

Corrosion of Metals in Biofuels

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Global annual production of bioethanol exceeds 36,500 million liters and that of biodiesel exceeds 3,524 million liters. During production, transportation and usage, biofuels come in contact with various materials (metals and non-metals). Therefore it is important to understand the influence of biofuels on materials as well as the influence of materials on biofuels. This paper summarizes our current understanding of the corrosion of metals in biofuels. The sources of biofuels have been reviewed. Different types of corrosion experienced in biofuels have been discussed. Techniques to investigate the corrosivity of biofuels have been analyzed. Future developments in this field have been highlighted.

Sources of Biofuels

There are two main types of biofuels (1, 2):

- Bioethanol, which is blended with petroleum.
- Biodiesel, which is blended with diesel.

Bioethanol is produced by hydrolysis of a feedstock (Figure 1) (1-3). The feedstock is typically a biological source that contains sugar or materials (such as starch or cellulose) that can be converted into sugar. Most bioethanol is produced from sources containing sugar, e.g. sugarcane and sugar beets. Common starch-containing sources of bioethanol include corn, wheat, barley, rye and other cereals, while typical sources of cellulosic materials include agricultural wastes, forest residues, municipal solid wastes (MSW), wastes from pulp/paper processes and energy crops. Because the conversion of cellulose into sugar is relatively more difficult than the conversion of starch into sugar, the use of cellulosic feedstocks for bioethanol production is new and not yet applied on a large scale.

Biodiesel is produced by a transesterification process (3). Figure 2 presents typical sources of biodiesel. Oilseed crops are the primary feedstock for biodiesel production. The choice of feedstock depends on agricultural, geographical and climatic conditions. Soybean, rapeseed, cottonseed, sunflower seed, jatropha, pongamia and palm are common sources (4). In some circumstances oilseed crops are directly used as fuel sources, without transesterification; such fuels are commonly known as pure plant oil (PPO).

The use of algae as a source of biodiesel appears promising. The cultivation of algae requires less land area than the cultivation of other feedstocks. As an example, the production of one million gallon/year of biodiesel from algae requires about 100 acres of

land, while jatropha requires 4,500 acres and soybean requires 16,000 acres of land to produce the same amount of biodiesel. Another advantage of algae is that it can be cultivated in waste, brackish or saline water. Algae can be grown in a wide range of temperatures and varied environmental conditions. Efforts are being initiated to set up algal biorefineries to obtain multiple products from the same strain of algae (5).

Biofuels Scenario in Different Countries

The key driver for the increased demand for biofuels is the environmental concern associated with using fossil-based fuels. Several countries have set targets for the production and utilization of biofuels. For example, the European Union initially set a non-binding target that fuels should contain 2% of biofuels in 2005 and 5.75% of biofuels in 2010.

Bioethanol was first produced from maize several years ago. Ethanol is produced from maize in the United States, India, and China. Global bioethanol production is concentrated in two countries: Brazil and the United States. In Brazil, 50% of the 357.5 million tons of sugarcane produced in 2003-2004 was used for ethanol production (6).

Worldwide, oilseed crops (which are sources for biodiesel) are produced over a total area of 166.36 million hectares with a productivity of 1777 kg/ha (7). Biodiesel production is concentrated in the European Union with Germany and France leading the production.

Current global production of bioethanol and biodiesel are given in Tables 1 and 2 respectively (8-10).

Standards for Characterizing Biofuels

It is important that the biofuels produced from various sources in different countries meet some common standards so that their performance can be correlated with their chemical and physical properties. Several standards developed to characterize fossil fuels can be applied to biofuels (11). However these standards are developed from the perspective of characterizing biofuels with respect to their energy content and are not suitable for characterizing the biofuels with respect to corrosivity. Some standards from which indirect information on corrosivity can be obtained are presented in Table 3. Figure 3, described in ASTM G205, provides the most comprehensive approach to evaluate the corrosivity of crude oil under storage and transportation conditions. ASTM G205 is by far the most suitable standard to evaluate corrosivity of biofuels under storage and transportation conditions.

Key Corrosion Issues with Biofuels

The electrical conductivity of biofuels is low, so it was initially assumed that they are non-corrosive; however, field experience has been different. The significant corrosion issues associated with biofuels are summarized in the following paragraphs.

Stress-corrosion cracking (SCC) has occurred in carbon steel in contact with fuel grade ethanol (FGE) in the United States (Figure 4). However, SCC has not been experienced

in other countries, including over thirty years of experience in Brazil. In the USA, SCC has affected facility tanks and piping downstream of the point of manufacture until the ethanol is blended with gasoline. R&D carried out in several organizations on the mechanisms of ethanol SCC has lead to the following understanding (12, 13):

- Dissolved oxygen is a major contributor to SCC in fuel grade ethanol. Removing oxygen decreases susceptibility to SCC in laboratory slow strain rate tests.
- SCC occurs in strained areas associated with non-post-weld heat-treated areas and/or tank floor areas that are not adequately supported.
- The presence of chloride ions significantly increases the susceptibility to SCC.
- SCC does not significantly depend on steel grade.
- Corrosion inhibitors do not have any effect on SCC of steel, but some studies indicate that film-forming amines may have a slight inhibitory effect.
- SCC tendency increases as ethanol content increases; below ethanol contents of 10% SCC does not occur.
- SCC does not occur in ethanol containing more than 4.5% water.
- pH and acetic acid do not have a significant influence on SCC.
- The source of the ethanol has some influence on SCC tendency.
- The influence of variations in ethanol chemistry on SCC is the subject of ongoing studies.

