Effects of Quenching Media on the Corrosion Behaviour of Welded Austenitic Stainless Steel joints in Sulphuric Acid and Simulated Sea Water

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Abstract

The enormous financial loss, product loss and contaminations as a result of weld corrosion have proved to be a serious challenge to the fertilizer, petrochemical and oil and gas industries. The corrosion behaviour of 3mm thick type 304 austenitic stainless steel (ASS) weldments were investigated in the post weld heat treated (PWHT) condition. The as-received ASS was cut into cuboid pieces of 50mm x 20mm x 3mm and butt welded using shielded metal manual arc welding (SMMAW) technique, stress releaved at 600^0C which were later prepared into standard specifications of tests samples before heating them to 910^0C quenched into some selected vegetable (Jatropha and Neem) oils and finally tempered to 300^0C. The physiochemical characteristics and suitability of vegetable oils as alternative quenchants to SAE 40 engine oil for industrial heat treatment were compared. Conventional corrosion coupon (gravimetric) and potentiodynamic polarisation resistance methods of corrosion measurements were evaluated in suphuric acid (0.5M H_2SO_4) and simulated sea water (3.5%wtNaCl) environments. From the results obtained, it was observed that there was a significant improvement in the corrosion behaviour as welded samples quenched in SAE 40 engine oil and jatropha oil showed lower corrosion penetration rate (CPR) of 0.0169mm/yr and 0.0225mm/yr respectively compared to the control sample (0.0338mm/yr) at the active period 72hrs; 0.0032mm/yr and 0.0080mm/yr respectively compared with the control (0.0080mm/yr) during the period of 504hrs when exposed to acidic environment. The samples generally showed very strong passivation at different potentials with time in simulated sea water. Linear polarisation data and curves showed linear relationship between corrosion density (j_{corr}) and corrosion rate; corrosion potential (E_{CORR}) and corrosion resistance respectively, which agreed with the results obtained in weight loss method. It was concluded that vegetable oils such as jatropha and neem oil are suitable alternative quenchants to SAE 40 for industrial heat treatment.

Keywords: ASS, Corrosion, Electrolytes, Passivation, PWHT, Quenchants, Weldment.

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1. INTRODUCTION

The unending search for novel mechanical properties has led to significant development in materials with high strength and long life span. The usefulness of austenitic stainless steel (ASS) in industrial applications and development cannot be over-emphasized. It has found various applications in many engineering industries due to its excellent properties which ranges from high tensile strength, good impact resistance, corrosion and wear resistances.

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This material is used in almost all environments that require an optimization of these properties, some of which are low and high pressure boilers and vessels, fossil-fired power plant, flue gas desulphurization equipment, evaporator tubing, super heater reheating tubing and steam headers and pipes to mention but a few [1].

They are the most easily machinable, weldable and heat treatable of the stainless steel family and can be welded by all welding processes [2]. The microstructure of austenitic stainless steel consists of a mixture of austenite (γ) and delta ferrite (δ) phases [3]. Austenite, which is paramagnetic, has a face centered cubic (fcc) crystal structure, and is the predominant phase in these alloys [3]. The modification of microstructures to effect changes in metallic materials is done chiefly by alloying and heat treatment [4]. Heat treatment enhances the properties of stainless steel such as corrosion

resistance, microstructure, and mechanical strength.

Many research findings have proved that failure of austenitic stainless steel could be weld prone or propagated [5]. Weldments are indispensable parts of most ASS structure fabrication. This welding causes structural changes thereby making the fusion and heat affected zone (HAZ) hard and brittle due to heat generation during welding. Fully hardened steel is not suitable for use directly [6]. It has been discovered that weldments are more susceptible to corrosion attack than those of the parent metals when subjected to unfavourable environments, which lead to the gradual and catastrophic failure of the steel structures [7], [8]. Corrosion is the destruction of a material resulting from exposure and interaction with the environment [9]. Corrosion increases running costs and reduces plant efficiency, availability and product quality but could be prevented easily by using well established techniques [8], [10].

