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ELECTROCHEMICAL AND MICROSTRUCTURAL ANALYSIS OF MILD STEEL INTERACTIONS IN JATROPHA BIODIESEL FUEL

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Abstract: Jatropha biodiesel fuel as a renewable energy source has gained prominence over time. The utilization of jatropha biodiesel fuel and its effects on engine components in automobiles needs to be interrogated to provide sufficient data on metal degradation and solution contamination. Electrochemical (EIS, OCP and PDP) and morphological (SEM) studies on the behaviour of mild steel immersed in jatropha biodiesel fuel is investigated in this research. The study elucidated on the jatropha biodiesel fuel-metal surface interactions at room temperature (25℃) and different time intervals (0, 7, 28, 42 and 56 days). Electrochemical measurements established that mechanism of corrosion of the mild steel surface was physical and this corroborated with morphological investigations which showed gradual but consistent surface degradation over time due to material dissolution in the biodiesel fuel (corrosion rate (CR) of 103 mpy, corrosion potential (E_{corr}) of -586 mV/SCE, corrosion current (i_{corr}) of 16.23 μA/cm^2, and standard deviation of 0.44). Thus, the degradation of mild steel in jatropha biodiesel fuel showed high level of surface instability resulting in significant corrosion over time and therefore provided a means of ascertaining the compatibility and effectiveness of the mild steel with regards to the specific biodiesel fuel in automobile engines. Essentially, the corrosion attack arose mainly due to the presence of C_{18} fatty acid groups in the jatropha biodiesel fuel. To mitigate the attack on the mild steel by the fatty acid molecules, corrosion inhibitors that are environmentally friendly, non-reactive and non-toxic could be incorporated into the fuel.

Keywords: Metal alloy; Biodiesel fuel; Surface deterioration, Surface studies; electrochemical analysis.

1.0 INTRODUCTION

Recent advances in the direction of renewable energy fuels has been heightened by the rather unstable nature of fossil fuel energy demand and supply occasioned by the world energy crises globally. The development of these alternatives, are largely based on non-edible sources which are readily available and cultivated all year round. The inherent nature of the conflict between food and energy utilization and consumption has prompted the continued used and adaptation of non-edible source as the major feedstock for biomass-based fuels. Jatropha oil seeds has found readily available avenue for this biofuel due to its abundance and non-competitive edible nature (Monteiro *et al.,* 2018; Chandran, 2020; Kugelmeier *et al.,* 2021).

Jatropha biodiesel fuel obtained from jatropha oil seeds via transesterification reaction is a major source of renewable energy fuel for automobiles and other domestic purposes (Adama, 2021). Its use in this regard has been acknowledged globally and there has been greater interest in its production and utilization for vehicular activities. However, there has been limited actual utilization on a commercial scale of the application of the jatropha biodiesel fuel in the automobile industry due to some of the inherent properties associated with the biofuel. These properties include the fact that the chemical composition and other characteristics like high viscosity, auto-oxidation (oxidative instability), and high volatility, makes biodiesel to be corrosive to automobile and industrial engine parts that run on diesel (Fernandes *et al.,* 2013; Jakeria *et al.,* 2015; Fazal *et al.,* 2019). Notwithstanding these shortcomings, the advantageous properties in the use of jatropha biodiesel fuel in engines include higher inherent lubricity, higher cetane number, and flash point, very low contents of aromatic and sulfur compounds, and fewer exhaust emissions (Ahmmad *et al.,* 2018; Alves *et al.,* 2019; Fazal *et al.,* 2019; Adama 2021).

