dissolved at high temperature. That means corrosion of metals increase at higher temperature which the table 3 shows.

Table 3: The corrosion rate of copper immersed in B45D35E20 [3].

Temperature	W (mg)	D (g/cm^3)	T (h)	A (in^2)	Corrosion rate (mpy)
Room Temperature	10	8.96	408	5.274	0.277
50°C	12	8.96	408	5.354	0.327

In addition, the hardness of the copper also change after immersion test. In general it is increase after immersion test and it was hardener at room temperature than at 50°C as in table 4.

Table 4: Change in hardness of copper in different temperatures [3].

Temperature	Load (g)	Dwells (s)	Vickers Hardness (HV)	Vickers Hardness (MPa)
Before	500	10	1.27	12.45
After in room Temp	500	10	56.5	554.10
After in 50°C	500	10	41.9	410.90

After immersion test the properties of the blended fuel were tested such as fuel's color change and finding total acid number (TAN) by titration. The TAN values was 1.368 mg NaOH/g for the fuel before testing and it was increased to 1.473 in room temperature and 1.579 at 50°C . From Figure 18 the color of fuel was yellow before testing then it converted to greenish color which caused by transformation of copper species during change in oxygen content and temperature of the fuel. From all previous results, B45D35E20 blend can be used as fuel for diesel engine at 60°C and no need for corrosion inhibitors [3].

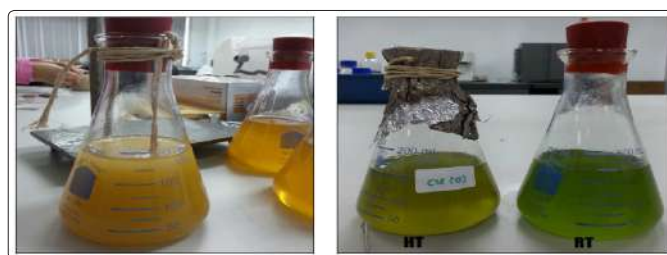


Figure 18: Changes in the B45D35E20 fuel's color before and after immersion test at 408 h [3].

Corrosion Behaviour of Aluminium Exposed To A Biodiesel

Biodiesel can be produced from biological sources or by converting animal oil or vegetable oil to biodiesel by transesterification process. Strong base such as NaOH or KOH always used as catalyst in the transesterification process. After that some impurities were removed to enhance the quality of biodiesel such as water, fatty acid, catalyst and glycerol [16].

It is very important to remove these impurities because it may affect some parts of the automotive industry like pistons and engine. Many parts in automotive industry are made from aluminium because this metal has high strength to density ratio which make it very useful in the industry. Aluminium alloy can be corroded by biodiesel and corrosion behavior and resistance is depending on the pH of biodiesel. When the pH increased and biodiesel became more alkaline, corrosion resistance of aluminium decreased which led to dissolve oxide layer on the aluminium surface. The real problem was that the catalyst which was used during transesterification process was alkaline. The solution was to wash biodiesel to remove any impurities or contaminations. Corrosion behavior of aluminium exposed to biodiesel was tested and it gave a quantitative indication about the purity of the used biodiesel. Also, corrosion behavior investigated during different stages of biodiesel's washing process [16].

The biodiesel used for the test was extracted from canola oil which was taken from the supermarket. In the experimental part a conventional three electrode cell was used and 99.999% pure aluminium was used as working electrode. The auxiliary electrode was Pt foil. While, the reference electrode was Ag/AgCl. The test was done by immersing of aluminium in biodiesel and measure the E_{ocp} (open circuit potential) with time as in the figure 19 [16].

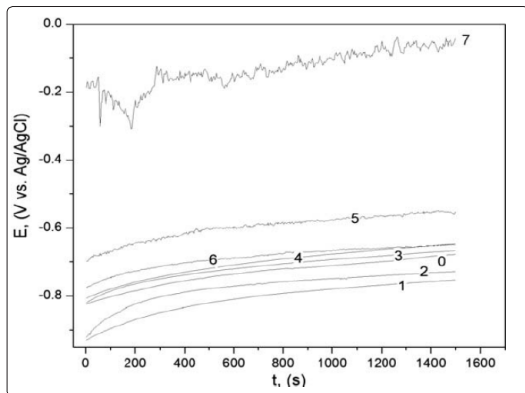


Figure 19: Open circuit potential of pure aluminium exposed to biodiesel after a different number of wash cycles [16].

In general, E_{ocp} was negative value then it started to increase with time. E_{ocp} value reach below -600mv for the first two washing cycles. This shows that there were some impurities in biodiesel which start reacting with aluminium surface and start forming corrosion products on the Al surface. When aluminium immersed in alkaline solution such as solution contains KOH or NaOH, it started forming oxide layer which was Al(OH)₃. While, after several washing cycles as in the Figure 19 and at last cycle the E_{ocp} become positive with time. Another result was that, there was decrease of corrosion current density i_{corr} with number of wash cycles. But high i_{corr} for the first two cycles was because during these two washing cycles the contaminants were not removed from biodiesel. From these results corrosion of aluminium can be used as quantitative indicator for the corrosivity of biodiesel. In addition, corrosion behavior of biodiesel that has alkalis impurities in was similar to corrosion occur in aluminium in the presence of aqueous solutions [16].

Behavior of Palm Biodiesel and Petroleum Diesel on Automotive Material

It is important to study the effect of biodiesel and petroleum diesel on engine parts which are exposing to diesel for long time. There

was a test done to study corrosion behavior of some metals when they exposed to biodiesel (B100) and petroleum diesel (B0). These metals were stainless steel, copper and aluminium and the test was done at 80°C for 1200h. Corrosion behavior were investigated by measuring the loss in the weight and changes on metal surface [17].

Palm biodiesel and petroleum diesel were tested to see their effects on corrosion behavior when they come on contact with different metals which were Cu, Al and stainless steel. These metals were choosing to the test because they are usually used in broad range in manufacturing automotive parts. When they come in contact with diesel or biodiesel they were reacting together and cause fuel degradation. The specimens which used in the test were copper 99.99%, aluminium 99% and 316 stainless steel containing Cr, Ni, Mn, Si, and C with following percentages respectively 18%, 11%, 2%, 1%, 0.08%. These specimens were immersed in the palm biodiesel (B100) and in petroleum diesel (B0) at 80°C for 1200 hours [17].

Corrosion rate

The result of the corrosion rate was shown in the Figure 20. There were big corrosion rate differences for copper and aluminium in both B0 and B100 while corrosion rate of stainless steel was very low in both biodiesel and diesel. Corrosion rate of Cu and Al were higher in B100 more than in B0. Biodiesel was more corrosive than diesel because of the unsaturated acid component that present in B100. Also, B100 more corrosive due to water content and TAN number which increased when biodiesel was oxidized. Another observation was the increasing in corrosion rate of copper at higher temperature and longer exposed time. At higher temperature the motion between the metal and fluid increase which lead to higher corrosion rate. Palm biodiesel was slightly more corrosive than fat biodiesel [17].

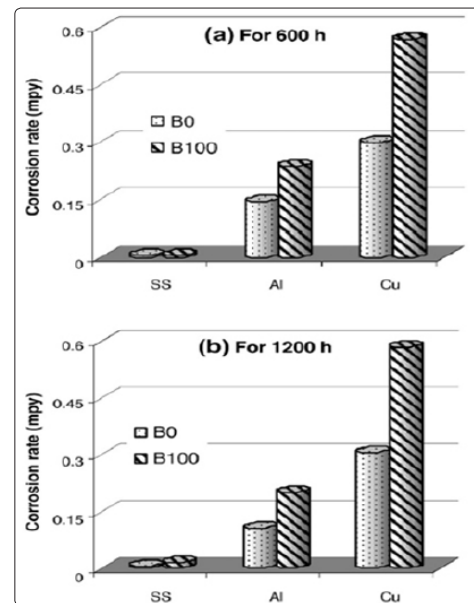


Figure 20: Corrosion rate of SS, Al and Cu immersed in diesel and biodiesel at 80°C for (a) 600h and (b) 1200h [17].