Corrosion experience with biodiesels is not as extensive as with bioethanol. From this limited experience, corrosion issues associated with biodiesel are summarized in the following paragraphs:

- Corrosion and microbiologically influenced corrosion (MIC) have been reported in tanks storing biodiesel. Corrosion mainly occurs due to the entry of moist air during storage and/or contamination of the fuel with water during transportation.
- Corrosivity of biodiesels depends on the origin of the fuel, but the ingredients causing the difference are not understood. For example, Table 4 compares the corrosivity of two biodiesels of Indian origin (14-16).
- The effects of other factors (including pH, temperature, and biodiesel composition) on the corrosivity of biodiesel are not well understood.
- Accumulation of water also accelerates microbial growth leading to the occurrence of MIC. It appears that because of their biological origin biodiesels have a higher potential for supporting microbial activities than fossil-based diesels.
- The behaviour of fossil fuels at higher temperatures (800-1500°C) is well understood, but fundamental studies on the behaviour of biodiesel/biofuel/fossil fuel combinations at these high temperatures are lacking.
- SCC like that observed in the presence of fuel-grade ethanol has not been reported in biodiesel from any source.

Techniques to Study Biofuel Corrosivity

The applicability of some techniques for investigating the corrosivity of biofuels is reviewed in the following paragraphs.

Mass Loss

One of the most common methods to investigate the corrosion rate is by mass loss. ASTM G31 provides guidelines to conduct mass loss experiments. ASTM G1 provides information on cleaning the samples before and after the experiments. A procedure to investigate the corrosivity of biofuels by mass loss method is as follows:

- Weighed specimens are immersed in biofuel (B100), biofuel containing small amounts of water (typically 1ml of 3% NaCl solution in 99 ml of biofuel (B99)) and 3% NaCl solution for a certain period (typically 100 hrs).
- Specimens are removed after the set intervals of time and dipped in sodium hydroxide solution to remove excess oil, washed with distilled water, dried, and reweighed.
- The loss in mass is determined and the corrosion rates are calculated using Eqn. 1:

$$\text{Corrosion rate (mpy)} = \frac{3.45 * 10^6 * \text{mass loss(grams)}}{\text{Density(g/cm}^3\text{)} * \text{Area(cm}^2\text{)} * \text{Time(hours)}} \quad (\text{Eqn.1})$$

As an example, the corrosion rate of aluminum in biodiesel produced from *Pongamia pinnata* (B100), 3%NaCl, and in B99 (99 ml of biodiesel and 1 ml of 3% NaCl) is presented in Fig. 5. The corrosion rate of Al in B100 is far lower than that in 3% NaCl solution. This result is not unexpected because the conductivity of B100 is 3 orders of magnitude less than that of 3% NaCl solution. However higher corrosion rate is observed in B99 than in NaCl solution (Fig. 5). This anomaly may be due to lower corrosion rates and to the cleaning procedure.

Electrochemical Techniques

The mechanism of corrosion taking place in an aqueous phase is electrochemical. Therefore a broad range of electrochemical techniques can be used to investigate corrosion rate, including the polarization resistance method, Tafel extrapolation method, cyclic potentiodynamic method, potentiostatic method, galvanostatic method and galvanic current method (17, 18). Standards to measure corrosion rate using these techniques are also available. The main advantages of electrochemical techniques include their sensitivity to low corrosion rates, short experimental duration, and well-established theoretical understanding.

Among these methods, the polarization resistance method is commonly used for corrosion measurement. ASTM G59 provides a procedure to use this technique. Briefly, measurements are performed using a potentiostat in the potential range of -0.02V to $+0.02\text{V}$ with respect to corrosion potential, and at a scan rate of 0.1667 mV/s . The experiments are performed in a glass beaker containing the aerated, unstirred test solution with a platinum electrode as the counter electrode, a saturated calomel electrode as the reference electrode and the metal specimen (with an exposed area of 1 cm^2) as the working electrode.

Figure 5 compares the corrosion rates of aluminum in biodiesel obtained from *Pongamia pinnata* as determined by the mass loss and polarization resistance methods. In general, the corrosion rates determined by the polarization resistance method are higher than those determined from the mass loss. Because of the low conductivity of biodiesels accurate results are frequently not obtained by the polarization resistance method. It should also be noted that the polarization resistance method measures instantaneous corrosion rate, whereas the mass loss method measures time-averaged corrosion rates.

Scanning Electron Microscopy (SEM)

One common method to investigate the nature of corrosion products is SEM analysis. For SEM analysis samples are typically mounted in low-shrinkage epoxy under vacuum prior to grinding and polishing progressively to 0.05 μ m using colloidal silica. The mounted specimen is then coated with a thin layer of palladium for electron conduction purposes prior to SEM examination. Figures 6 and 7 present SEM images of aluminum samples exposed to biodiesel from *Pongamia pinnata* for 100 hours. SEM images taken from the cross-section are shown in Figures 7 and 8.

From figures 6 through 8, the followings observations can be drawn:

1. The sample surface is extremely rough (Figure 6). The EDS spectrum taken from the surface shows Al and Fe, which are known species in aluminum alloys. The minor O peak is attributed to a thin aluminum surface oxide which forms on any aluminum surface exposed to air. The surface seems to be a machined surface without any polishing. The expected organic film is either too thin to be observed by techniques used in this paper or non-existent.
2. In between large surface asperities, some very thin and small patches exist as identified in the high-magnification optical and SEM images in Figures 7 and 8. However, with such surface condition, it is difficult to determine with confidence that they are the organic film. It is possible that they are contaminants/residues from prior to the experiment. The actual coating may be too thin to be resolved in SEM.
3. EDS work on those small patches (Figure 8) was not possible due to limited spatial resolution.
4. To study such a thin coating, transmission electron microscopy (TEM) work is needed. However, such analysis is not trivial due to the following reasons:
 - a. the film is so thin that an advanced TEM sample preparation technique is needed,
 - b. the film could be electron beam-sensitive (depending on the chemical composition), requiring a weaker TEM beam.