Different studies have enunciated the corrosion inducement properties of jatropha biodiesel fuel on different metal types in terms of their degradation and corrosion abilities. Understanding the behaviour of these metals in the biodiesel fuel has helped to demonstrate the ability of these metals to withstand corrosive attacks when such metals are immersed in the jatropha biodiesel fuel. The use of both ferrous and non-ferrous metals in jatropha biodiesel fuels were investigated by Kaul *et al.,* (2007). They compared the corrosive characteristics of *Jatropha curcas, Karanja, Mahua,* and *Salvadora* biodiesels with those of diesel fuel and observed that *Jatropha curcas* and *Salvadora* biodiesels were more corrosive for both ferrous and non-ferrous alloys as compared to *Karanja* and *Mahua* biodiesels. However, they were unable to ascertain the electrochemical behaviour and morphological structure of the metals in the jatropha biodiesel fuels as well as the fossil diesel fuel. Ahmmad *et al.,* (2018) observed the presence of different fatty acid in jatropha biodiesel fuel on three different metals investigated for corrosion degradation by the biodiesel fuel. They noted that the attack on the metal alloys were evidently linked to the presence of the different fatty acids in the biodiesel fuel. Dharma *et al.,* (2019) investigated the corrosion behavior of mild steel immersed in blends of *Jatropha curcas* biodiesel and *ceiba pentandra* biodiesel fuels and characterized the resulting metal surface via scanning electron microscope (SEM). Others studies on the degradation of different metals in jatropha biodiesel fuel were carried out by Akhabue and Nduka, (2016), Akhabue *et al.,* (2014) and Chourasia *et al.,* (2020). Interestingly, these studies mostly investigated the weight loss and other properties of jatropha biodiesel fuel with limited information on the electrochemical mechanism and surface analysis of the resulting metal microstructural surface and solution interactions. This research seeks to elucidate on this identified gap by providing detailed and comprehensive analysis on the mechanism of electrochemistry associated with the interactions of jatropha biodiesel fuel with mild steel as well as time-evolved microstructural features of the alloy material in the jatropha bio-fuel. The findings would make significant contributions to research on alternative fuel utilization.

2.0 MATERIALS AND METHODS

2.1 Materials

The materials and reagents employed in this research include, *Jatropha* biodiesel fuel, analytical grade chemicals such as methanol, potassium hydroxide pellets, acetone, ethanol, hydrochloric acid, and diethyl ether purchased from Sigma Aldrich and used without further purification. Polishing papers, thread, mild steel coupons were obtained from Faculty of Engineering Central workshop of Edo State University Uzairue, Edo State, Nigeria. The equipment used included immersion bottles, Buchler torramet specimen dryer, Carl Zeiss EVO MA-10 high resolution scanning electron microscope (SEM) and Gamry 600 potentiostat equipped with the Echem Analyst software for data analysis.

2.2 Methods

Jatropha seeds are a good source of jatropha oil which are already been used to produce jatropha biodiesel fuel (Adama, 2021; Della-Torre *et al*., 2021; Mohammed-Dabo *et al*., 2012). Jatropha biodiesel fuel was produced in this research from jatropha oil via two-step esterification-transesterification reaction following procedures earlier reported (Adama, 2021). The characterization procedures, physicochemical analysis, fatty acid profiling, and fuel property analysis of the jatropha biodiesel fuel were as reported in an earlier study (Adama 2021; K.K. Adama *et al.,* 2023). The elemental composition of the mild steel coupons used in this research was reported in an earlier study (Adama and Onyeachu, 2023).