Surface morphology

There was no big change in the surface of stainless steel in both biodiesel and in the diesel. While, there were high pitting corrosion in the surface of both aluminium and copper when they exposed to biodiesel as in Figure 21. In general pitting corrosion was higher

in copper more than in aluminium and stainless steel. That was because the replacing of oxygen from Cu_2O with CuO layer from copper surface [17].

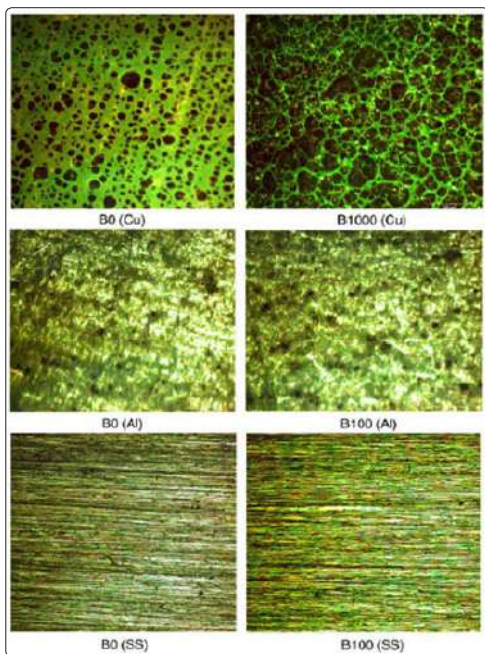


Figure 21: Surface morphology of stainless steel, copper and aluminium after they were exposed to B0 and B100 for 1200h at 80°C [17].

Compositional changes

Figure 22 shows the change in the composition of both B0 and B100 when they come in contact with copper, aluminium and stainless steel at 80°C for 120h. Copper shows large changes in color compared with other metals when it exposed to both B0 and B100. That was an indicator for presence of metal species in the fuel and change in the diesel and biodiesel compositions. Compared with diesel, the results of the test showed that metal species content were more and high in biodiesel. So, biodiesel was more corrosive than diesel. For example, Cu species were more in biodiesel and copper was more reactive in biodiesel. There were different types of copper oxides formed which gave different color. Such as pale green which indicate presence of copper carbonate in biodiesel. The presence of red color due to cuprite oxide in the diesel. In addition, aluminium was behave like copper when Al presence in biodiesel more than in diesel. The presence of metal species in the fuel may degrade fuel composition. Another result was that, biodiesel can oxidize itself and metal species can react as catalyst in that reaction. Total acid number (TAN) was differing before and after B0 and B100 exposed to different metals. In general TAN was increased for both Al and Cu in same trend after they were exposed to B0 and B100. While corrosion rate for both Al and Cu were different, their TAN have same trend which means that metal play role in determining the corrosiveness of the fuel. TAN was higher in biodiesel and that is mean oxidation is related to TAN. Copper worked as strong catalyst to oxidize biodiesel. Moreover, water was important factor that increase fuel corrosive and biodiesel was more corrosive because it can absorb more water. In the beginning of the test there were no water content in both as-received B0 and B100 samples. But water content increased after immersing test of different types of metals. Water content was similar for different metal immersed in biodiesel

which indicated that water content was not depending in the type of the metal. Water can increase corrosion due to it help the growth of microorganisms. Also, when water presence in the biodiesel at high temperature that can lead to hydrolyze esters and triglycerides and that will increase the corrosion rate due to different types of fatty acid that can produce different types of fatty acids. Hydro carbon of biodiesel was not causing corrosion but the corrosivity of biodiesel was due to the presence of fatty acid components, water and oxygen. But there was no change in the surface of stainless steel [17].

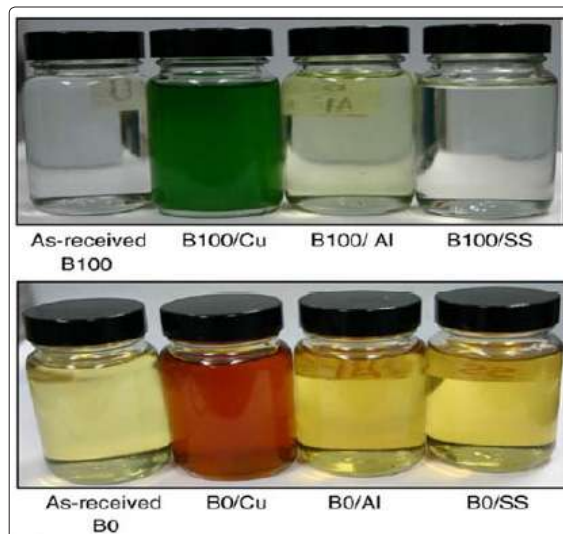


Figure 22: Change the compositions of biodiesel and diesel when they exposed to AL, Cu and SS [17].

Palm Biodiesel Degradation and It Is Effect on Automotive Materials

Automotive engine parts are made from ferrous and non-ferrous materials which come on contact with diesel or biodiesel. Corrosion products from different types of materials of automotive parts were studied to see their effect on the corrosion rate. The test was done by immersing copper, brass, and cast iron in palm biodiesel or in biodiesel. Then fuel composition was tested by identifying total acid number TAN, density and viscosity. From these tested material copper produce more corrosion products compared with other materials. The experimental testes showed that palm biodiesel was more corrosive than diesel due to moisture absorbance, auto oxidation and the presence of other contaminations. It is very important to detect corrosion products to give information about how materials degraded in biodiesel. For example, when copper immersed in biodiesel can convert fuel colour from colorless to green which means there was change in fuel properties [18].

The material which were used in the test were Cu 99.9%, brass (58.5% Cu and 41.5% Zn), Al 99.9%, cast iron which contain C, Si, Mn, P and S in this percentage respectively 3%, 1.84%, 0.82%, 0.098%, 0.089% and Fe: balance. The test takes place for 2880h at different room temperatures from 25°C to 27°C and in palm biodiesel (B100) and petroleum diesel (B0). The weight of the coupons was measured before and after immersion test. Surface morphology also was compered for each sample before and after testing. Figure 23 showed corrosion rate for copper (Cu), aluminium (Al), cast iron (CI) and brass (BS) which tested at room temperature for 2880h in B0 and B100. In general, corrosion rate for biodiesel was more than diesel. Copper had higher corrosion rate (0.39278 mpy) then

barss (0.209898 mpy). While cast iron had lower value for corrosion rate which was 0.112232 mpy and for Al it was 0.173055 mpy [18].

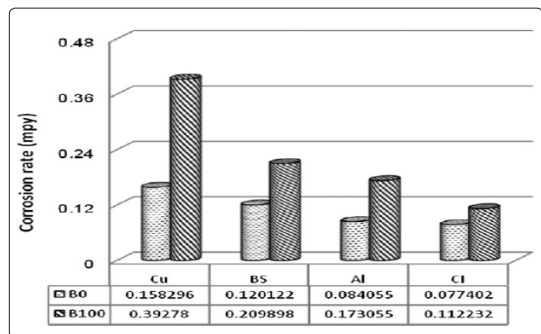


Figure 23: Corrosion rate for copper, aluminium, cast iron and brass which tested at room temperature for 2880h in B0 and B100 [18].