Non-Contact Profilometry

Non-contact profilometry techniques can be used to measure the surface topography of corroded materials without altering the sample surface through physical contact by a stylus (19-21). Two common types of commercial non-contact profilometers are those that employ the principle of optical triangulation (laser profilometers) and those that employ axial chromatism (confocal profilometers).

In laser profilometers, a collimated beam of light from a laser is projected onto the target surface through a focusing lens. The reflected light is then focused onto a photodetector by a second lens. The position at which the reflected beam strikes the detector varies with the height of the target surface. The signal generated by the detector varies in proportion to the position at which it is struck, and thus a profile of the surface is generated from the changing signal as the laser scans across the surface. The axial resolution of laser profilometers is on the order of micrometers.

Confocal profilometers project a beam of white light through a lens with a high degree of chromatic aberration, resulting in each constituent wavelength focusing at a unique distance from the lens. The height of a specific point on the target surface is given by the wavelength of the monochromatic light whose focal length coincides with the surface. The reflected light passes first through a spatial filter to suppress all but the focused wavelength, and then through a diffraction grating for spectral analysis. Similar to the laser profilometer, the position at which the reflected beam strikes the detector varies with its wavelength (and therefore the height of the target surface), and the signal generated by the detector varies with the position at which it is struck. The axial resolution of confocal profilometers is on the order of nanometers.

A primary advantage of scanning profilometers such as those based on laser and confocal technologies is their automatic nature. Once the user has defined the scan parameters (area, step size and scan speed are the most important) and started the scan, the instrument proceeds with the scan without requiring any further input. As a result, the number of data points can be increased, increasing the lateral resolution of the resulting profile and reducing the possibility that important features will be missed. The lateral resolution of the data is ultimately limited by electronic data file size, instrument demand (determines the amount of time permitted for a single scan) and the lateral resolution (spot size) of the beam itself, rather than by the number of measurements it would be practical to make manually. In addition to automated scanning, software packages that perform a wide range of data operations and analyses are available. For example, pit depth distributions and standard surface parameters can be calculated automatically, and the surface topography can be imaged in 2 or 3 dimensions. As an example, surface topography and pit depth distribution obtained by laser profilometry for an aluminum sample after 100 hrs immersion in biodiesel produced from *Pongamia pinnata* is presented in Figure 9.

The automated scanning and data manipulation capabilities of profilometry instruments and their associated software make them powerful tools for the study of corrosion, but there are certain limitations inherent in these systems of which users must be aware. The need to balance lateral resolution (step size) with scan time and data file size, especially for large areas, was mentioned previously. Using the software packages to manipulate the data may lead to artifacts, particularly for complex operations such as those intended to remove or separate features such as large-scale curvature or periodicity. In some software packages, the algorithm employed to identify surface features is designed to exclude anomalies, so it is possible that very narrow pits might be excluded from the peak/pit distribution calculations. It is also important for users interested in pit depth values to

realize that peak heights and pit depths are measured relative to some point on the corroded surface, and it is up to the user to relate that point back to the height of the uncorroded surface if desired. For example, the highest point on a measured surface may correspond to the original uncorroded surface, or it may correspond to a surface deposit or a point below the original surface that has been exposed by metal loss due to general corrosion. Laser profilometers usually have narrow focal depths that can limit the area that can be measured in a single scan when the height of the surface varies greatly (e.g. small radii of curvature), while confocal profilometers tend to be capable of measuring larger height ranges.

Current and Future Trends in Biofuels

The Steel Tank Institute conducted a study of the corrosion of steels in two different biodiesels mixed in varying proportions with ultra-low sulfur diesel according to the ASTM specifications D6751 (for 100% biodiesel) and D7467 (for biodiesel and diesel mixture). Small amounts of water were also added to some samples. The corrosion rates in both biodiesels and their blends were quite low based on a two-month mass-loss measurement (22). The Strategic Alliance for Steel Fuel Tanks (SASFT) recently completed a study called "Durability performance of fuel-tank steels in biodiesel fuels". This study found that biodiesel degrades to produce aggressive corrosive environments, and that the corrosion rate depended on both the material and type of biodiesel (23).

The Pipeliner Biodiesel Steering Committee (PBSC) has identified several areas that need further research to assure the safe transportation of blends above B5. According to the committee, there are relatively few technical challenges associated with movement of up to B5 blends in pipelines carrying only gasoline, diesel, or heating oil. There are, however, a number of technical items that need to be scrutinized before approval of biodiesel blends above B5 in pipelines (24).

R&D carried out on the corrosivity of biofuels to date indicates that:

- the corrosivity of biofuels depends on impurity content;
- sophisticated methodologies are needed to properly evaluate the corrosivity of biofuels;
- biofuels conforming to established performance specifications may not have similar corrosion characteristics; and
- it is important to understand the behaviour of materials in biofuels, so that the design and construction of storage, transportation, and operation systems handling biofuels can be carried out in a cost-effective manner.

Currently R&D is being carried out to understand the effects of minor constituents on corrosivity of biofuels, to develop safe limits for any deleterious constituents in biofuels and to develop a method to rapidly monitor biofuel corrosivity in terms of any deleterious effect.