2.2.1. Electrochemical investigations

The electrochemical investigations were used to elucidate on the mechanistic properties of the mild steel in the jatropha biodiesel fuel. The information obtained from the electrochemical studies would include dielectric and charge transfer phenomena at the mild steel-biodiesel solution interface, corrosion potential, corrosion kinetics, passivation, and passivation breakdown and any pitting corrosion tendencies. The electrochemical experiments conducted include open circuit potential (OCP), electrochemical impedance spectroscopy (EIS) and potentiodynamic polarization (PDP). The experiments were conducted using a Gamry 600 potentiostat equipped with the Echem Analyst software for data analysis. For system stability and reduction in the effects of charging current, free dissolution potential of mild steel in the jatropha biodiesel fuel was determined before the commencement of other electrochemical analyses by recording the open circuit potential (OCP) for 1800 s. Hence, all the electrochemical tests were accomplished at OCP condition (Adama and Onyeachu, 2023; Onyeachu *et al.*, 2022). The electrochemical tests were performed in a cell assembly made up of three electrodes namely; mild steel; as the working electrode (WE), silver/silver chloride (Ag/AgCl); as reference electrode (RE). The reported potentials in this research are with regards to Ag/AgCl while platinum wire was employed as counter electrode (CE). A 3.5 % NaCl solution was chosen as the electrolyte for the current investigation so as to simulate a near seawater environment. The EIS was measured using a signal amplitude perturbation of 10 mV within a frequency range of 100 kHz–10 mHz. The potentiodynamic polarization analysis was conducted at the potential range of $\pm 250\ mV$versus OCP at scanning rate of 0.2 mV s^{-1}.

2.2.2. Microstructural characterization

Scanning electron microscopic (SEM) investigation was utilized to elucidate on the surface morphology of the mild steel before and after immersion in the *Jatropha* biodiesel fuel. The microstructure of the mild steel was investigated using an EVO MA-10, Carl Zeiss, Hamburg, Germany, high-resolution scanning electron microscope that was operated at 20 kV. The mild steel coupons were washed, cleaned, and polished repeatedly after each immersion periods using a conductive carbon phase to enhance their conductivity and were held firmly on the sample holder using double-sided carbon tape before being inserted inside the sample chamber.

3.0 RESULTS AND DISCUSSION

3.1 Electrochemical Analysis

3.1.1 Open circuit potential (OCP)

The open circuit potential (OCP) for the mild steel immersed in the jatropha biodiesel fuel is shown in Figure 1. At the beginning of the electrochemical measurements, it was essential to achieve a steady state condition (Onyeachu *et al.,* 2022). Hence, in the absence of current, OCP was first recorded. Careful observation showed that the jatropha biodiesel fuel started developing stable line which was an indication that the allocated time was adequate to attain the steady state condition. The average OCP tended toward more negativity indicating that the mild steel was corroding in the biodiesel fuel. The trend was observed to stabilize after 1800s. Thus, the plot similarly indicated a large variation in the OCP with respect to time. At the inception, the jatropha biodiesel fuel tended more to negative direction indicating the degradation of mild steel in the biodiesel fuel. Ultimately, the jatropha biodiesel fuel became more passive with time implying that the jatropha biodiesel fuel was very corrosive on the mild steel alloy.

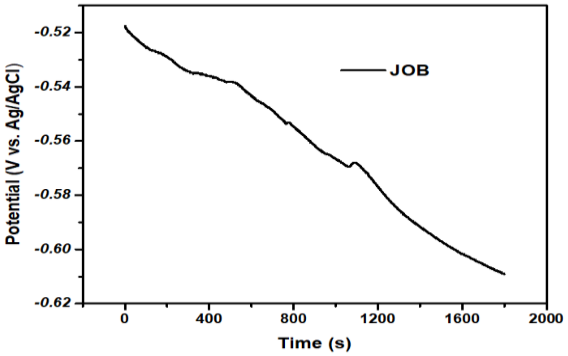
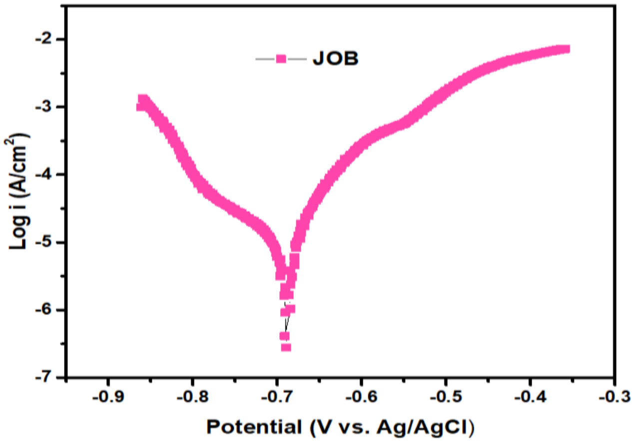


Figure 1: Open Circuit Potential (OCP) for Mild Steel in Jatropha Biodiesel Fuel.