Quenching of steels involves the process of heating a part above upper critical temperature to austenitizing temperature and holding at this temperature for a specified soaking time followed by intense cooling in a suitable quench medium. Quenching prevents the formation of ferrite or pearlite and allows the formation of bainite or martensite. Quench hardening is one of the most important processes of heat treatment that can improve the performance of steel greatly. It is also one of the major causes of rejected components and production losses due to cracks and distortion in the material [11]. It has been discovered that poor quenching processes give birth to microstructural deficiency which will in turn affect the mechanical properties of steels [11]. Quenchants are been extensively used for the control of corrosion in weldments. Conventionally, water and used-SAE 40 are employed as relieve in welded joints but were found to be inducing cracks or dimensional changes on the quenched component due to their high cooling rate [12]. Oil extracts from plants such as *Jatropha and Neem* are available as alternatives. These oils are cheaper, non-edible, readily available, safer, renewable, biodegradable, ecofriendly and less pollutant when compared with the conventional mineral oils [9].

Therefore, the technical challenge of quenching is to select the quenching medium and process that will minimize the various stresses that develop within the part so as to reduce cracking and distortion.

Many techniques are been employed to study the corrosion behavior of metals. Conventionally, the weight loss (gravimetric) method is used but electrochemical techniques are also available. Electrochemical techniques of corrosion measurement are currently experiencing increasing popularity among corrosion engineers, due primarily to the rapidity with which these measurements can be made. Long term corrosion studies, such as weight loss determinations may take days or weeks to complete, while an electrochemical experiment will require, at most, several hours. The speed and accuracy of electrochemical measurements are especially useful for those metals or alloys that are highly resistant such as stainless steels [13]. The potential oils from plant extracts as quenching media for the protection of metal weldments are not thoroughly investigated and are not well understood. Therefore, the objective of this work is to explore the suitability of some vegetable oils such as Jatropha and Neem as alternative quenchants to the conventional used-SAE 40 engine oil on the corrosion protection of Austenitic Stainless Steel joints in sulphuric acid and simulated sea water.

2. MATERIALS AND METHOD

2.1 Materials

Austenitic stainless steel (ASS) and welding electrodes were sourced locally at Kakuri market, Kaduna state, Nigeria. Welding was performed direct current electrode positive (Table 1). Jatropha oil, Neem oil, used-SAE 40 engine oil with physiochemical composition (Table 2) determined at National Research Institute for Chemical and Technology (NARICT) Zaria-Nigeria., Tetraoxosulphate (vi) acid., Sodium chloride.

2.2 Methodology

2.2.1 Welding Procedure

ASS was Butt welded with the aid of shielded manual metal arc welding process. Sixty four samples were cleaned of dirt and oil and a

grinding machine was used to grind the surfaces in order to obtain smooth and uniform surfaces according to [6].

Plate 1: Jatropha plant with seed

Plate 2: Neem plant with seed

Table 1: Welding parameters

Electrode type	Electrode diameter	Polarity	Current	Voltage
E6013	3.2mm	DCEP	60A	400V

Table 2: Physiochemical properties of quenchants

Quenchant	Physiochemical properties		
	Viscosity (cSt) at 27.6 and 60 rpm	Flash point ()	Specific gravity
Neem oil	45.8	244	0.907
Jatropha oil	44.5	225	0.980
SAE 40 oil	46.3	260	0.868

2.2.2 Post Weld Heat Treatment

Two post weld heat treatments were adopted [18] viz:

i. Stress relieve annealing

This treatment carried out by heating the samples from ambient temperature up to 600^{0}C and then soaked at this temperature for 30 minutes after which they were removed from the furnace and allowed to cool in air.

ii. Quenching and Tempering

The previously stress relieved samples were again heated to the austenite temperature 910^{0}C, soaked at this temperature for 30 minutes. They were removed and plunged into the cans that contain the Jatropha oil, Neem oil and SAE 40 engine oil at ambient temperature. These quenched samples were reheated to 300^{0}C and allowed to soak for 30 minutes, after which they were removed from the furnace and allowed to cool in air.

Prior to corrosion test, these samples were cleaned of dirt and oils, grinded with the aid of a grinding machine to obtain smooth and uniform surfaces.

2.3 Preparation of 0.5M H_2SO_4

0.5Molar from concentrated H_2SO_4 of specific gravity = p = 1.82, %Purity = d = 98%, molar mass=98g, was prepared as follows:

The molar concentration of acid:

$$\text{Molarity} = \frac{10*p*d}{molar\ mass} \quad (2.1)$$

$$= \frac{10*1.82*98}{98} = 18.20\text{M}$$

The amount of H_2SO_4 required to prepare 0.5M H_2SO_4 in 1000cm^3 using serial dilution method can be expressed as follows:

$$C_1V_1 = C_2V_2 \quad (2.2)$$

where C_1 = original concentration; V_1 = volume required from original solution; C_2 = concentration required, V_2 = volume of new concentration required.