In the future the demand for both bioethanol and biodiesel will continue to increase. In addition to established sources biofuels will be produced from newer sources, therefore it

is important to understand the influence of biofuels on materials as well as the influence of materials on biofuels.

Summary

- As the demand for more biofuels increases, newer infrastructures will be built using various materials.
- It is important to understand the corrosion and stress-corrosion cracking of metals in the presence of biofuels, so that appropriate materials are selected to construct the infrastructure.
- It is important to understand the influence of different types of biofuels (especially the influence of impurities) on corrosion.

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Figure 1: Feedstock for ethanol production (4)

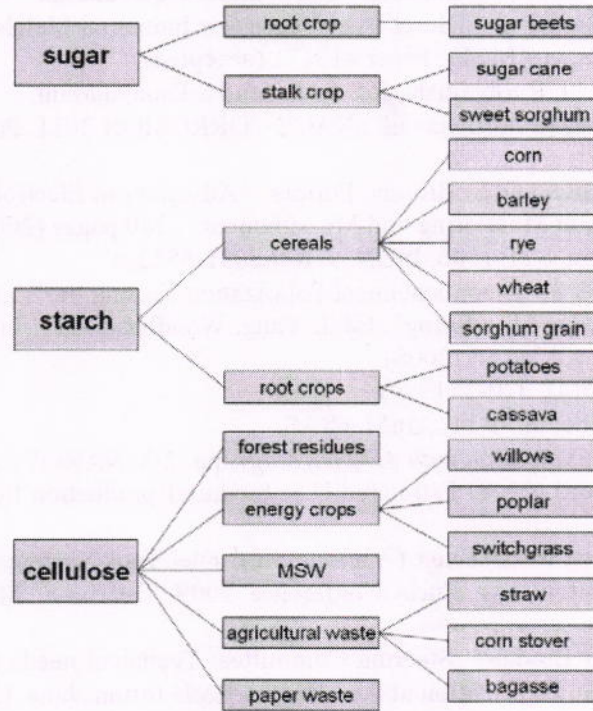


Figure 2: Feedstock for biodiesel production (1-5)

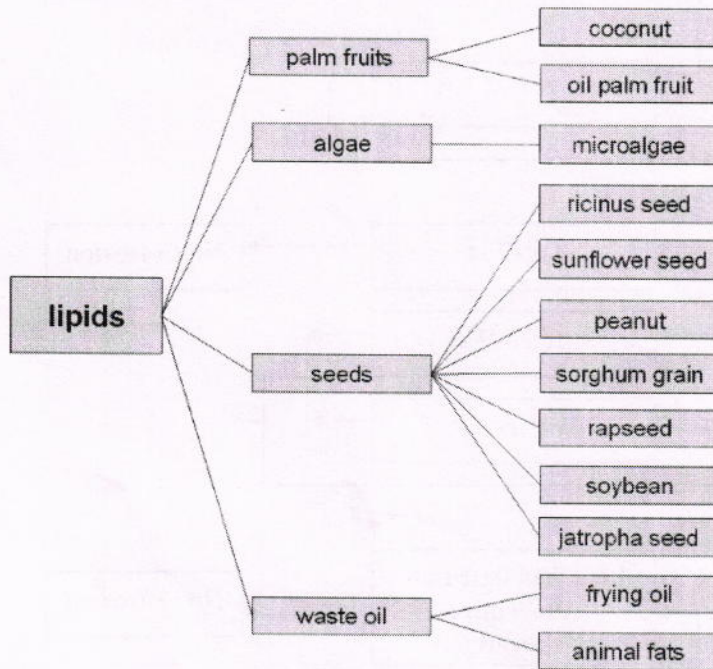


Figure 3: Predicting Corrosivity of Crude Oil (ASTM G205)