3.1.2 Potentiodynamic polarization (PDP)

Potentiodynamic polarization analysis was carried out to further probe into the interaction of the mild steel in the jatropha biodiesel fuel. The investigation revealed the variation in the anodic and cathodic current density created by the mild steel in the jatropha biodiesel fuel from the partial reactions taking place in the fuel. Figure 2 shows the Tafel plots of the potentiodynamic polarization for mild steel in the jatropha biodiesel fuel. The plot for the mild steel in the jatropha biodiesel fuel illustrates the cathodic and anodic currents generated in the course of electrochemical corrosion reactions which is actually a redox reaction that occurred when the metal was immersed in the biodiesel medium. Thus, the left and the right hand sides represents the anodic and cathodic activity of the mild steel in the jatropha biodiesel fuel. The shifting of the curve to higher corrosion current densities is an evidence of the mild steel corrosion in jatropha biodiesel fuel which corroborates with the results obtained in the OCP. The quantitative values of all the polarization parameters for the jatropha biodiesel fuel is presented in Table 1. From the table, it can be observed that the corrosion density shown by jatropha biodiesel fuel is high. The implication is that more electron is loss due to excess current (Tian *et al.,* 2017). This observation supports the finding that the jatropha biodiesel fuel successfully corrodes the mild steel immersed in the medium. The salient parameters resulting from the technique employed is shown in Table 1 with the corrosion potential and current density values (Table 1). The anodic and cathodic Tafel slopes values (βa and βc, respectively) of the jatropha biodiesel fuel are also presented in Table 1. The corrosion effects of the mild steel in the jatropha biodiesel fuel is computed as $103\,mpy$ representing the corrosion rate in the fuel as generated automatically from the EC-Lab 10.40 software by inputting into the software the equivalent weight of mild steel.

The values in Table 1 compare favorably with similar results obtained by Deyab and Keera (2016) in their study of the corrosion and corrosion inhibition of carbon steel in stored biodiesels. However, limited studies presently exist on biodiesel-induced corrosion studies to the best of the authors' knowledge.



4.0

Figure 2: Tafel Polarization Curves (PDP) for Mild Steel in Jatropha Biodiesel Fuel

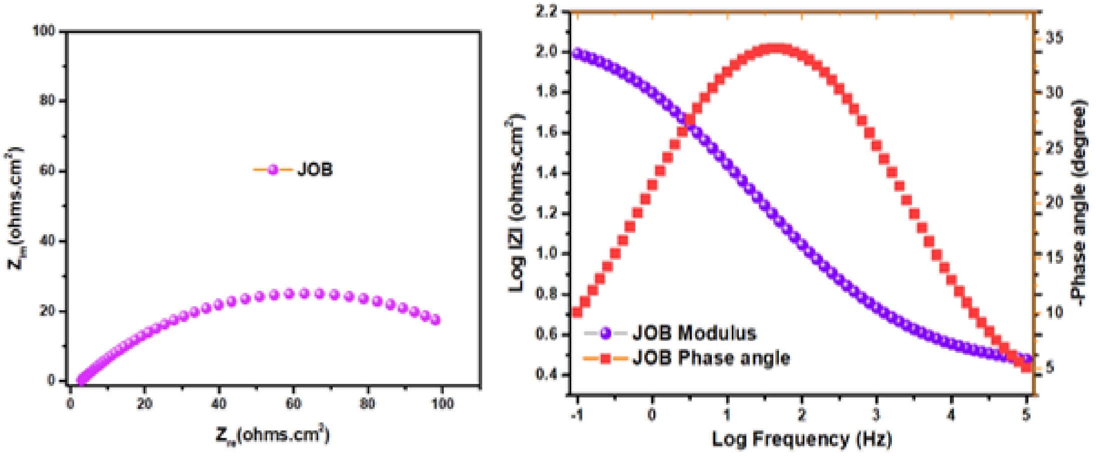
Table 1: Tafel Polarization Parameters for Mild Steel in Jatropha Biodiesel Fuel