Therefore, C_1 = 18.20M; V_1 = ?; C_2 = 0.5M; V_2 = 1000cm^3

$V_1 = \frac{C2V2}{C1} = \frac{0.5*1000}{18.20}$ =27.473cm^3 = 27.50cm^3

Then volume of water = $V_2 - V_1$ = 1000cm^3 – 27.50cm^3 = 972.50cm^3

Therefore, 0.5M H_2SO_4 solution was prepared by adding 27.50cm^3 of concentrated H_2SO_4 in 972.50cm^3 of distilled water [14].

2.4 Preparation of simulated sea water (3.5%wt NaCl)

Sea water is often described as a 3.5%wt NaCl and a lot of simulations of corrosion of stainless steel in sea water are carried out in a solution of 35g/L NaCl in distilled water [8]. Therefore, simulated

sea water was prepared by adding 35g of Sodium Chloride (NaCl) to 1000cm^3 of distilled water in a conical flask and then mixing thoroughly to ensure complete solubility and homogeneity according to the method of [8].

2.5 Corrosion test

Prior to the corrosion tests; two methods were adopted in the research which includes:

i. Corrosion coupons (weight loss) method
ii. Linear polarisation resistance method

2.5.1 Corrosion coupons (gravimetric) Method

The initial weights of both the heat treated and untreated (control) coupons were measured with the aid of an electrical analytical weighing balance. Seven each of the coupons were tied with the aid of a thread and suspended simultaneously in the beakers containing the electrolytes (0.5M H_2SO_4 acid solution and simulated sea water) and labeled as J1, J2,…, J7; N1, N2,…, N7; E1, E2,…,E7 and C1, C2,…., C7 denoted as coupons quenched in Jatropha oil; Neem seed oil; Engine oil and Control respectively were tested in sulphuric acid (H_2SO_4) solution and then, J8, J9,…, J14; N8, N9,…, N14; E8, E9,…, E14 and C8, C9,…, C14 were tested in simulated sea water.

Four coupons each (J1, N1, E1 and C1) from H_2SO_4 solution and (J8, N8, E8 and C8) from simulated sea water were removed after a period of 72hours (three days). These tested coupons were washed using a brush, rinsed with water, cleaned with a towel, dried and reweight to determine the final weight so as to ascertain the weight loss and recorded using equation 2.3 above. This procedure was followed after another 72hours until all the coupons were tested. In each case, the corrosion penetration rate CPR was computed using equation 2.4 below according to the method of [7].

$$\Delta W = W_i - W_f \quad (2.3)$$

ΔW is the mass loss (g)
W_i is the initial mass of the coupon (g)
W_f is the final mass of the coupon (g)

Where; ΔW, ρ, A, and t are specified in units of milligrams, grams per cubic centimeter, square centimeter, and hours, respectively. For stainless steel, ρ = 8.0g/cm^3, K= constant whose magnitude depends on the system of units used. It is 87.6 for CPR in mm/yr [7], [8].

Therefore,

$$CPR = \frac{87.6\,\Delta W}{\rho A t} \quad (2.4)$$

The area A of the cuboid shaped welded coupons used for this research was computed using the equation 2.5 where, A = area; L = length = 1.5cm; b = breath = 2.0cm; t = thickness = 0.3cm.

$$A = 2(Lb + Lt + bt) \quad (2.5)$$

A = 8.1cm^2

2.5.2 Potentiodynamic Polarisation (PDP) Method

polarisation studies was performed using a three-electrode cell, with platinum as counter electrode, Ag/AgCl as reference electrode and sample (1 x 1 cm^2 exposed area) as working electrode. Before polarization, each sample was immersed in the selected electrolyte for 30 minutes to allow the potential to reach a steady value. Samples were polarized at a scan rate of 0.001mV/min to +300mV and -300mV with respect to the open circuit potential (OCP). The potential and polarization behavior were recorded using Palm-Sens^3 potentiostat. Polarisation curves were plotted using Auto lab data acquisition system, both the corrosion rate and potential were estimated by the Tafel extrapolation method using both the anodic and cathodic branches of polarisation curves.

