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Investigation of Mild Carbon Steel Immersed in Jatropha Biodiesel Fuel: Spectroscopic and Surface Studies

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***Abstract**: The use of renewable energy fuels in the automobile industry has seen progressive interest and increased research in recent times. Different types of steel alloys have been considered as materials in the construction of the fuel delivery and storage systems of the automobile engines which are conduit for passage of these biofuels to the specific engine combustion chamber. The nature of the interactions between the surface of the mild steel alloy and the specific biodiesel molecules needs to be interrogated to ascertain the extent and degree of biodiesel activity-induced corrosion. In this study, FTIR, UV-Vis spectrometry and optical microscopy techniques were used to elucidate on the biodiesel molecule-mild steel surface interphase interactions. UV-Vis investigation revealed pronounced peaks around 900 nm and 1050 nm consistent with signals due to poly-unsaturated components of the biodiesel. FTIR analysis showed peaks around 1700 cm^{-1} which indicated the presence of C=O and C-O functional groups that is associated with triglycerides. Peak signals around 2950 cm^{-1} are attributed to anti-symmetric and symmetric stretching vibration of C-H in CH_2 and CH_3 while peaks around 1150 cm^{-1} indicated the stretching vibration of C-O ester. The results revealed jatropha biodiesel-induce corrosion was physically adsorbed on the steel surface leading to surface degradation over time and were evident in the optical surface micrographs which were equally supported by the FTIR and UV-Vis analyses. Thus, carbon steel alloy material selection and testing to ascertain compatibility and effectiveness is vital when biodiesel is to be used as fuel in automobile engines.*

Keywords: Carbon alloy, biodiesel fuel, microscopic characteristics, ultraviolet/visible spectrometry, Fourier transform-infrared spectroscopy.

1. INTRODUCTION

The energy demand for the automobile industries is still largely provided from non-renewable energy sources such as coal and crude oil even with advancement in energy developments [1, 2]. In Nigeria, most of the fuel for the automobile industry is still from fossil-petroleum produced locally and imported from refineries outside the country [3]. Diesel is a major component of petroleum that is heavily employed to derive energy in the Nigerian society. Most industries use diesel machines during production. In the transportation sector, private vehicles, buses, trucks, and ships also consume significant amounts of diesel and gasoline [1, 4, 5]. The combustion products of conventional diesel (and other fossil fuels) are very harmful to the environment and atmosphere, thereby, contributing significantly to global warming and climate change. The human health impact of diesel exhaust emissions is also of growing concern. The diesel exhaust contains a complex mixture of particulate and gaseous pollutants such as carbon monoxide, carbon dioxide, nitrous oxides, benzene, 1, 3-butadiene, formaldehyde, soot and many other particulate matter [6]. Many international laws have been promulgated to regulate the use of fossil fuels for energy generation because of the associated environmental and health implications [7, 8]. Moreover, it has been speculated that hydrocarbon-based fossil fuels, such as petroleum, are becoming depleted in nature [9, 10]. This has continued to encourage engineers and scientists to undertake research aimed at developing highly efficient and sustainable alternative sources of energy.

Biodiesel has attracted significant attention as a complement to conventional fossil-derived diesel due to some salient properties. These include environmental safety, renewability, biodegradability and higher flash point and cetane number [11, 12]. However, the corrosion-inducing properties of biodiesels is a serious challenge for diesel engine parts. Biodiesel is always considered as an oxygenated fuel that contains mono-alkyl esters of long-chain fatty acids (C_{14}–C_{24}) [13, 14]. Hence, unstable and oxidatively-reactive. Due to this propensity, it can easily attack engine components such as fuel tank, fuel pump, fuel filter, engine valve, piston, and cylinder liner [15]. The presence of saturated and unsaturated fatty acids in biodiesel makes it quite susceptible to oxidation over time. The aqueous corrosive ions which are produced via this oxidation could also promote microbial growth and may hydrolyse the methyl esters to produce more corrosive fatty acids. This increases the total acidity of biodiesel and, hence, makes it highly corrosive [15]. Biodiesel is more conductive and corrosive than fossil diesel because of the presence of oxygen in biodiesel and the electronegativity of oxygen. This property enables biodiesel to inflict corrosion damage on metal surfaces. The conductivity arises from the electrolytic dissociation of organic fatty acids, water or esters contained in biodiesel. These species dissociate into radicals or ion groups that contain oxygen (which acts as the main factor that enhances the corrosiveness of biodiesel). Additionally, biodiesels naturally contain microorganisms and the presence of aerobic and anaerobic microorganisms also increases biodiesel's acidity and further contribute to biodiesel-induced corrosion [14].