Biodiesel	E_{corr} (mV/SCE)	i_{corr} (μAcm^{-2})	*CR* (mpy)	Bc (mV)	Ba (mV)	SD
Jatropha Biodiesel	-586	16.23	103	245	46	0.44

Where, CR = Corrosion rate; E_{corr} = Corrosion potential; i_{corr} = corrosion current; β_C = Cathodic Tafel constant; β_a= Anodic Tafel constant; SD = Standard deviation

3.1.3 Electrochemical impedance spectroscopy (EIS)

Figure 3 (A) shows the Nyquist plot for EIS data for the mild steel in the jatropha biodiesel fuel. Figure 3 (B) also presents the Bode plots for EIS data for the mild steel in the jatropha biodiesel fuel. The EIS provides a deeper understanding and knowledge of the kinetics occurring at the mild steel-jatropha biodiesel fuel interface. The figure showed single depressed capacitive loop dominated by charge transfer phenomena at high and low frequency regions (Berdimurodov *et al.,* 2021; Yadav *et al.,* 2013). Similarly, the nature of the depressed semicircle represents recognized features associated with a solid electrode. In practical terms, the impedance response demonstrated a depressed semicircle due to dispersion frequency as a result of the fractal and electrode geometry. The rise in the semicircle is an indication of reduced electron transfer process taking place at the mild steel-jatropha biodiesel fuel interface which is represented by the Nyquist plots (Figure 3A). Furthermore, Figure 3A indicates that the mild steel corrodes in the jatropha biodiesel fuel which is a demonstration of the typical deterioration behaviour of the mild steel in the biodiesel fuel. The semicircle is the capacitive loop showing evidence of corrosion product accumulation at the mild steel surface-jatropha biodiesel fuel interface. In the Figure 3A shown, the Nyquist curve displays irregular suppressed semicircle due to frequency dispersion. This observation correlates to non-ideal nature of the mild steel surface which is a reflection of the roughness and non-homogeneity of the surface. In addition, the diameter of the capacitive loop represents the charge transfer resistance ('Rct') for mild steel surface corrosion. However, the Nyquist plot does not display evidence of frequency of the corrosion dispersion. Hence, the necessity for a unique representation of the plot using the Bode plots. Figure 3B shows the modulus and phase angle representations. Careful observations shows that there was gradual formation of film layer or scales in the presence of the jatropha biodiesel fuel due to the formation of greasy-like deposit on the mild steel surface. The mild steel remained in contact with the jatropha biodiesel fuel-mild steel interface with the formation of a new layer, thus matches with one-time constant circuit representation (Deyab and Keera, 2016). The evidence of corrosion product accumulation at the double layer is seen in the rise in both curves. This indicates that the mild steel actually corroded in the jatropha biodiesel fuel (Deyab and Keera, 2016).



(A) **(B)**

Figure 3: (A) Nyquist **(B)** Bode plots of Electrochemical Impedance Spectroscopy (EIS) for Mild Steel in Jatropha Biodiesel Fuel