3. RESULTS AND DISCUSSION

3.1 Results

3.1.1 Chemical composition of austenitic stainless steel sample

The result of the X-ray florescence (XRF) analysis carried out on the stainless steel sample is summarized in Table 3 below.

Table 3: Chemical composition of Austenitic stainless steel

Elements	C	Ni	Cr	Mo	Fe	Others
%wt	0.06	8.34	20.12	0.188	68.7	2.59

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3.1.2 Gravimetric behaviour of welded ASS coupons in the presence of 0.5M H_2SO_4 and 3.5% NaCl.

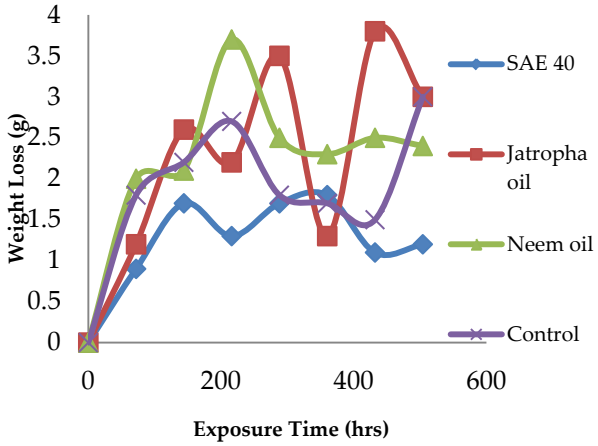

Fig. 1: Weight loss - time responses of untreated (control) and treated welded ASS coupons in the presence of 0.5M H_2SO_4 solution.

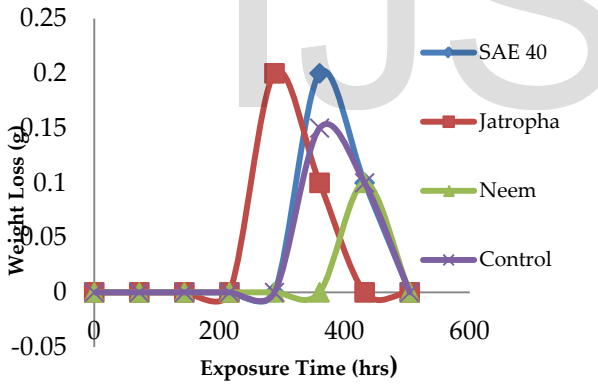

Fig. 2: Weight loss - time responses of untreated (control) and treated welded ASS coupons in the presence of 3.5% NaCl.

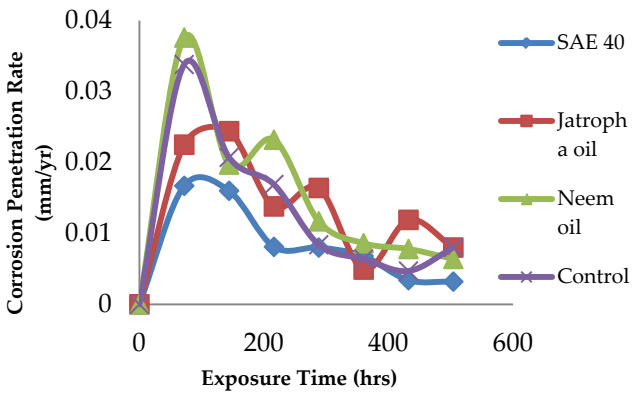

Fig. 3: Corrosion penetration - time responses of untreated (control) and treated welded ASS coupons in the presence of 0.5M H_2SO_4 solution.

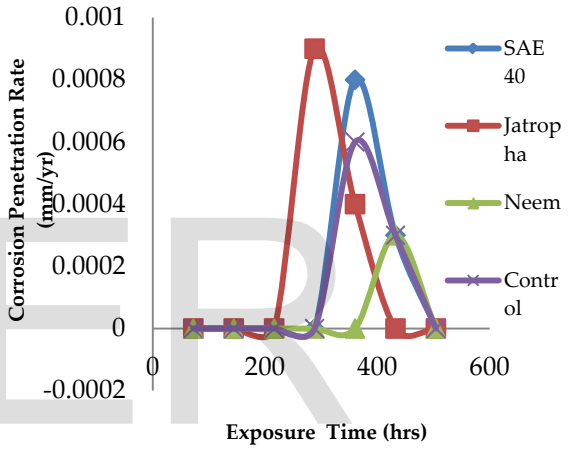

Fig. 4: corrosion penetration - time responses of untreated (control) and treated welded ASS coupons in the presence of 3.5% NaCl.