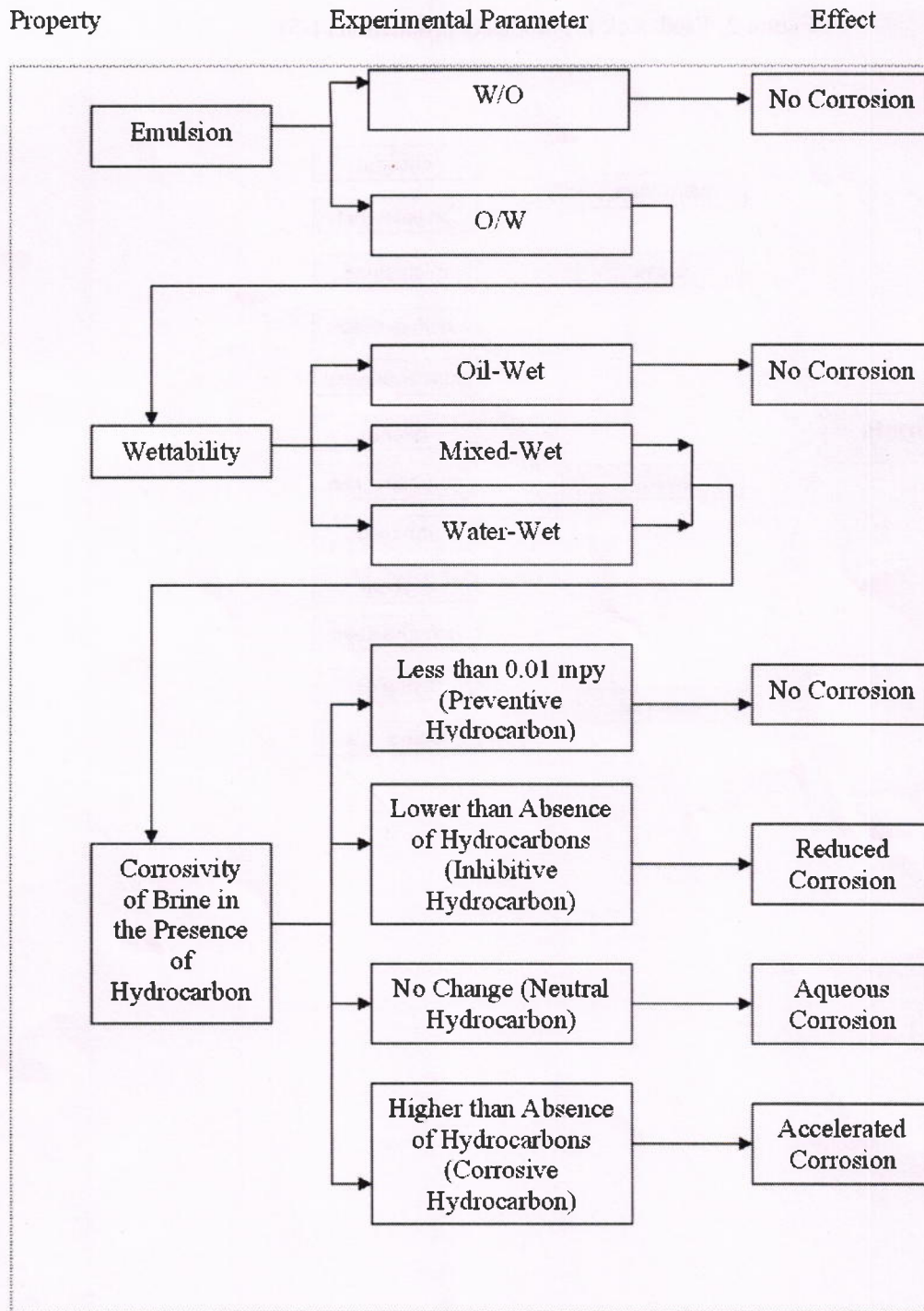


Figure 4: SSC Observed in Terminal Piping System Containing Fuel Grade Ethanol (12)

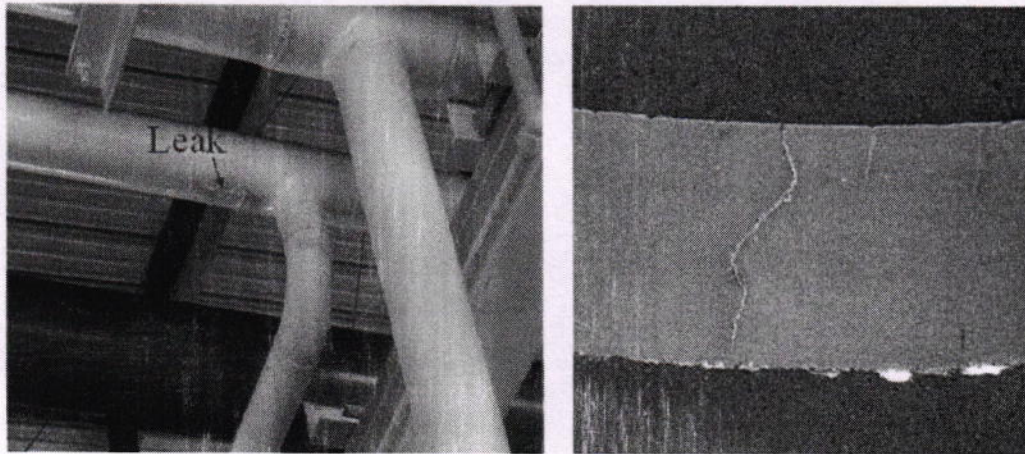


Figure 5: Comparison of Corrosion Rate of Al in Biodiesel by Mass Loss and LPR techniques (16)

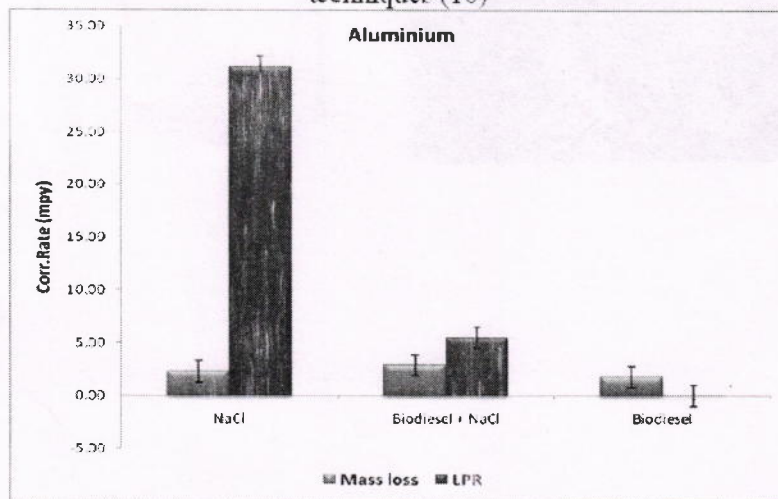


Figure 6: Planview SEM images and EDS spectrum of Al Samples Exposed to Biodiesel