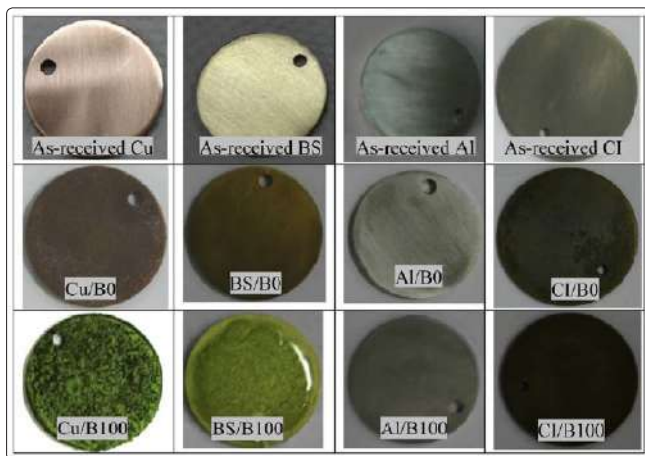


Figure 24: Surface morphology for copper, aluminium, cast iron and brass that tested at room temperature for 2880h in B0 and B100 [18].

From Figure 24 it was clearly seen that there were change in the color of the materials before and after the test. Copper was brown and brass was yellow then green layer formed in both materials in biodiesel. But both converted to reddish color in diesel. Cast iron converted from grey to reddish to black in biodiesel. However, there was no change in Al colour in B0 and in B100. The results from XRD indicate that when copper or copper alloys exposed to biodiesel they was forming pale green CuCO_3 . But in diesel cuprite oxide was formed which has red colour. These changes in the colours of different materials means there were changes in fuel content as in Figure 25. Another result from the tested samples was there were change in the density and viscosity of both B0 and B100 tested at room temperature for 1880 h [18].

Biodiesel content of oxygen was 10 to 12 wt% but, diesel did not contain oxygen. In biodiesel metals oxidized easily to produce different metal oxides and different metal compounds (high oxygenated species) which could accelerate corrosion rate. In [18] M.A. Fazal said " XRD results show that diesel exposed copper surface contains only a small concentration of CuO , Cu_2O and $\text{Cu}(\text{OH})_2$ formation while biodiesel exposed copper shows much higher amount of CuCO_3 along with $\text{Cu}(\text{OH})_2$, $\text{Cu}(\text{OH})_2 \cdot \text{H}_2\text{O}$, $\text{CuCO}_3 \cdot \text{Cu}(\text{OH})_2$, CuO and Cu_2O . Brass forms ZnO and CuO in diesel and relatively higher concentration of CuCO_3 , $\text{Cu}(\text{OH})_2$, ZnO , CuO and Cu_2O in biodiesel".



Figure 25: Change in color and compositions of biodiesel and diesel when they exposed to AL, Cu BS and CI [18].

Comparative of corrosion behavior of magnesium and aluminium in palm biodiesel

Using of light metals in automotive industry was increased during last years because it can reduce energy consumption and it play important role in enhance engine performance. For that, aluminium and magnesium start used to replace heavy weight metal like cast iron. The improvement in fuel consumption was changed from 1.9% to 8.2% and in the same time the reduction of the vehicle weight reached 10%. Some engine component which made from aluminium were piston, cylinder, transmission and piston ring. While, some engine parts were made from magnesium such as valve covers, housing, cylinder head cover and intake manifolds. All of these parts that made from magnesium or aluminium were coming in contact with biodiesel and the corrosivity of biodiesel is more than conventional petroleum diesel. Aluminium can resist corrosion in biodiesel more than other metal such as copper or brass alloy. Magnesium has same strength as aluminium but, magnesium is lighter than aluminium by 35% [19].

Aluminium and magnesium were exposed to palm biodiesel for 1440h and then both samples were tested by x-ray diffraction (XRD), total acid number (TAN) and Fourier transform infrared spectroscopy (FTIR). The content of aluminium alloy that used during testing were 95%, 3.86%, 0.55%, 0.99% and 0.03% of Al, Mg, Fe, Mn, and Si respectively. Pure magnesium sample was consist of 99.9% Mg and the rest were different percentages of Al, Fe, Cr and Zn. These samples of aluminium and magnesium were prepared as coupons with thickness of 2mm and diameter of 8mm and their weight were measured and recorded. Then 6 samples were immersed in two liter of biodiesel in different beakers and these beakers were covered to prevent entering any contaminants and impurities. The samples left in the biodiesel from 720h and 1440h at between 25 to 27°C (room temperature). After that, the weight of coupons were measured after they were washed by deionized water then by acetone. These lost in the weight referred to corrosion rate which measured as milli-inch per year (mpy). Corrosion rate of biodiesel in aluminium was 0.1230 mpy for 720 h exposition period and it was 0.0527 mpy for 1440h duration. In magnesium, corrosion rate were 3.0910 mpy and 2.6563 mpy at 720h and 1440h respectively. In general, corrosion rate was decrease with increasing immersion time as in Figure 26. Also, it

was clearly observed that corrosion rate of magnesium was more than that in aluminium. That was due to the reactivity of magnesium is high with biodiesel and Mg is less noble in galvanic series if we compare it with aluminium [19].

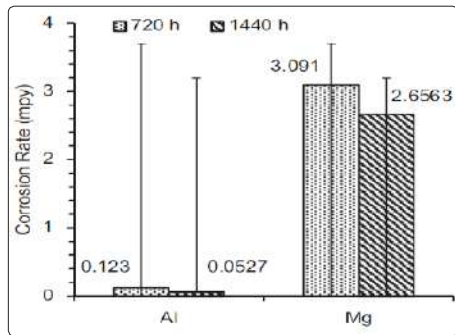


Figure 26: Corrosion rate of Al and Mg immersed in palm biodiesels for different periods [19].

Figure 27 shows the SEM micrographs of magnesium and aluminium before and after they were exposed to biodiesel. For aluminium there was no changes in its surface even after 1440h exposition. While, magnesium coupon had significant changes and its surface and uniform corrosion appeared as sticky polymer. On the other hand, XRD showed that the protective layer formed on the surface of the metal cannot be detected by XRD because it was too thin layer. In addition to that, total acid number of biodiesel was 0.27 mg. Then it was increased when it was measured for aluminium which was 0.29 mg KOH/g and for magnesium it was 0.87 mg KOH/g as in Figure 28. The values of TAN were slightly similar for both aluminium and magnesium while, corrosion rate of Mg was higher which means there was no strong relation between corrosion rate and TAN values. Biodiesel became more acidic when it exposed to moisture or oxygen. Magnesium was more active metal if compare to aluminium and there electrode potential were -2.37 V for Mg and -1.67V vs SHE for Al. The passivation region of Al was in pH range of 5 to 7 but, for Mg it was in the pH range of 12 to 14. So, the formation of gel layer on the top of the magnesium coupon it will help to decrease corrosion rate when immersion time take longer [19].

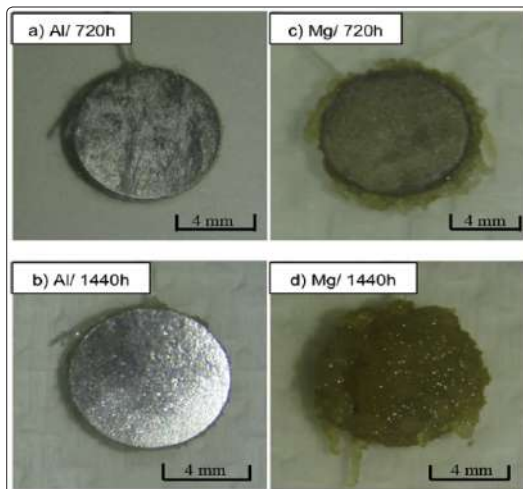


Figure 27: The SEM micrographs of magnesium and aluminium exposed to biodiesel in different periods [19].