Figure 1 show the welded untreated control and welded quenched ASS test samples subjected to corrosive attack in sulphuric acid solution; a significant intermittent increase and decrease in the weight loss with time within the period of the experiment was observed. This suggests that the electrolyte or test environment is not safe for the use of type 304 stainless steel welded joints as contained in the recent research of [10].

Figure 3 shows that the samples exposed to sulphuric acid initially experienced preferential corrosive attack at a very high potential (0.035mm/yr for sample quenched in neem oil) corresponding to 72hrs; this could be due to sensitization along grain boundaries in the HAZ or due to high residual stresses induced during welding and quenching which caused the depletion of chromium oxide film as a result of

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precipitation of carbide thereby leaving the localized bare metal to be strongly attacked by the electrolyte [8], [10]. It also depicts the active region of the stainless steel degradation which was due to concentration of sulphate ion (SO_4^-) present in the corrosion reaction. This trend is in conformity with the fact that the degradation of materials in acidic environments has direct consequence on the media concentration as posited by [1], [14], [17]. The samples experienced sharp decrease in the corrosion rate with increase in exposure time (144hrs); this could be attributed to the electro-deposition of an impervious metal oxide film, which is a solid interfacial oxide compound that protects the metal against further oxidation. This trend of corrosion profile clearly depicts that a limit was attained where passivation phenomenon sets in leading to a gradual decrease in corrosion rate as reported by [8], [14].

The corrosion rate of the samples later increase at different potentials with increase in exposure time; this could be due to local compositional variation or heterogeneity after solidification, leading to less stable passive film or presence of impurities that retard the formation of protective film or accelerate its degradation [8]. The corrosion rate finally decreased at about 0.008mm/yr corresponding to 504hrs; this behaviour could be attributed to the fact that there remains no direct contact between metal surface and corrosive ions by corrosion products and excess of cations near the metal surface as the reaction proceeds or as a result of the stifling effect of the corrosive medium by the corrosion deposit which has weakened the acid test environment as contained in the researches of [18], [10]. It can be observed that samples quenched in Jatropha oil and SAE 40 engine oil gave comparatively better performance by recording lower corrosion rate from the beginning to the end of the experiment; this suggested that heat treatment increased their corrosion resistance in the acid medium than the other test samples.

Figure 2 shows that the samples exposed to simulated sea water were very resistant to the test environment at the initial stages of the experiment with zero weight loss when exposed for the period of 288 hours except for the sample treated in Jatropha oil (0.2g). The samples later experienced weight loss at different rates and finally dropped. This suggests that the severity of the electrolyte is less aggressive when compared to acidic media [8]. Figure 4 shows the variation of Corrosion Penetration Rate (CPR) with Exposure time of control and quenched coupons exposed to simulated sea water (3.5% NaCl). There was an observed and recorded zero corrosive attack within the space of 288 hours except for the sample treated in Jatropha oil which recorded about 0.0014mm/yr. This could be attributed to the rapid formation of protective chromium oxide layer on the metal surface which acts as a barrier to restrict the diffusion or precipitation and corrosive attack of Cl^- ions [15].

It has been reported that any compound capable of donating free chloride ions (Cl^-) to an aqueous (water-based) solution has the potential for causing failure in austenitic stainless steels [10]. Therefore, chemical breakdown of the oxide films was due to concentration of Cl^- anions which seems to decrease the pitting potential in the weld zone. In welds, internal stresses in local areas may be produced, causing the migration of Cl^- anions to these areas. This further caused localized attack by the aggressive electrolyte made as a result of concentration of Cl^- as posited by [16].

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3.1.3 Potentiodynamic polarisation behavior of ASS in the presence of 0.5M H_2SO_4 and 3.5%NaCl.

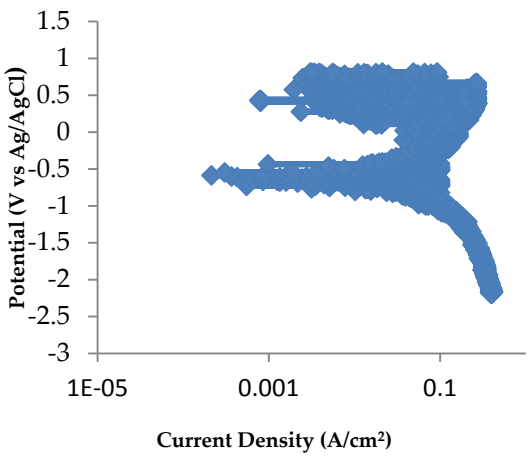

Fig. 5: Potentiodynamic polarisation curve of welded untreated (control) ASS in the presence of 0.5M H_2SO_4 solution.