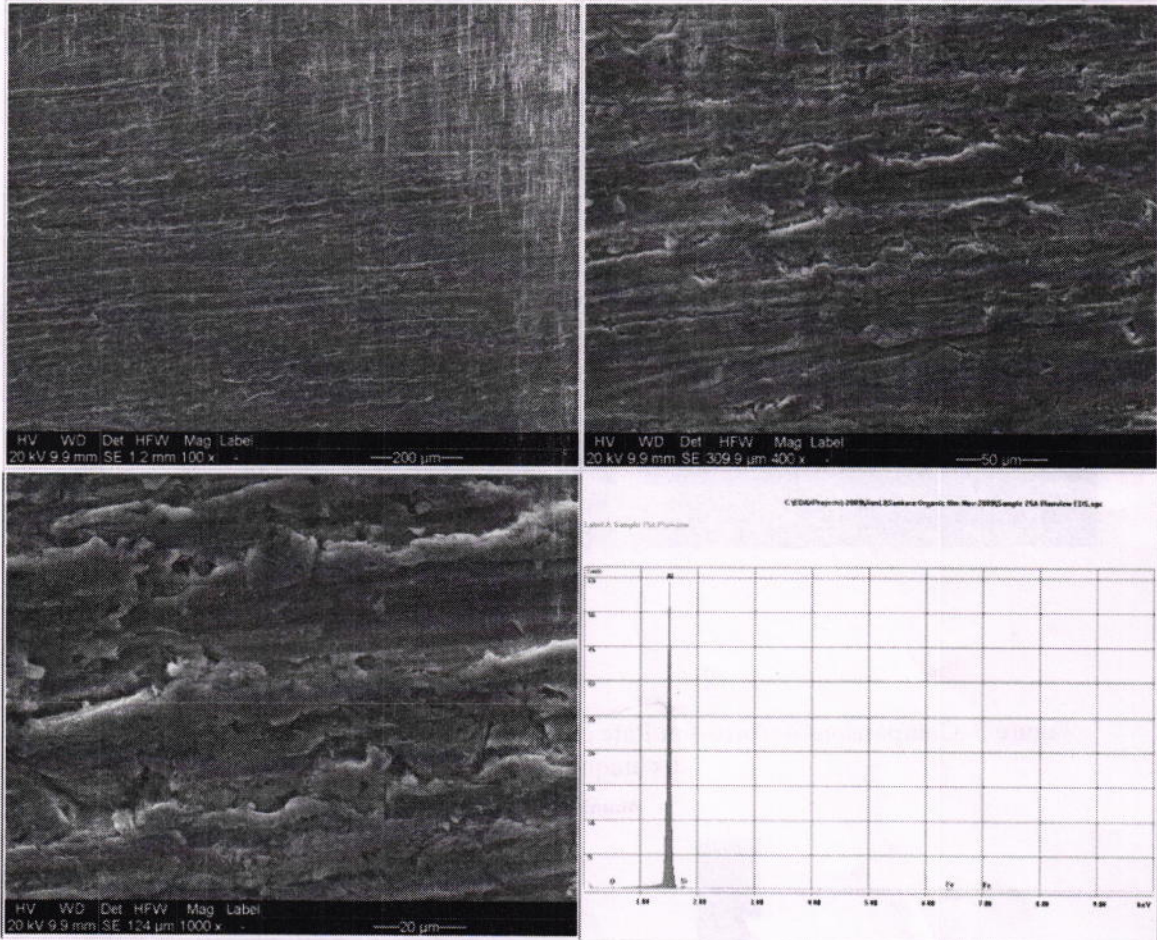


Figure 7: Optical images of the polished cross section (Same sample as in Figure 6)

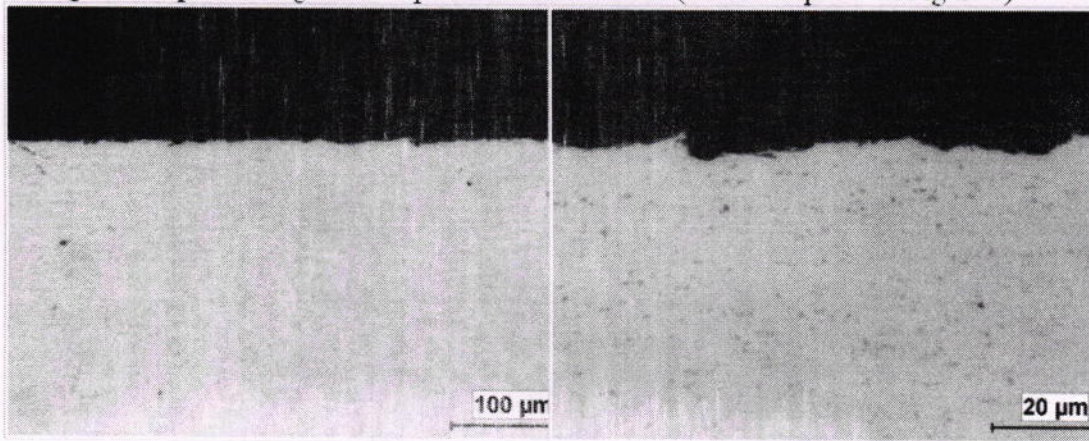


Figure 8: SEM images taken from the polished cross section (Same sample as in Figure 6)