Jatropha curcas, is a drought-resistant shrub or tree that grows wild or in semi-cultivated environments [16]. The oil derived from *Jatropha curcas* can be easily trans-esterified into a liquid biofuel [11]. Jatropha seed oil possesses biodiesel and jet fuel production potentials. *Jatropha curcas* may be commercially grown as a crop or as a hedge to protect fields from grazing animals and also to prevent erosion in areas that experience low to high rainfall (Kumar and Sharma, 2008). Moreover, the residual press cake (after the oil extraction) is useful as a fertilizer, and as organic waste materials which can be digested to produce methane-rich biogas [8, 18]. Among the crops identified as energy crops for first generation biofuels, *Jatropha curcas* has been acknowledged as one of the promising candidates [19]. Jatropha oil contains approximately 24.60% of crude protein, 47.25% crude fat and 5.54% moisture. The oil also contains both saturated and unsaturated fatty acids. The major saturated fatty acids are Palmitic acid (16:0) at 14.1% and stearic acid (18:0) at 6.7%, oleic acid (18:1) at 47.0% and linoleic acid (18:2) at 31.6% [16, 20, 21]. It is abundantly available as a non-edible crop.

Over the years, the corrosion impact of jatropha biodiesel on metallic materials used for fabricating diesel engine parts has been investigated and reported [7, 22-27]. Iron forms the base metal component of steel and is readily oxidized in wet environments. This makes steel highly susceptible to corrosion when exposed to biodiesel. In a diesel engine, fuel usually makes contact with different types of materials while it passes from the fuel tank to the combustion chamber. The main components which are most affected by biodiesel include the fuel tank, fuel pump, fuel filter, engine valve, piston, and cylinder liner [15]. The rate of the corrosion of these components depended on the physicochemical properties of the biodiesel and its parent plant feedstock. Oxidation and several other chemical reactions convert biodiesel esters into several mono-carboxylic acids which further accelerates the corrosion rate of the materials. Steel constitutes the most employed material used to manufacture most of these engine components. The importance of steel is based on its excellent strength, natural abundance and ease of alloying with other elements. This has continued to encourage scientific investigations to understand the corrosion properties of steel-based alloys in biodiesel derived from plant sources. For this purpose, conventional research focuses on non-edible biodiesel sources which do not affect food supply and abundance. Hence, the choice of jatropha biodiesel obtained from non-edible oil source which is in abundance locally.

Fazal *et al.* [28] used x-ray diffraction technique to confirm that the major corrosion products formed on steel during corrosion in biodiesel were iron products of corrosion. This mechanism was attributed to the presence of oxygen and water in biodiesel. Shahabuddin *et al.* [8] investigated the corrosion behaviour of different automotive materials that included stainless steel, aluminium, cast iron, and copper in different volumes of jatropha biodiesel using static immersion test and scanning electron microscopy (SEM). The study revealed that the highest corrosion rate occurred on copper, while the lowest was detected in stainless steel. Akhabue *et al.* [26] investigated the effect of biodiesel fuel made from jatropha curcas seed oil on the corrosion rates of mild carbon steel and aluminium, and compared this biodiesel corrosion rates with the corrosion impact of the conventional petroleum diesel and its blend. The results showed that the corrosion rates for the tested alloys were higher in the biodiesel compared to the conventional fuel and when the biodiesel was combined with the conventional fuel. Ahmad *et al.* [24] investigated the corrosion of copper, stainless steel and aluminium alloys in jatropha biodiesel to determine concentration of fatty acids in the biodiesel. They observed the presence of oleic, linoleic, palmitic, and stearic acids as the major fatty acids. Spectroscopic and structural studies were however not conducted. Dharma *et al.* [23] investigated the corrosion behaviour of mild steel immersed in fuel blends of jatropha curcas biodiesel and ceiba pentandra biodiesel to determine corrosive rate and acid value. No detailed characterization except SEM and weight loss was conducted. Akhabue and Nduka [25] investigated the corrosion behaviour of brass, galvanized steel and stainless steel in blends of jatropha biodiesel and diesel via weight loss method, visual observation and physicochemical characterization. The corrosion rates were observed to increase with increases in the volume of the diesel added. Detailed spectroscopic or morphological investigation were however not carried out. Chourasia *et al.* [22] investigated the behaviour of numerous biodiesel including jatropha biodiesel corresponding to corrosion when exposed to the surface of metal coupons. Their results showed high corrosion rate degradation.