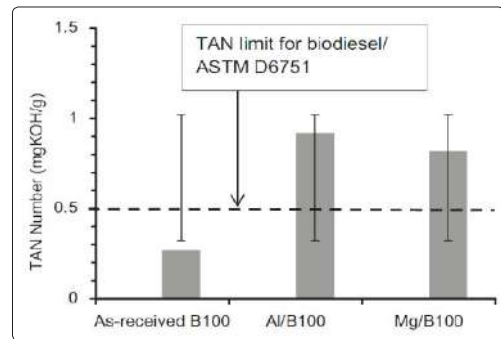


Figure 28: Total acid number of biodiesel exposed to Mg and Al for 1440h [19].

Palm Biodiesel Effect on Astm 1045 Mild Steel

The effect of commercial diesel and palm biodiesel on the ASTM 1045 mild steel was studied at different exposed period 30, 60 and 120 d and that was done at 27, 50 and 80 °C. SEM, XPS, XRD and FT-IR all were used to test composition and morphology of corrosion products. In addition, total acid number, temperature and exposed time were affection fuel degradation. From previous studies they agree on that increase water content, temperature, exposed time and oxide content will increase corrosion rate. It was very important to study corrosion products and corrosion behaviour caused by diesel or biodiesel on ASTM 1045 mild steel because many automotive parts are made from ASTM 1045 mild steel. So, by knowing corrosion products it will be easier to deal with biodiesel or diesel during storage and transportations [20]. ASTM 1045 mild steel or carbon steel (contains C, Mn, Si and Fe at 0.2%, 0.5%, 0.5% and 98.8% respectively) immersed in diesel 0# and in palm biodiesel at different temperatures and different time periods. Figure 29 shows the corrosion rate which was increased with increasing both time and temperatures [20].

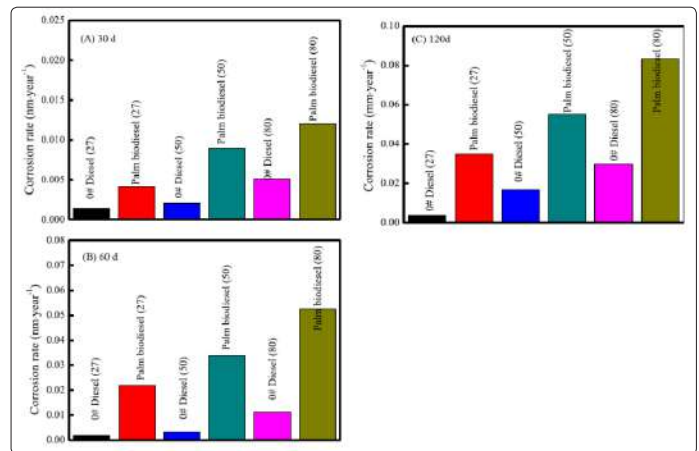


Figure 29: Corrosion rate of ASTM 1045 mild steel in palm biodiesel and diesel at 27 °C, 50 °C and 80 °C for 30, 60, and 120d [20].

From the test it appeared clearly that corrosion products on the ASTM 1045 mild steel were higher in the palm biodiesel compared to diesel as shown in figure 30, 31 and 32 [20].

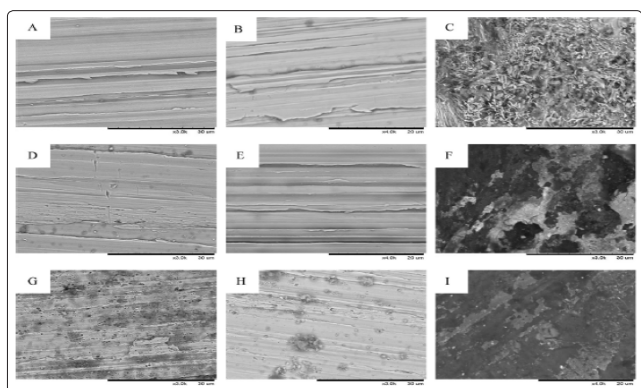


Figure 30: Surface morphology of ASTM 1045 mild steel in #0 diesel at different temperature 27, 50 and 80 °C for different exposure periods (A) 27 °C-30 d (B) 50 °C-30 d (C) 80 °C-30 d (D) 27 °C-60 d (E) 50 °C-60 d (F) 80 °C-60 d (G) 27 °C -120 d (H) 50 °C 120 d (I) 80 °C-120 d [20].

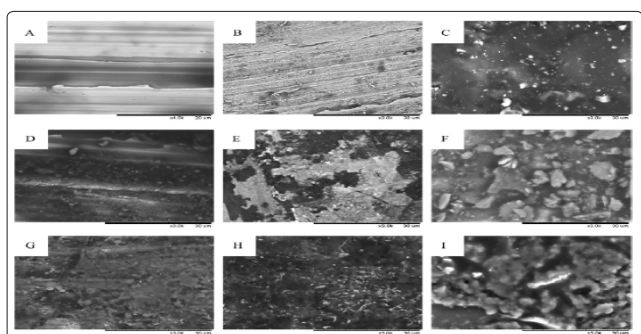


Figure 31: Surface morphology of ASTM 1045 mild steel in palm biodiesel at different temperature 27, 50 and 80 °C for different exposure periods (A) 27 °C-30 d (B) 50 °C-30 d (C) 80 °C-30 d (D) 27 °C-60 d (E) 50 °C-60 d (F) 80 °C-60 d (G) 27 °C -120 d (H) 50 °C-120 d (I) 80 °C-120 d [20].

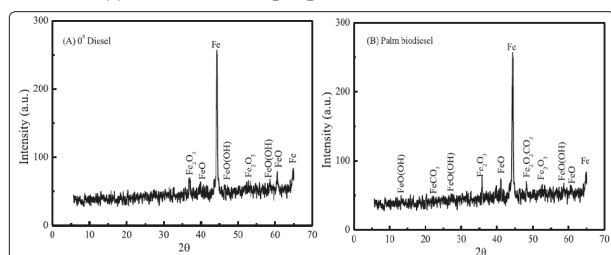


Figure 32: XRD spectrum of products produced as result of contact between palm biodiesel and biodiesel #0 with ASTM 1045 mild steel for 120d at 80°C [20].

Characteristic of corrosion on diesel engine parts affected by biodiesel from seed oils from Indian origin

There were some benefits from using biodiesel such as increase the employment and economy, less global warming issue and decrease dependence on the conventional oil. Some countries who use biodiesel were USA, Canada, Malaysia, Europe, Austria, Germany and India. Biodiesel was extracted from non-edible oils such as “*Jatropha curcas*, *Pongamia glabra* (Karanja), *Madhuca indica* (Mahua) and *Salvadora oleoides* (Pilu)” [21]. By cultivating 5 million hectares of *Jatropha* and *Pongamia* (Karanja) around 5 to 7.5 million ton of biodiesel was produced. Biodiesel can cause plug in the fuel filter and injector because biodiesel has can dissolve tank

deposits and elastomers. By testing different materials the result were as following: it is recommended to use fluorinated elastomers for biodiesel service, some materials like copper, brass, bronze, lead, tin and zinc were forming sediments when they react with biodiesel, but when it was compatible to be reacted with aluminium and stainless steel [21]. Two metal parts (piston liner and piston metal) were subjected to static immersion test in biodiesel extracted from non-edible oil like *Madhuca Indica*, *Pongamia glabra*, *Jatropha curcas* and *Salvadora* for 300 day at 15°C and 40°C. More than 50% of the fatty acid composition of biodiesel was unsaturated fatty acids. Penetration and corrosion rate of piston liner and piston metal was as in table 5 [21].