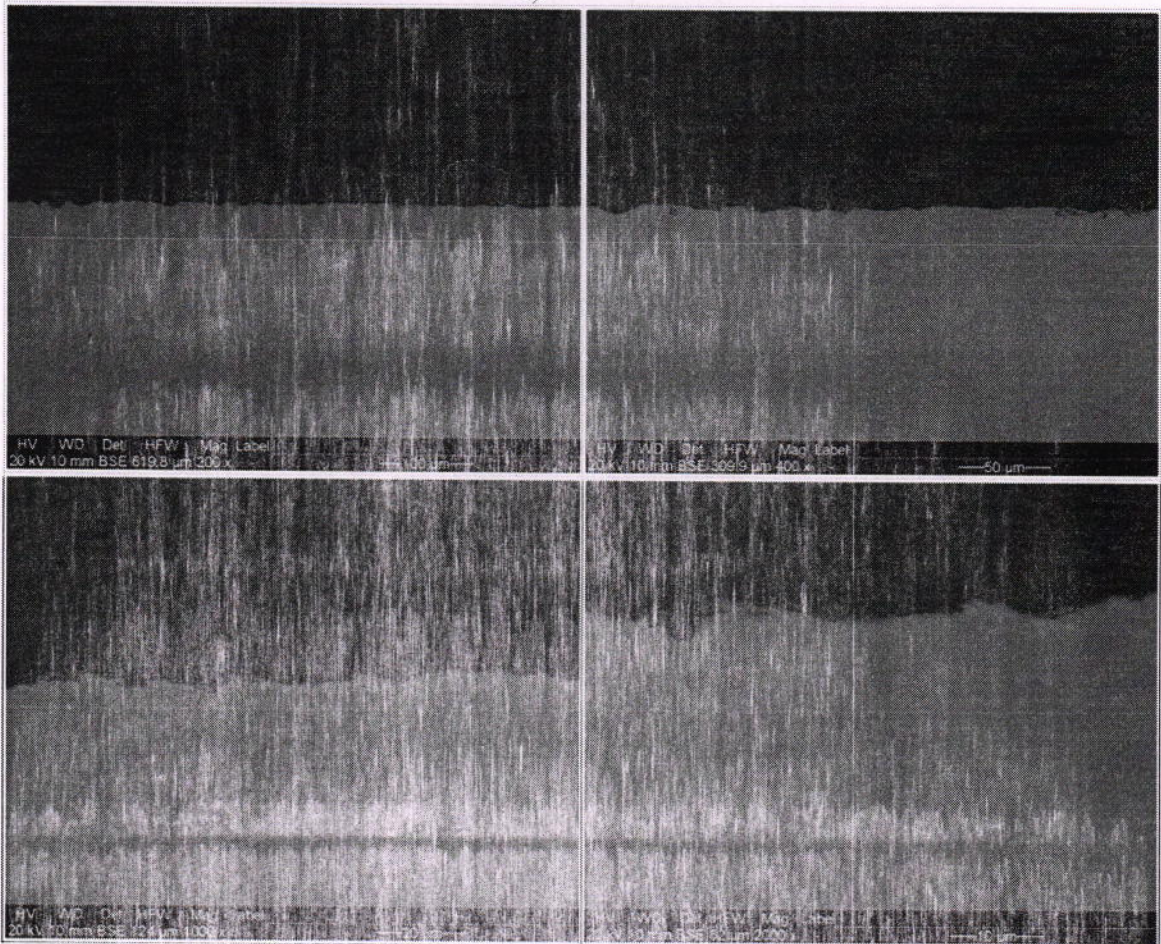


Figure 9: Surface topography and pit depth distribution obtained by laser profilometry for an aluminum sample after 100 hrs immersion in biodiesel produced from *Pongamia pinnata*. All depths reported relative to the highest point on the surface.