In all of these reports, the authors have only focused on understanding the rate of corrosion of the alloys without considering the understanding of the mechanism by which these corrosion phenomena occur. This is important so as to shed more light on the appropriate selection of steel alloy for fabrication of specific engine part which have constant contact with the biodiesel. Such mechanisms could be understood by undertaking detailed spectroscopic and surface investigations. This analysis will explain the mode of surface interaction between the organic molecules in the biodiesel and the metal atoms released from the alloy surface during the corrosion. Furthermore, the spectroscopic measurement technique will enable us to understand the nature of chemical reaction between the organic molecules in the biodiesel and the metal atoms from mild steel surface.

It is generally acknowledged that diesel fuel does not have corrosive effect as much as biodiesel on engine parts exposed to such fuel. Thus, continuous exposure of biodiesel fuel in engine and fuel lines are important areas of investigation as such examination would enhance the compatibility of various metallic materials toward the use of the non-blended biodiesel fuels. However, to date, research on the use of only jatropha biodiesel fuel in corrosion study as well as the corrosion behaviour of different carbon alloys in jatropha biodiesel fuel is still limited, to the best of the authors' knowledge. Therefore, this study is significant in that it examines and provides in-depth spectroscopic and surface investigations of the corrosion behaviour of mild steel in pure jatropha biodiesel fuel. In the present study, UV-Vis and Fourier transform infrared (FTIR) spectrophotometers were employed to probe for any iron-organic species interaction during metal immersion while optical surface microscopy of the corroded steel surface was investigated after the various immersion periods to provide an idea on the extent of surface degradation caused by the biodiesel.

2. MATERIALS AND METHODS

2.1 Materials

The materials and reagents used in the present study include, jatropha biodiesel, methanol, potassium hydroxide pellets, polishing papers (#400, #600 and #800 grit sizes), thread, mild carbon steel coupons, analytical grade acetone, ethanol, hydrochloric acid and diethyl ether. The equipment used include Buchler torramet specimen dryer, Cary 60 UV-Vis, spectrometer, Shimadzu IR Affinitty-1, Japan FTIR equipment, and a high-resolution camera (ANDOR camera series iDUS CCD) with 1024 x 256 pixels, 26 um pixel size, peak QE of 95% (Vis) made by Oxford Instruments, U.K.

2.2 Methods

2.2.1 Original source and preparation of jatropha biodiesel fuel

The jatropha biodiesel fuel was produced from jatropha oil using transesterification process. The biodiesel production was performed using two-step reaction involving acid-catalysed esterification and base-catalysed transesterification. The jatropha oil had an initial value of 23.35 mg KOH/g as free fatty acid value which was above the 0.50 mg KOH/g (1%) value of oil limit for satisfactory direct transesterification reaction using a base catalyst. Hence, the free fatty acid was first converted to esters in a pretreatment process with methanol using a molar ratio of oil to methanol of 1:12 at reaction temperature of 60 ℃ and reaction time of 120 mins as reported by several researchers [16, 20]. The base-catalysed transesterification of the esterified jatropha oil was then conducted using methanol as the alcohol and potassium hydroxide as the catalyst. The transesterification process was investigated at a molar ratio of 6:1 for methanol to jatropha oil following similar procedures reported by different authors [16, 20].