Table 5: corrosion rate and penetration of piston liner and piston metal tested for 7200h in different non-edible biodiesel [21].

S. no	Piston liner/oil	Acid value mg/g KOH	Piston metal/oil	Acid value mg/g KOH
1	<i>Jatropha curcas</i>	19.54	<i>Jatropha curcas</i>	14.48
2	<i>Pogamia glabra</i>	17.52	<i>Pogamia glabra</i>	14.39
3	<i>Maduca indica</i>	19.72	<i>Maduca indica</i>	11.30
4	<i>Salvadora</i>	2.51	<i>Salvadora</i>	2.38

There was reduction in the weight of piston liner metal when it tested with all different types of biodiesel. There was higher corrosion in *J. Curcas* more than *Mahua* and *karanja*. That was because *J. Curcas* has double bond which make it oxidized faster and more corrosive. While for piston metal the weight loss was approximately similar or in the same range. Although, there was exaptation for *Salvadora* oil which loss weight 10 times more than *J. Curcas*. Biodiesel extracted from *Salvadora* was the most corrosive one because it contained high Sulphur. *J. Curcas* biodiesel was second corrosive fuel in the test due to C18:2 fatty acid. table 6 showed total acid number of the different non-edible used in immersion test [21].

Table 6: TAN of the different non-edible used in immersion test for piston liner and piston metal [21].

S.no	Diesel/ biodiesel	Piston metal			Piston metal		
		Wt. loss mg	Corrosion rate		Wt. loss mg	Corrosion rate	
			mdd	mpy		mdd	mpy
1	Undoped diesel (petro)	0.1	0.0091	0.0058	0.3	0.0273	0.0065
2	Biodiesel JC oil	0.2	0.0182	0.0117	3.6	0.3279	0.0784
3	Biodiesel Karanja	0.1	0.0091	0.0058	0.3	0.0273	0.0065
4	Biodiesel Mahua	0.1	0.0091	0.0058	0.3	0.0273	0.0065
5	Biodiesel <i>Salvadora</i>	2.1	0.1913	0.1236	6.1	0.5556	0.1329

From Table 6, all biodiesel fuel indicates an increase in total acid number (TAN) which has higher values for piston liner metal compared to piston metal. Also, TAN for *Jatropha*, *Pogamia* and *Maduca* biodiesel because of un saturation in the oils which increase oxidation and corrosion rate. The corrosion occurs during using *Salvadora* was due to high content of sulphur [21].

Corrosion of 1018 steel caused by moniliella wahieum y12 and it is effect on biodiesel degradation:

Moniliella wahieum Y12 is one type of basidiomycetous fungus which isolated from 20% blended biodiesel (B20). Biodegradation of biodiesel was studied in the presence of Moniliella wahieum Y12 by measuring mass loss of the 1018 steel. The rate of degradation of the metal was at 3.56×10^{-2} mg/h. The cause of corrosion was the acidity of the medium which lead to increase mass reduction of the metal which exceeded that in control at 30 days by 70%. Microbial impurities enhance corrosion rate due to reducing the stability of fuel and microbial contamination could form biofouling. Microbiologically influenced corrosion (MIC) can cause corrosion due to forming biofilm and anoxic that increasing oxidation rate [22].

Strain Y12 was a type of M. wahieum (ATCC MYA-4962). Cultivation of Y12T lead to degradation of biodiesel. That was observed from losing of chromatographic peaks from biodiesel (B100). GC/FID was used to determine the loss of three dominant chromatographic peaks. Degradation rate of biodiesel was completed after 70 h when all of biodiesel completely used and the rate was at 3.56×10^{-2} mg/h. But fatty acid methyl esters degradation rate was not found. The tested 1018 steel coupons has content of carbon and other compositions that similar to materials used in manufacturing many storage vessels for biodiesel. Oxidation rate was increasing for carbon steel when biodegradation of biodiesel increase due to M. wahieum Y12^T found in the fuel. After 30 days, mass reduction of the metal coupon was around 0.49 mg. While after 60 testing days, mass reduction was increased to 0.85 mg. But, when Y12^T presence in the medium, mass loss increased by 70% compared to controlled sample tested for 30 days and it increased by 50% compared by tested sample for 60 days [22].

Microorganisms growing were depending on chemical component of the medium. This microorganism used biodiesel as carbon source to grow. Biodiesel degradation rate and degree was depending in the saturation hydrocarbon component and chain length of fatty acid. Moniliella wahieum Y12^T was depend on both saturation and unsaturation hydrocarbon and it utilized both in same rates. Biodegradation of different biodiesel was depending on bioavailability and viscosity. Biodegradation was slower for higher viscosity biodiesel. Because of emulsion formation, the rate of degradation was depending on the biodiesel dispersion. Corrosion was tested for 1018 steel in biodiesel degradation due presence of Moniliella wahieum Y12. As result for 30 day testing, 1018 steel corrosion was formed due to acidification, oxidation, and biofilm formation during the test. Also, pH was decreased from 7.0 to 5.3. In addition, there was film formed at fuel/water interface in tested coupon as in Figure 33 [22].



Figure 33: 1018 metal coupon corrosion induced by Moniliella wahieum Y12 in biodiesel as the carbon source. Chemical oxidation of the control coupon after 60 days incubation [22].

The effect of sunflower biodiesel in degradation of automotive material

Aluminium, copper and ferrous alloy were tested by immersed test in conventional petroleum diesel (B0), sunflower biodiesel (B100) and biodiesel blend (B20). The test was done for 3000h at room temperature (22-28°C) to 60°C. Then both corrosion products and corrosion rate were studied. Scanning electron microscopy (SEM) was used to study mass loss, change in the surface morphology and chemical microanalysis of the tested materials. X-ray diffraction (XRD) was used to detect corrosion products. Metal corrosion depends on temperature. Copper can be corroded faster than aluminium and carbon steel. This study gave idea about the suitable conditions for biodiesel storage [23].

During the test Food-grade sunflower oil was used (methanol anhydrous 99.8%). Sunflower methyl ester (SFME) was produced in the lab. Static immersion was done for aluminium (99% pure), copper (99.9% pure) and mild carbon steel contain C, Mn, P and S in following percentages respectively 0.32%, 0.5%, 1%, 1% and Fe. Biodiesel was tested to know some of its properties such as acid number, iodine value, water content and carbon residue. Acids and fatty acids that cause corrosion and degradation of biodiesel was determined by acid number. 117 g I₂/100g was iodine value for sunflower biodiesel which means the presence of high content of unsaturated fatty acids with double bonds. That led to more oxidation of biodiesel. More oxidation was accelerated corrosion rate because of transformation of esters into mono-carboxylic acids such as acetic acid and formic acid. Water content in biodiesel decrease heat of combustion because water presence lead to reduce conversion of triglycerides to biodiesel. Corrosion rate of different materials in different fuel at room temperature shown in Figures 34 and 35 show the results at 60°C [23].

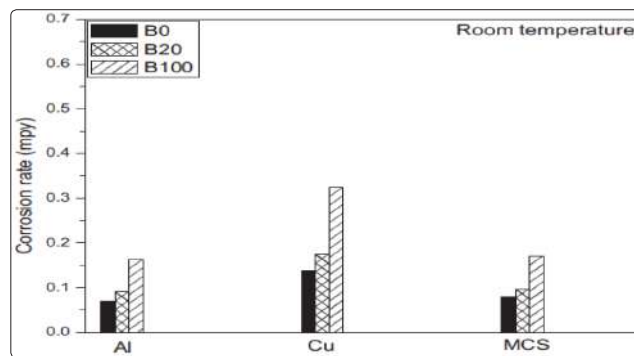


Figure 34: Corrosion rate of aluminium, copper and mild carbon steel tested in B0, B20 and B100 at room temperature [23].