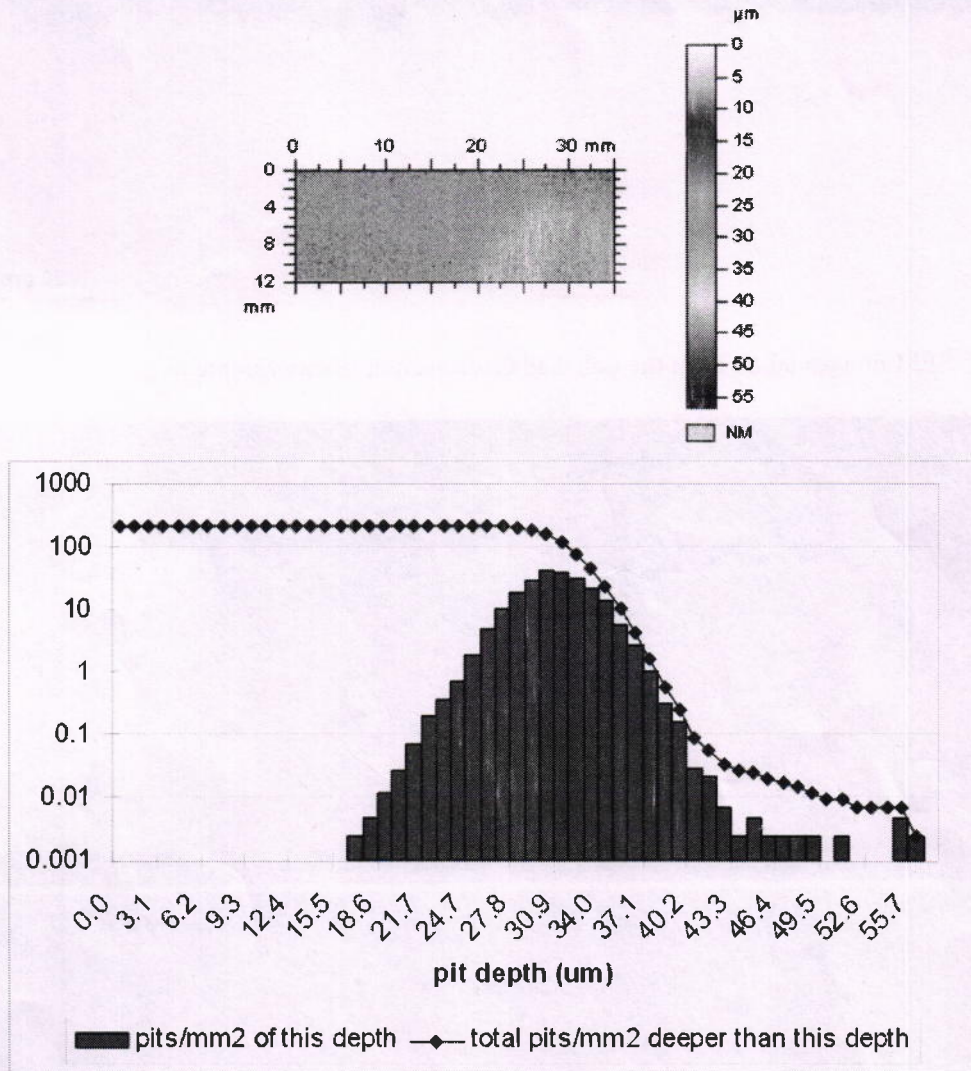


Table 1: Global production of bioethanol (9, 10)

Country/region	Ethanol production (million liters)	Share of total ethanol production (percent)
Brazil	16,500	45.2
United States	16,230	44.5
China	2,000	5.5
European union	950	2.6
India	300	0.8
Canada	250	0.7
Colombia	150	0.4
Thailand	60	0.2
Australia	60	0.2
World total	36,500	100.0

Table 2: Global production of biodiesel (9, 10)

Country/region	Biodiesel production (million liters)	Share of total biodiesel production (percent)
Germany	1,921	54.5
France	511	14.5
Italy, Austria, Denmark, UK, Czech Republic, Poland, Spain Sweden	9-227	0.1-6.4
Europe total	3,121	88.6
United States	290	8.2
Others	114	3.2
World total	3,524	100.0

Table 3: ASTM Standards that can be used to understand corrosivity of biofuels

S.No	ASTM Standard	Title	Scope
1	G205	Standard Guide for Determining Corrosivity of Crude Oils under Pipeline Operating Conditions	This guide describes methods for determining the corrosivity of crude oils by a combination of three properties: <ol style="list-style-type: none"> 1. emulsion of the oil and water, 2. wettability of the steel surface, and 3. corrosivity of water phase in the presence of oil
2	D6751	Standard Specification for Biodiesel Fuel (B100) Blend Stock for Distillate Fuels	This specification covers biodiesel (B100) Grades S15 and S500 for use as a blend component with diesel fuel oils defined by Specification D 975 Grades 1-D, 2-D, and low sulfur 1-D and 2-D.
3	D6751	Standard Specification for	This specification covers biodiesel fuel

		Biodiesel Fuel Blend Stock (B100) for Middle Distillate Fuels	blend stock, B100, in Grades S15 and S500 for use as a blend component with middle distillate fuels and prescribes the required properties of diesel fuels at the time and place of delivery.
4	D7467	Standard Specification for Diesel Fuel Oil, Biodiesel Blend (B6 to B20)	This specification covers fuel blend grades of 6 to 20 volume percent (%) biodiesel with the remainder being a light middle or middle distillate diesel fuel, collectively designated as B6 to B20.
5	D7095	Standard Test Method for Rapid Determination of Corrosiveness to Copper from Petroleum Products Using a Disposable Copper Foil Strip	This test method covers the determination of the corrosiveness to copper of aviation gasoline, aviation turbine fuel, automotive gasoline, natural gasoline, or other hydrocarbons having a vapor pressure no greater than 124 kPa (18 psi), cleaners (for example, Stoddard solvent), kerosene, diesel fuel, distillate fuel oil, lubricating oil and other petroleum products.
6	D7220	Standard Test Method for Sulfur in Automotive Fuels by Polarization X-ray Fluorescence Spectrometry	Provides information on the measurement of total sulfur in automotive fuels.
7	D7321	Standard Test Method for Particulate Contamination of Biodiesel B100 Blend Stock Biodiesel Esters and Biodiesel Blends by Laboratory Filtration	This method provides a means of assessing the mass of particulates present in a fuel sample, which results in plugging of the fuel system.
8	D6423	Standard Test Method for Determination of pHe of Ethanol, Denatured Fuel Ethanol, and Fuel Ethanol	The acid strength of high ethanol content fuels can be determined.
9	D6748	Standard Test Method for Determination of Potential Instability of Middle Distillate Fuels Caused by the Presence of Phenalenes and Phenalenones (Rapid Method by Portable Spectrophotometer)	This test method covers a procedure for the rapid determination of phenalenes and phenalenones in middle distillate fuels, including marine, automotive, heating and gas turbine fuel such as those specified in Specifications D 396, D 975, D 2069, and D 2880. Phenalenes and phenalenones affect the potential instability of fuels, leading to fuel degradation products during storage, which may cause performance problems.

Table 4: Comparison of corrosion rates of the selected metals in Jatropha oil and *Pongamia pinnata* biodiesel (14-16)

Medium	Corrosion rate (mpy)							
	Carbon Steel		Aluminum		Copper		Brass	
	Jatrop ha	Ponga mia	Jatrop ha	Ponga mia	Jatrop ha	Ponga mia	Jatrop ha	Ponga mia
100% biodiesel	0.76	0.75	0.89	1.77	0.24	1.03	0.24	0.39
99%biodi esel +1%Aqu ous phase*	1.34	3.85	0.97	2.91	0.09	0.76	0.27	0.32
100% Aqueous phase*	4.29	3.16	0.44	2.29	1.80	1.81	0.73	0.89

*Aqueous phase is 3% NaCl