2.2.2 UV-Vis spectroscopic investigation

The jatropha biodiesel samples obtained after immersion of the mild carbon steel coupons for the investigated days were analysed separately using UV-Vis spectrometry at the investigated conditions. UV-Vis spectroscopy was used to identify any complexion product as well as any chemical interaction between the metal and the jatropha biodiesel samples. The UV-Vis spectroscopy was carried out using Cary 60 UV-Vis, Agilent Technologies, U.S.A equipment. The ultra violet–visible (UV–VIS) experiments were conducted, after each immersion time within the range of (200–650 nm) using a dual beam operated at a resolution of 1 nm with a scan rate of 200 nm min^{-1}.

2.2.3 FTIR measurement

Fourier transform infra-red spectrometry (FTIR) was used to identify the characteristic functional groups (individual molecules) present in the jatropha biodiesel. The FTIR analysis of the biodiesel was performed using a Shimadzu IR Affinity-1, Japan spectrometry equipment. The FTIR measurements were conducted at the optimized immersion period for each of the jatropha biodiesel samples. 0.4 g of potassium bromide (KBr) was weighed and ground to powder. 0.001 g of jatropha biodiesel was weighed into the ground KBr and both were thoroughly mixed together and moulded into a disc. The disc was then inserted into the sample compartment of the FTIR instrument. The scan button was pressed and the IR spectrum generated. The sample was scanned from 1200 cm^{-1} to 3600 cm^{-1}. The sample analysis process included the source, the interferometer, the sample, the detector and the computer which generated the final spectrum for interpretation.

2.2.4 Optical micrograph investigation

Optical micrographs or images were obtained using a high-resolution camera (ANDOR camera series iDUS CCD) with 1024 x 256 pixels, 26 um pixel size, peak QE of 95 % (Vis) made by Oxford Instruments, U.K. The equipment was employed to study the interactions between the jatropha biodiesel molecules and the steel surface at the optimized immersion periods in order to generate and elucidate on the effects of surface degradation due to the jatropha biodiesel molecules.

3. RESULTS AND DISCUSSION

3.1 UV-Vis Spectroscopic Analysis

Figure 1 shows the UV-Vis spectrum after 14, 42 and 56 days of immersion. The immersion periods were chosen based on jatropha oil biodiesel effects on other metal alloys as reported by other researchers [7, 22-24, 26, 28]. Thus, after 14, 42 and 56 days of immersing the mild steel in the jatropha biodiesel, the solution was analysed with UV-Vis spectrophotometer in order to unravel any chemical interaction between the biodiesel molecules and the steel surface. The most visible peak is observed around 900 nm and 1050 nm and is consistent with the signals due to poly-unsaturated components of the biodiesel. Such poly-unsaturated compounds include linoleic acid methyl ester containing C_{18} molecules [3, 26]. This result agrees with different studies conducted by several researchers that showed that the linoleic acid was majorly responsible for attacking the steel surface and inducing the biodiesel corrosion [3, 5, 27, 28]. Figure 1 further shows that the intensity of the C_{18} peak decreases over the time of immersion. This observation could be an indication that the components of the carbon steel were continuously depleted in the biodiesel solution as a result of their adsorption on the steel surface [3, 5, 26, 29].

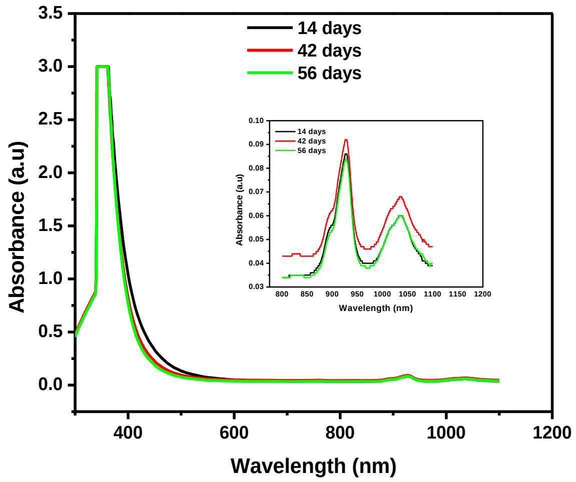

Figure 1: UV-Vis spectra for mild steel immersed in jatropha biodiesel at 14 days, 42 days, and 56 days