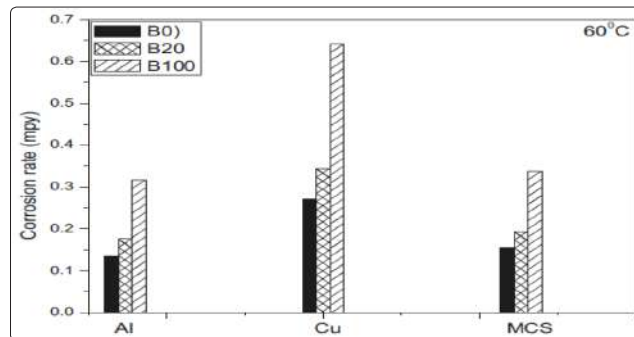


Figure 35: Corrosion rate of aluminium, copper and mild carbon steel tested in B0, B20 and B100 at 60°C [23].

It was clearly seen from the charts in Figures 34 and 35 corrosion rates were increased when bioethanol concentration increased in the fuel blend. Corrosion rate of biodiesel was increased in order aluminium, mild carbon steel and copper which have these values 0.162201 mpy, 0.170124 mpy and 0.323615 mpy respectively. Corrosion rate were doubled when temperature was raised. XRD pattern show the presence of base aluminium, base copper and base iron. Aluminium showed less corrosion rate because of the film of metal oxide that was formed in the surface of the metal. But mild carbon steel and copper show less corrosion resistance. Another result from immersion test was that by increasing the temperature the total acid number was increased then as result oxidation number was increased. Also, oxygen content was higher in biodiesel and biodiesel blend compared to diesel fuel. The results obtained from EDS were similar to those from XRD. SEM results showed no corrosion in aluminium surface when it immersed in B0 and B100 at room temperature. But there was protective layer formed on the metal surface when Al exposed to diesel and biodiesel fuel at 60°C. So aluminium was compatible material to be use as material come in contact with biodiesel. On the other hand copper sample was corroded or showed pitting in biodiesel and in the diesel at both room temperature and at 60°C. It is better to not expose biodiesel to atmosphere, because that may increase microbial growth due to water content in the atmosphere. Some suggested solutions to inhibit or prevent corrosion are using corrosion inhibitors, antioxidants and using material with low unsaturated content [23].

Corrosion inhibitors

Rosemary Extracts as Inhibitor for Corrosion in Aluminium Exposed To Biodiesel

Rosemary extracts tested to be corrosion inhibitor for aluminium immersed in biodiesel. Then the morphology of aluminium surface was tested by SEM. As result for that study when Rosemary extracts concentration increased the inhibition efficiency increased with decreased of temperature but corrosion rate decreased [24].

In the experimental part of that study, the aluminium sheet which was used contains 99.89% Al with Cu, Si, Mg, and Zn in different percentages. Rosemary leaves were grained after it were dried then it mixed with 300 ml of ethanol solution at 313 K and it was there for 48 h. After that, the sample was cooled and filtered. Then filtered sample evaporated at 352 k to get rid of ethanol. Different concentrations of Rosemary were prepared from dissolving from 0.1g to 0.5g of powder extract in 10ml ethanol (95%) then biodiesel was added to the solution and their total volume was 1.0 L. Aluminium samples were weighted before they were immersed in Rosemary inhibitor solutions with different concentration for 2000 h. Table 7 shows different values for corrosion rate and inhibition efficiency when aluminium sample tested in different concentrations of Rosemary extract [24].

Table 7: Corrosion rate (CR) and inhibition efficiency ($\eta_w\%$) values for the corrosion of aluminium in biodiesel in the absence and presence of different concentrations of Rosemary extracts at 298K [24].

Rosemary extracts conc. (g/L)	C_R (mg/cm ² h) x 10 ⁻⁵	$\eta_w\%$
Blank (biodiesel)	1.785	-
0.1	0.665	62.7
0.2	0.424	76.2
0.3	0.308	82.7

0.4	0.155	91.3
0.5	0.075	95.7

It was clearly seen from Table 7 that there was decrease in corrosion rate of aluminium with increasing the concentration. While there was increase in inhibition efficiency with increasing the concentration of Rosemary extracts. These results were because the adsorption of the organic contents of Rosemary extract on the surface of aluminium which were worked to protect aluminium surface from corrosion. Some organic component that presented in Rosemary extract were carnosol, carnosic acid, ursolic acid, homoplantagin, cirsimaritin and genkwanin as if Figure 36. Rosemary extract has properties of antioxidative due to its components which led to reduce the rate of biodiesel oxidation. That means Rosemary extract can work as corrosion inhibitor for corrosion of aluminium exposed to biodiesel [24].

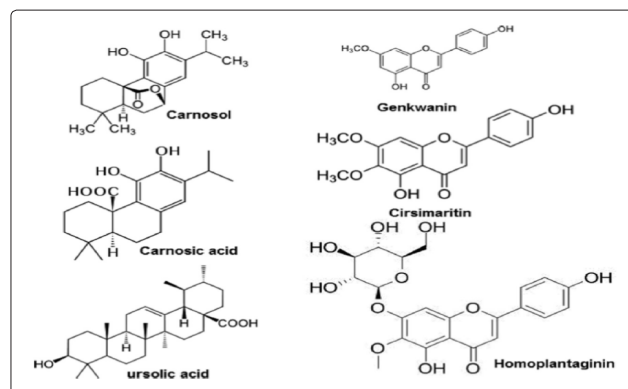


Figure 36: Common contents present in Rosemary extract and their chemical structure [24].

Corrosion inhibitors and its effect on corrosion behaviour of cast iron immersed in palm biodiesel:

There are different corrosion inhibitors can be used to control the corrosion caused by biodiesel. An example for these inhibitors was n-butylamine (nBA), ethylenediamine (EDA) and tetra-butylamine (TBA). These inhibitors were tested with cast iron immersed in palm biodiesel for 1200 hours at room temperature. Then the changes on the surface of the metal, weight reduction and total acid number were studied. The concentration of the acid was measured by FTIR. While surface morphology was studied by SEM. X-ray diffraction was used to study oxide layer. From the experimental part the results shows that tert-butylamine (TBA) was the most inhibitor that can resist corrosion in palm biodiesel [25].

Some studies showed that biodiesel such as the one that extracted from salvadora and jatropha curcas was more corrosive than diesel for piston linear metal. The composition of piston linear metal was cast iron. Pitting corrosion was formed in the specimens that immersed in fuel blended with biodiesel, but pitting was not found in the sample immersed or exposed to biodiesel. It was very important to find suitable corrosion inhibitors for biodiesel. Amine based compounds were good inhibitors for B0, B5 and B100. Because, they can form monolayer of protective film on the interface between metal and solution. Table 8 shows different corrosion inhibitors that were tested with palm biodiesel. Corrosion rate of palm biodiesel (B100) was tested in the presence and absence of corrosion inhibitors which gave different results as in bar graph that shown in Figure 37 [25].

Table 8: Different corrosion inhibitor for palm biodiesel [25].