3.2 Microstructural Analysis

3.2.1 Scanning Electron Microscopy (SEM)

The surface morphology of the mild steel coupons were examined in two static states using high resolution scanning electron microscope: before immersion and after immersion in the Jatropha biodiesel fuel at the different investigated periods of 0 day (before immersion), 7, 28, 42, and 56 days (after immersion) respectively as shown Figure 4 (a – e). In analyzing the morphology of the mild steel surfaces, SEM observations of the relevant surfaces were based on the number of days the mild steel were exposed to the Jatropha biodiesel fuel. The micrographs (Figure 4 (b-e)) exhibits rough surfaces after the various immersion periods with patches of passive products on the surface of the mild steel. With the increase in immersion periods (days), the passive product of corrosion is observed to agglomerate into continuous and dense aggregates thus providing greater surface corrosion coverage for the mild steel. Further analysis shows corrosion materials which could likely be portions of localized corrosion (Adama and Onyeachu, 2022). The micrographs further shows complete structural surface damage with different degrees of degradation due to the localized attacks which were physically induced. It could be deduced that as the length of days increased, the number of shapes or structures per unit area increases and the shapes and sizes become larger (Chuck *et al.*, 2010; Fardilah *et al.*, 2022). As seen in the figures, a complete structural coverage of the steel coupon was obtained on the mild steel surface at different periods (days) showing high levels or degrees of degradation over time (Kugelmeier *et al.*, 2021; Onyeachu *et al.*, 2022). Figure 4a shows the topographical features of the mild steel before immersion in the biodiesel fuel. The surface structure reveals uniform and even microstructure with no deposition of corrosion product because of non-contact of the fatty acids associated with the biodiesel fuel. There had been no mild steel-biodiesel interactions at this stage. Figure 4b shows the morphology of the immersed mild steel in the jatropha biodiesel fuel after 7 days. It could be observed that the surface structure gradually began to show evidence of corrosion degradation as a result of the deposition of corrosion product on the mild steel surface. Some pits, crevices and solid materials evenly distributed could be observed on the mild steel surface as indications of localized attacks by the biodiesel component (David *et al.*, 2019; Monteiro *et al.*, 2018). Within this region, it is believed that metal possess higher energies that makes them transport from the interior of the alloy matrix towards the alloy surface where they participate in the corrosion reaction (Onyeachu *et al.*, 2022 ; Kugelmeier *et al.*, 2021; Monteiro *et al.*, 2018). Figure 4c similarly reveals the structural degradation of the mild steel surface by the fatty acid components of the biodiesel after 28 days of immersion. The figure reveals a more broadened and deeper degradation evident by the agglomeration and clustering of the microspores of the corrosion product with increase in duration of immersion. The topographical features exhibited after 28 days of immersion became more pronounced and evenly spread on the mild steel surface due to the more aggressive and intensive attack by the fatty acid molecules of the biodiesel on the mild steel surface. The surface has become highly corroded with corrosion products which has become more pronounced. The same scenario was observed at 42 days and 56 days after immersion as shown in Figures 4d and 4e respectively. The intensity of attack shown in Figure 4e seems to be more aggressive due to continuous acidification of the fatty acid molecules of the jatropha biodiesel with time; hence, increased corrosion degradation after 56 days of immersion. From a time-dependent perspective, it could be stated that time-lag also increased the extent of acidification within the jatropha biodiesel fuel which caused more free fatty acids in the biodiesel molecule to be generated within the system and therefore, increased the rate or extent of attack on the mild steel surface (Adama *et al.,* 2021; Adama 2021; Chandran 2020; Chourasia 2020; Alves *et al.,* 2019). This observation further accentuates the fact that time-lag increased the rate of attack of the mild steel surface by the fatty acid components of the jatropha biodiesel fuel as reported by different researchers (Chandran 2020; Chourasia 2020; Alves *et al.,* 2019). It could also be observed that the mild steel corrodes significantly, which was exhibited by severely degraded microstructures with randomly distributed structures or pits at different investigated periods (Chuck *et al.,* 2010; Fardilah *et al.,* 2022). The randomly distributed and uneven structures could be attributed to the formation of protective corrosion products as a result of physical adsorption on the mild steel surface (Kugelmeier *et al.,* 2021; Onyeachu *et al.,* 2022).