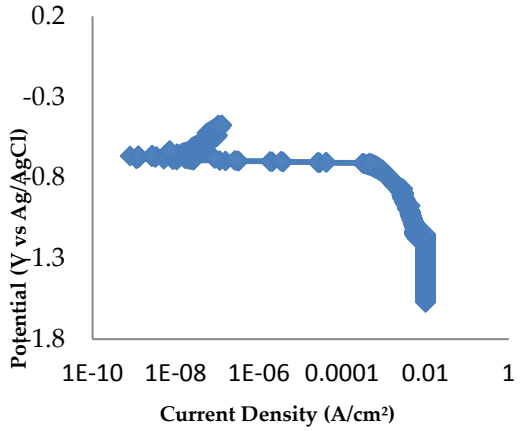

Fig. 7: Potentiodynamic polarisation curve of welded ASS treated in Neem oil in the presence of 0.5M H_2SO_4 solution.

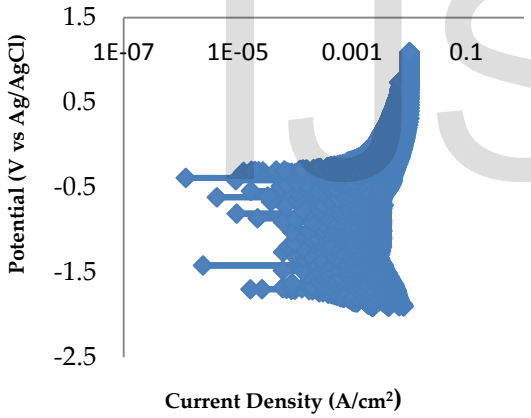

Fig. 6: Potentiodynamic polarisation curve of welded untreated (control) ASS in the presence of 3.5% NaCl.

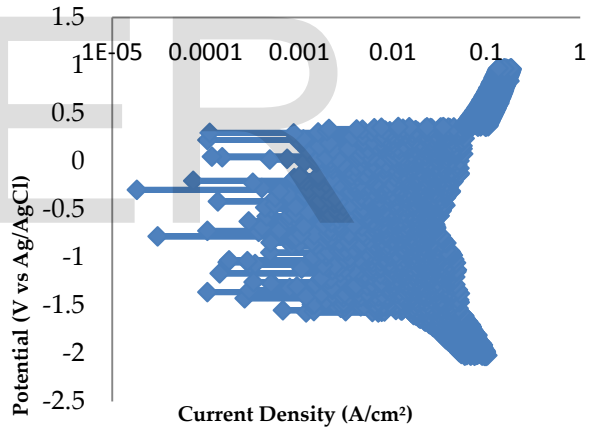

Fig. 8: Potentiodynamic polarisation curve of welded ASS treated in Neem oil in the presence of 3.5% NaCl.

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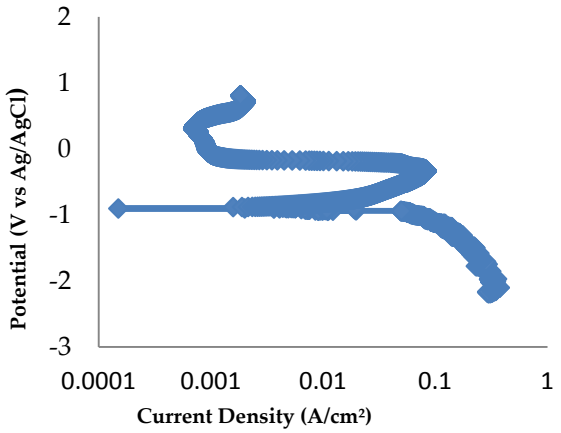

Fig. 9: Potentiodynamic polarisation curve of welded ASS treated in Jatropha oil in the presence of 0.5M H_2SO_4 solution.