3.2 FTIR Spectrometry Analysis

To confirm the various functional groups, present in the biodiesel before and after immersion of the carbon steel in the biodiesel molecule, FTIR spectroscopy was used. The FTIR spectra in Figure 2 shows the profile of the jatropha biodiesel before mild steel immersion and the corrosion product layer formed on the steel surface after 56 days of immersion in the biodiesel. The peak around 1700 cm^{-1} indicates the presence of C=O that is present in the triglycerides. The peak signal around 2950 cm^{-1} is attributed to anti-symmetric and symmetric stretching vibrations of C-H in CH_2 and CH_3 [30, 31]. The peaks around 1150 cm^{-1} indicated the stretching vibration of C-O ester. These peaks in the pure biodiesel are also present for the corrosion product scale formed on the steel surface after the longest immersion time. This indicates that the biodiesel interacts with the steel surface using these functional groups (especially the C-O and C=O groups). This finding supports the results obtained from UV-Vis analysis and confirms that the biodiesel attacks the steel surface using its organic acids (most possibly the linoleic acids) [3, 32].

The various functional groups in the raw jatropha oil and produced jatropha biodiesel was also ascertained by the use of FTIR spectrometry. Figure 3 shows the FTIR spectra obtained for the raw jatropha oil and the produced jatropha biodiesel. Observation of the figure shows that the spectrum contains peaks at almost the same position with few exceptions. This suggest that the jatropha oil and biodiesel have similar functional groups except in few locations even after the oil has undergone transesterification reaction to produce the biodiesel [32].

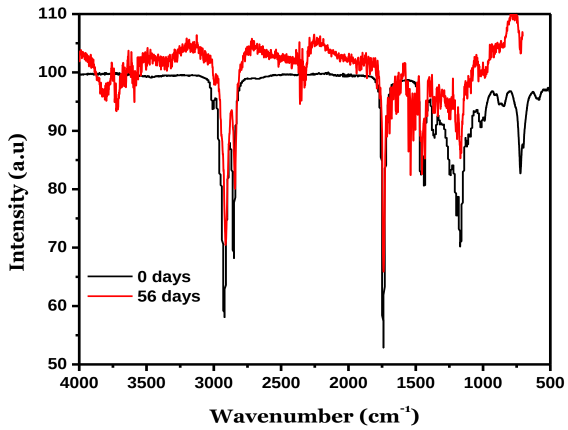

Figure 2: FTIR spectra for mild steel immersed in jatropha biodiesel at 0 day and 56 days

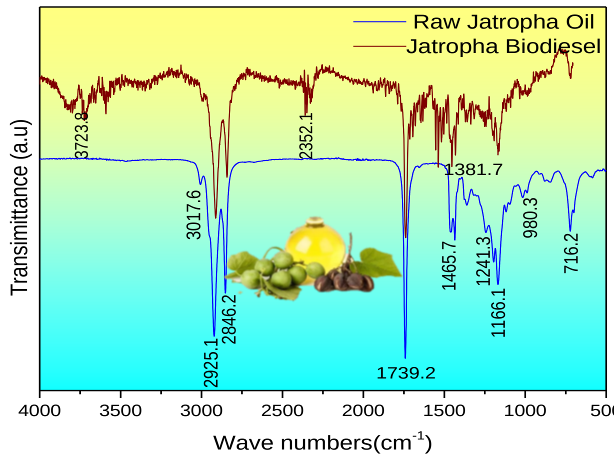

Figure 3: FTIR spectra for raw jatropha oil and jatropha biodiesel

3.3 Optical Microscopic Analysis

Optical micrographs or images were obtained using a high-resolution camera (ANDOR camera series iDUS CCD) with 1024 x 256 pixels, 26 um pixel size, peak QE of 95 % (Vis) made by Oxford Instruments, U.K. The equipment was employed to study the interactions between the jatropha biodiesel molecules and the steel surface at the optimized immersion periods in order to generate and elucidate on the effects of surface degradation due to the jatropha biodiesel molecules as shown in Figure 4. It was observed that the steel surfaces did not have even or uniform microstructure. After 42 and 56 days of immersing the steel coupons in the jatropha biodiesel fuel, the resultant surface feature is presented in Figure 4. From the figure, it is believed that the metal atoms possess higher energies that make them transport from the interior of the alloy matrix towards the alloy surface where they participate in the corrosion reactions [4, 33, 34]. There were pores and light agglomeration of structures observed on the steel surfaces and this was assumed to influence the corrosivity of the immersed steel coupons in the jatropha biodiesel fuel [15, 32]. Patches of corrosion deposition around the sparely dispersed steel surfaces were also observed on the micrograph which in turn influenced the corrosion of the steel material [23, 28].