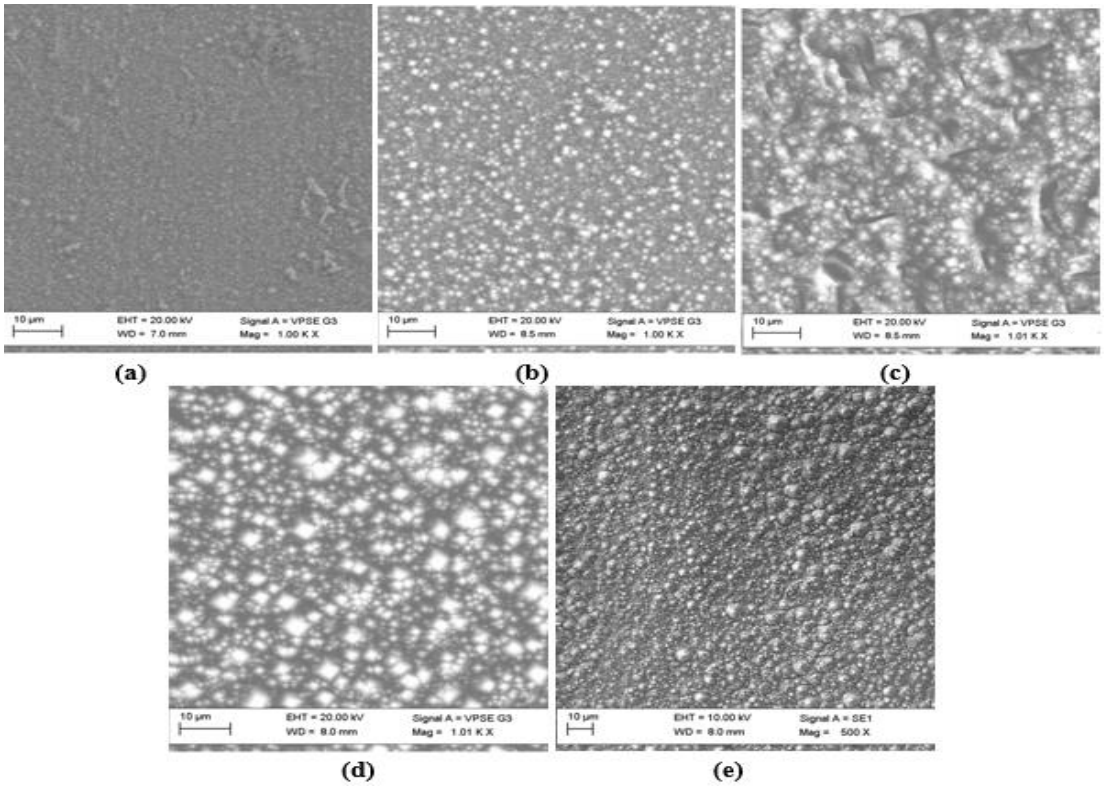


Figure 4: SEM Images of Mild Steel Before and After Immersion in Jatropha Biodiesel Fuel (a) 0 Day (before immersion) (b) 7 Days (c) 28 Days (d) 42 Days (e) 56 Days

Conclusion

The continuous exposure of biodiesel fuel in engines and fuel lines are important areas of investigation as such examinations would enhance the compatibility of various metallic materials toward the use of the non-blended biodiesel fuels. Corrosion of engine parts exposed to biodiesel fuel still remains a major challenge in the automotive industry. In this study, electrochemical and morphological studies of the interaction of mild steel in jatropha biodiesel fuel were investigated. The jatropha biodiesel fuel-mild steel surface interphase interactions were investigated for corrosion degradation during immersion for 0, 7, 28, 42 and 56 days respectively. Surface and solution investigations were conducted using electrochemical studies (OCP, EIS and PDP) and scanning electron microscopic techniques. No chemical reaction occurred between the iron atoms in the mild steel and the organic molecules in the biodiesel fuel indicating a physical adsorption on the steel surface. SEM microstructural investigations confirmed the ability of jatropha biodiesel fuel to corrode the mild steel surface during immersion. The rate or extent of attack on the steel surface by the biodiesel fuel components was time-dependent. This was corroborated by electrochemical investigations carried out. Thus, metal 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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