Name	Abbreviation	Chemical formula	Structural formula
Ethylenediamine	EDA	$C_2H_4(NH_2)_2$	$H_2N \diagdown \diagup NH_2$
n-Butylamine	nBA	$CH_3(CH_2)_3NH_2$	$\diagdown \diagup NH_2$
Tert-butylamine	TBA	$(CH_3)_3CNH_2$	$+ NH_2$

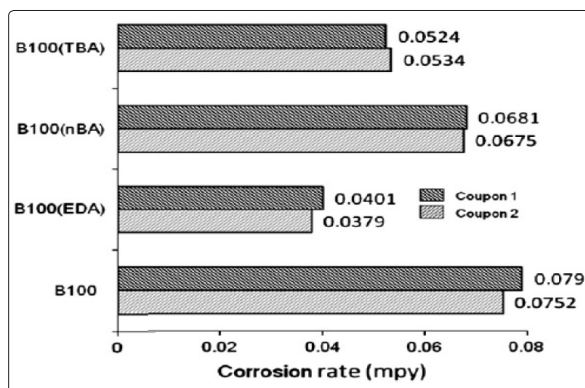


Figure 37: Different corrosion rate for palm biodiesel with three different inhibitors and without inhibitors [25].

Different inhibitors reduced corrosion rate in different rate as following order from less effective to most effective nBA < TBA < EDA. Another observation was that fat biodiesel was more corrosive compared to palm biodiesel. On the other hand, adding EDA to biodiesel led to degradation of the fuel [25].

Figure 38 shows different colors for oxidized layers due to different corrosion product on the surface. These products were mainly FeO and Fe₂O₃. These colors also represented different sediments on the surface which result from different inhibitors. These amine based inhibitors were reduced the formation of oxide layer in the surface due to forming hydrogen bond with metal surface [25].

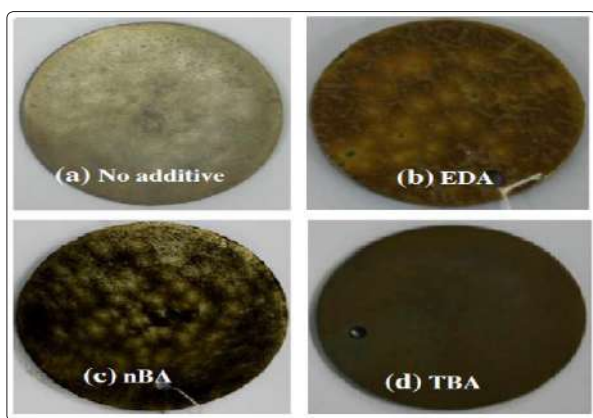


Figure 38: Different corrosion product formed on cast iron samples immersed on palm biodiesel [25].

Corrosion products that resulted from different corrosion inhibitors lead to change the composition of palm biodiesel and it gave different color as in Figure 39. The total acid number was changed after adding corrosion inhibitors as in the graph at Figure 40 [25].

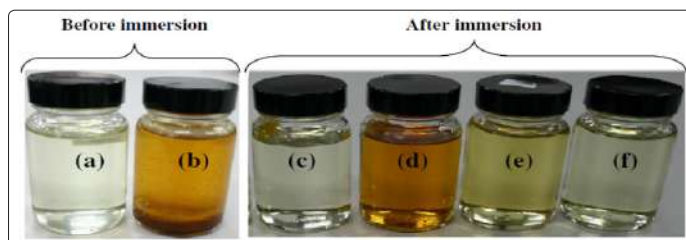


Figure 39: (a) as received biodiesel (b) biodiesel blend with EDA (c) biodiesel with cast iron (d) biodiesel with cast iron and EDA (e) biodiesel with cast iron and nBA (f) biodiesel with cast iron and TBA [25].

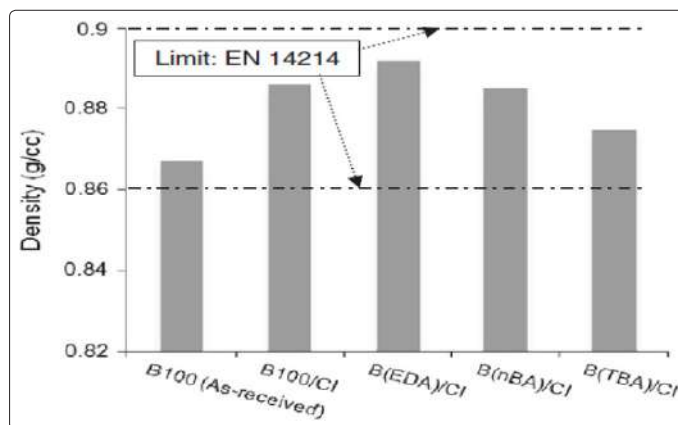


Figure 40: Different TAN for palm biodiesel (B100) and for B100 with inhibitors [25].

Controlling Biodiesel Corrosion of Carbon Steel by Natural Substances

When bioethanol produced it has inert behavior, but it become more corrosive during storage period. Corrosion problems usually occur because of blending diesel with biodiesel. Biodiesel was degraded when it exposed to metals and then it became more corrosive as results of forming some acids such as formic, acetic, propionic, and caproic acids. Another factors lead to increase the corrosivity of biodiesel were water content, temperature of water and contact with light. Metal ions were released as result of the presence of corrosive biodiesel. Then they were forming radicals which accelerate corrosion. So, it is very important to store biodiesel in suitable conditions. Corrosion inhibitors were one effective solution for this problem because they can reduce metallic corrosion. Inhibitors will not stop or prevent corrosion but it will reduce it is effects and it is rate. Amine based inhibitor such as primary amine, diamines, aminoamines and oxalated amines were the most effective corrosion inhibitors for diesel. Static immersion test was done for copper in biodiesel for 24 hours with adding antioxidants. From that, antioxidants was worked as corrosion inhibitors because it can form protective film layer [26].

Immersion test was done to study the effect of three natural substances as corrosion inhibitors. The results of the test showed that these three products can reduce corrosion of carbon steel. One of these three substances was steric acid which can form protective film layer. Another two inhibitors were propyl and beta-carotene which are antioxidant as in Figure 41 [26].

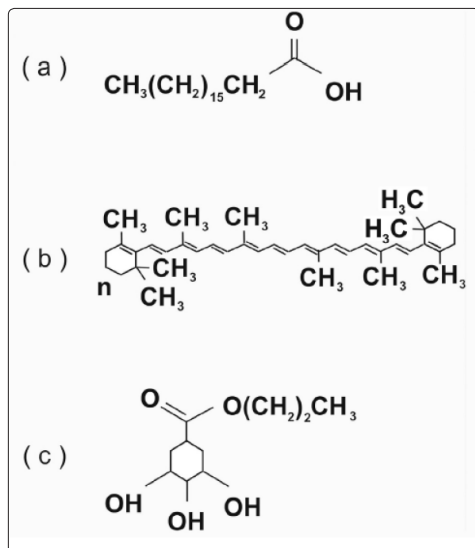


Figure 41: The chemical structure of (a) steric acid (b) Beta-carotene (c) propyl gallate [26].

The results of the immersion test indicate that pure bioethanol B100 was more corrosive compared to biodiesel with additives. While propyl gallate showed the best efficiency to inhibit corrosion compared to other tested additives as in Table 9 and Figure 42. The inhibition reaction was as following

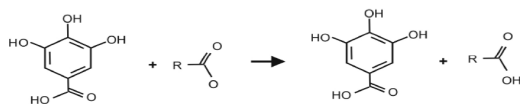


Table 9: Different corrosion rate, corrosion efficiency resulted from immersion test of carbon steel in biodiesel B100 [26].