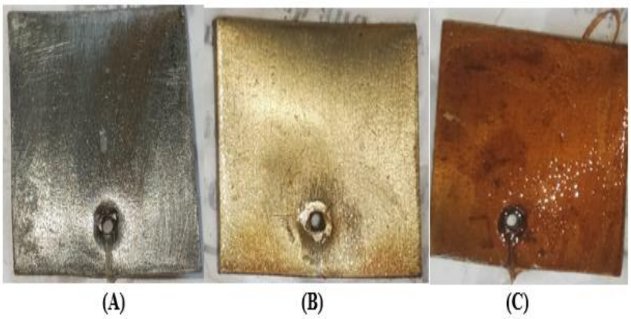

Figure 4: Optical microscopic images of mild steel immersed in jatropha biodiesel at (a) 0 day (b) 42 days (c) 56 days showing different degrees of corrosion degradation with time

Additionally, Figure 4 shows the resultant steel surface after 0, 42 and 56 days of immersing the steel sample in the Jatropha biodiesel. The regions where corrosion is initiated can be seen in the gradual change in complexion or colour in the steel surfaces. Some patches or pits could be seen on the carbon steel surface as indications of localized attack by the biodiesel components [15, 33]. Pitting corrosion is usually attributed to local corrosion cells created by the scratches made on the sample during sample preparation by polishing. Within these regions, it is believed that metal atoms possess higher energies that make them transport from the interior of the alloy matrix towards the alloy surface where they participate in the corrosion reactions [1, 22, 32]. After 56 days, the surface of the steel has become highly corroded with somewhat brownish scale deposited on the steel surface. This implies that time increases the rate of attack of the steel surface by the biodiesel components [3, 16, 35-37]. Furthermore, from the images of Figure 4, it could be observed that at day 42 and 56, corrosion degradation is setting in during early immersion in the biodiesel due to the attack of the mild steel surface by the fatty acid components of the biodiesel. After 56 days of immersion, corrosion degradation has become more intense due to the much longer immersion period occasioned by the aggressive attack on the steel surface by the biodiesel molecules. This result shows that over time, diesel engine parts made of steel would suffer corrosion-related surface degradation which would diminish their structural integrity and also contaminate the diesel system.

4. CONCLUSION

Corrosion of engine parts exposed to biodiesel still remains a major challenge in the automotive industry. In a direct compression engine, biodiesel fuel usually makes contact with different types of materials while it passes from the fuel tank to the combustion chamber. The rate of corrosion of these components is dependent on a number of salient factors. In this study, biodiesel molecule-mild steel surface interphase interactions were investigated for corrosion degradation during immersion for 14, 42 and 56 days respectively. Surface and solution investigations were conducted using UV-Vis spectroscopy, Fourier Transform Infrared (FTIR) spectroscopy and optical microscopic techniques. UV-Vis spectra analysis confirmed the presence of C_{18} fatty acid group as the major acid responsible for the attack on the steel surface which induce the corrosion process through physical adsorption on the steel surface. FTIR investigation further established the fact that the jatropha biodiesel interacted with the steel surface using C-O and C=O functional groups which further corroborated with that obtained from UV-VIS analysis. Thus, the biodiesel attacked the steel surface using the organic acids. Optical microscopic characterization confirmed that the steel surface suffers corrosion related surface degradation due to localized attacks by the biodiesel components from acidification within the biodiesel that release excessive fatty acid over time; hence the increased attack on the steel surface. Thus, carbon steel alloy material selection and testing to determine compatibility and suitability is important in the choice of how and when a biodiesel fuel is to be used in an automotive engine.

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