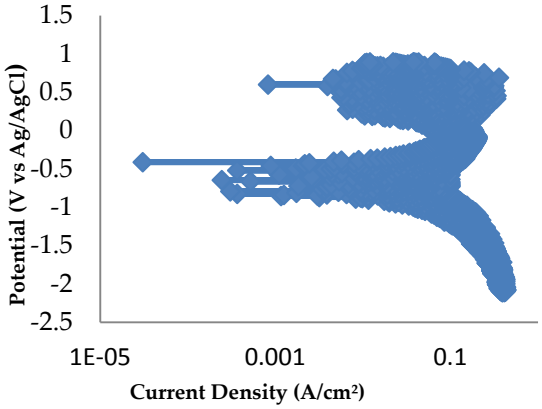

Fig. 11: Potentiodynamic polarisation curve of welded ASS treated in SAE 40 in the presence of 0.5M H_2SO_4 solution.

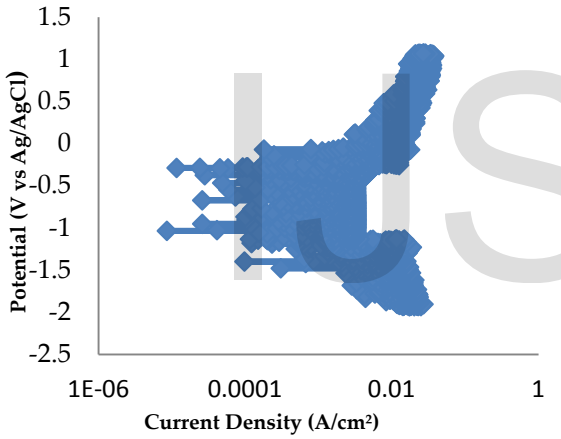

Fig. 10: Potentiodynamic polarisation curve of welded ASS treated in Jatropha oil in the presence of 3.5% NaCl.

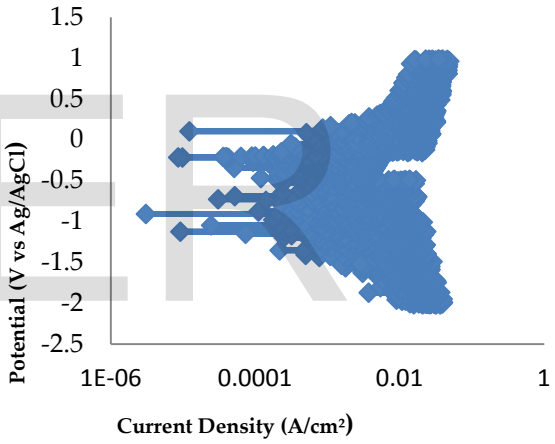

Fig. 12: Potentiodynamic polarisation curve of welded ASS treated in SAE 40 in the presence of 3.5% NaCl.

Figures 5, 7, 9 and 11 showed Evans diagram of welded untreated and treated ASS and welded quenched electrodes exposed to sulphuric acid (0.5M H_2SO_4) solution. The scanning curve of all the samples studied showed that both cathodic (passive region) and anodic (active region) corrosion reactions have occurred. At corrosion potential E_{CORR}, the point where cathodic and anodic curves intersect, the rate of hydrogen evolution is equal to the rate of metal dissolution, which means corrosion has not occurred at that point. The samples showed a relatively smaller

passive region (cathodic) and a prolonged active region (anodic), which differs, based on the quenching media used during heat treatment; this indicated high corrosion susceptibility and that the treatment has no significant effect on the weldment [19].

Figures 6, 8, 10 and 12 showed Evans diagram of welded untreated and treated ASS in the presence of simulated sea water (3.5%NaCl). It also indicated insignificant protection by the heat treatment. The size of the pitting loop seen is an indication of pitting tendency; the larger the loop, the greater the tendency to pit as contained in the research of [15].

Conclusions

1. Quenching in vegetable oils was found to provide comparable corrosion protection on the ASS weldment. However, in terms of severity of protection, jatropha oil was found to offer better resistance when exposed to acidic environment whereas neem oil performed in NaCl environment.
2. The removal of chromium carbide as a result of heat supplied during welding lead to high corrosion susceptibility in the weldment.
3. Severity of corrosion attack was found to higher in acidic environment than NaCl solution.
4. Investigation into the mechanism to which corrosion protection offered by oils from plant extracts can be conducted.

Acknowledgement

The authors wish to thank the entire staffs of The Department of Mechanical Engineering, Ahmadu Bello University, Zaria-Nigeria for their kind support.

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