System	Coupon	Corrosion Rate	Mean Corrosion Rate	Standard Deviation	Efficiency
		mm/year	mm/year		
Pure biodiesel (BD)	1	0.023	0.019	0.0021	-
	2	0.018			
	3	0.018			
Biodiesel with beta-carotene (BD+BC)	1	0.008	0.007	0.0010	62%
	2	0.008			
	3	0.006			
Biodiesel with propyl gallate (BD+GP)	1	0.003	0.003	0.0002	83%
	2	0.003			
	3	0.003			
Biodiesel with steric acid (BD+EA)	1	0.005	0.005	0.0005	75%
	2	0.004			
	3	0.004			

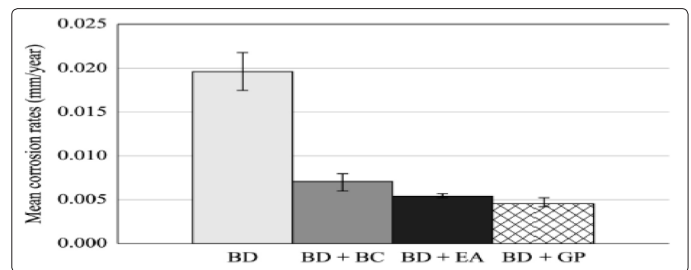


Figure 43: Corrosion rate of biodiesel B100 before and after adding three different additives [26].

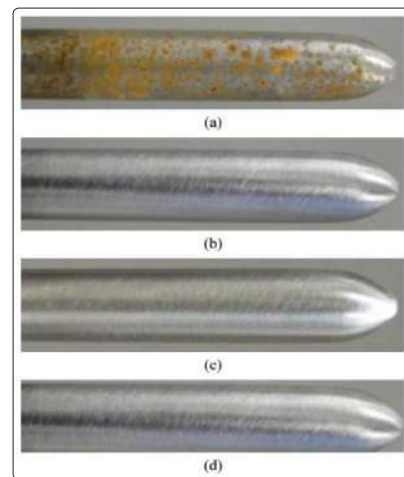


Figure 44: Results of tested carbon steel in biodiesel B100 (a) pure biodiesel (b) biodiesel with steric acid (c) biodiesel with propyl gallate (d) biodiesel with beta-carotene [26].

Properties of Biodiesel and Its Relation to Corrosion

Initiation, propagation and termination are the three steps of biodiesel oxidation. The result of oxidation was carbon base radical which gave peroxy radical and a hydroperoxides when it reacts with oxygen. The corrosivity of biodiesel was more than diesel because the presence of poly-unsaturated methyl ester which can oxidized then it formed acids. That will affect biodiesel properties which form corrosion and cause damage in the parts exposed to biodiesel. So, polyunsaturated content lead to increase the insoluble formation. Is But when antioxidant was added and its content increased, insoluble formation was decreased. Supercritical methanol method was used to reach oxidation stability for the biodiesel that has high content of peroxide. Biodiesel became less oxidative after it was exposed to supercritical methanol for 30 min at 270°C then peroxide in biodiesel was decreased. Peroxide value was less in biodiesel exposed to supercritical methanol in comparison with unexposed bioethanol. Hydroperoxides was affected by high temperature and it was decomposed [27].

Antioxidants were not used to stop corrosion but it can decrease the speed of corrosion because it is ability to reduce oxidation of biodiesel. Some examples for antioxidants are tert-butylated hydroxyanisole, tert-butylated hydroxy toluene and propyl galate. These antioxidants were very effective in increasing oxidation stability in biodiesel. The used amount of these products were from 100 to 1000 mg/kg. There are different natural and synthetic antioxidants that were studied and blended with biodiesel. Some examples are the following:

- tocopherol(α -T), butylate- dhydroxyanisole(BHA)
- butylatedhydroxytoluene(BHT); 2,5-di- tertbutylhydroquinone(DTBHQ)
- Ethanox4740;Ethanox4760E
- 2,2-methylene-bis-(4-methyl-6-tert-butylphenol)(MBMTBP)
- N, N-di-sec-butyl-p-phenylenediamine(PDA)
- propylgallate(PG); pyrogallol(PY);and tertbutylhydroquinone(TBHQ)

The most effective antioxidants were PY and JME. Some natural antioxidants were separated from vegetable oil during purification. So to get better oxidation stability for croton oil methyl ester (COME), synthetic antioxidant was used. Table 10 shows different antioxidants and their performance [27].

Table 10: Different antioxidants and their performance [27].

Biodiesel	Antioxidant	Concentration (ppm)	IP (h)	Reference
Karanja	PY	100	16	Das et al. [67]
Soybean	PY, TBHQ	100-300	>6	Ryu [75]
Soybean	TBHQ	1000	>12	Tang et al. [73]
Pongamia	PY	3000	34	Obadiah et al. [76]
FFA	PY	100	>6	Chen et al.[69]
JME	PY	100-250	>6	Chen et al.[69]
COME	PY	200	12	Kivevele et al.[71]
Moringa	PY, PG	200	>6	Kivevele et al.[74]
TBME	PY, PG	100	6.23	Chakraborty, [72]
JME	TBHQ, BHA	600	>6	Lamba et al. [77]
JME	PY	100	>6	Jain and Sharma, [64]

COME = Croton Megalocarpus oil Methyl Ester; TBME= Terminalia belerica Methyl Ester; JME=Jatropha Methyl Ester.

Another important factors that affect biodiesel degradation are water content and exposure to air. Different types of vegetables oil were tested when they placed in exposed and non-exposed containers for 30 months at room temperature. The results showed that all of acid value, viscosity, insoluble impurities and peroxide value were increasing with storage time. Another factor affecting biodiesel performance was exposure to the sunlight. Also, for long storage time for biodiesel it was suitable to do it at low temperature. In addition controlling the concentration of antioxidants was very important to reduce biodiesel degradation. Therefore, to get biodiesel with less degradation it is important to control and decrease exposure to light, oxygen and moisture that carried in low temperature [27].

Conclusion

Bioethanol can be extracted from biomass by fermentation process. Blending ethanol and gasoline has advantages of reducing environmental pollution and reducing dependence on crude oil. Although the exhaust gases produced during the using of bio ethanol were lower than exhaust gases came from regular fuel by following percentage HC83.5% and CO 89.6%, but one disadvantage of bio ethanol blended fuel was that it could form sedimentation on the engine's surface because of the combustions remains. Another disadvantage was to reach minimum fuel combustion which needs the new design for the carburetor to be able to use bio ethanol fuel mixture in combustion engine [28]. So, it was important to study corrosion rate of different metallic content of delivery system of different gasoline-ethanol blends. In general, bio ethanol is more corrosive than gasoline fuel.

Biodiesel can be produce by catalytic transesterification of fatty acid from vegetable oil. Biodiesel is better than petro-diesel because biodiesel has less emission. On the other hand, biodiesel has some disadvantages such as biodiesel is very sensitive to oxygen and it is autoxidation. Also, there are some secondary product from oxidation such as aldehydes and ketones which increase corrosion rate due to increasing total acidity number (TAN). Biodiesel was more corrosive than diesel because the biodiesel contain fatty acid and some contaminations or impurities [23]. Although biodiesel has more advantages comparing with diesel, biodiesel is more corrosive comparing with diesel. The corrosivity was increase when water content and free fatty acid were increased. Biodiesel absorb water more than diesel and that will cause water condensing on the metal surface and increasing corrosion. Also, biodiesel is auto-oxidation fuel which leads to more corrosive medium and degradation of fuel content. Non- ferrous alloys or metals were not compatible with diesel and they were corroded faster than ferrous alloys. An example for nonferrous metals are aluminium, zinc, copper, brass and bronze which work as catalysts that oxidized biodiesel even if the concentration of biodiesel is low. Corrosion inhibitors and antioxidant can be used to inhibit corrosion.

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