Electrochemical Behaviour of Mild Steel in Some Agro Fluids

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Abstract—**The corrosion behaviour of mild steel in some selected agro fluids such as apple, mango, grape, orange and the mixture of these agro fluids was electrochemically studied. A potentiometric analysis was employed to establish the comparative corrosion rates of this material in the agro fluids over the interval of five days for a sixty-day immersion period at a constant ambient temperature of 27±ºC. The chemical composition of both mild steel and the agro fluids was determined to ascertain corrosion mechanism for the reaction. Polarization behaviours of mild steel in the agro fluids were determined by Tafel extrapolation curves. The analysis showed that cathodic polarisation curves were almost identical irrespective of variation in concentration of the various fluids while the anodic polarization curves exhibited varying active and passive corrosion behaviour due to passivating oxide films. The analysis of the results further showed that corrosion rates of the metallic alloy decreased with increase in immersion period which could be attributed to gradual decline in the concentration of the acidic level in these fluids within the given range of potential and scan rate. Hence, the evolution of hydrogen gas and reduction of oxygen molecules from the reacting system were presumed to be major factors retarding corrosion of the solution involved. Microscopic analysis of the corroded mild steel was revealed by SEM and EDS showed the respective compositions of the mild steel after the electrochemical tests. The result obtained from the study showed that electrochemical corrosion rate over the duration of immersion has greatest effect on the metal studied in orange medium (1.530 mm/yr), followed by grape medium (1.403 mm/yr), mango medium (1.339 mm/yr) while the metal in apple medium experienced the least effect (1.301 mm/yr). However, the mixture of these agro fluids showed more corrosive effect than the individual agro fluids with corrosion rate of 1.672 mm/yr.**

Keywords: *agro fluid, mild steel, corrosion rate, exposure time, electrochemical behaviour.*

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I. INTRODUCTION

Corrosion exhibits significant effects on materials. It reduces the safe, consistent and effective equipment operations and structures which eventually lead to the loss of the affected material object [1], [2]. Metals form the essential bases for modern technological civilization. One of such areas is in the agro industry, where metallic alloys are widely used in the industrial processing and packaging of fruit juices. Most metallic corrosion result from electrochemical effects exhibited by these juices on the metals. Shreir *et al.* [3] described electrochemical corrosion as a heterogeneous redox reaction at metallic/non-metallic interface in which the metal is oxidized and the non-metal is reduced. When this occurs, corrosion is initiated by the flow of electrons between the electrode sites of different potentials in contact with aqueous electrolytic solution [4], [5].

Studies showed that metals can corrode when exposed to the atmosphere as well as in acidic solutions [6], [7]. According to these researchers, corrosion involves the transfer of electrons along the surface of the metal under the influence of a potential difference. Sharma and Sharma [8], observed that metallic alloys do not corrode in dry air or in water completely free of air but requires oxygen and water to occur. Corrosion is accelerated by acids or by contact with less active metals such as copper or lead. Certain salt solutions also accelerate corrosion, not only because they are acidic by hydrolysis, but also because of specific catalytic effects or reactions of the anions. Therefore, there is effective collision of particles which affect corrosion rate [9].

According to Costescu *et al.* [10], fruit juices are liquid, non alcoholic products with different degrees of clarity and viscosity, obtained through pressing or breaking up of fruits with or without sugar or carbon dioxide addition. Agro fruits exhibit high level of carboxylic acidity which would have corrosive effect on metals at different rates. Organic acids directly play an important role in the growth, maturation and acidity of the fruit, and also affect the shelf life of the fruit by influencing the growth of microorganisms [11], [12]. Organic acids such as citric, malic, oxalic and tartaric acids ranging from 0.1 to 30 g/L were found in orange, grape, mango and applejuices. However, there was a considerable difference in the organic acid content found in various types and brands of fruit juice [13]. According to Toaldo *et al.* [14], analysis of grape juices from *Vitis Labrusca L.* showed it contains significant amount of gallic acid, in addition to phenolics, monomeric anthocyanins and antioxidant from its seeds.

Apple concentrate was found to have higher amount of malic acid than other carboxylic acids [11]. Brae burn apples contained the highest amount of citric acid in apples; however, Granny Smith apples were the overall most acidic apples tested. High pressure liquid chromatography (HPLC) has been studied to be very efficient chromatographic technique in determining chemical composition of organic acids in agro juices although absolute precaution is required [12]-[13], [15].

Environmental factors like oxygen concentration in water or atmosphere, the pH of the electrolyte, temperature, concentration of various salts solutions, and many more in contact with the metal play a significant role in the rate of corrosion of metals even if such metallic materials are completely homogeneous in nature. Meanwhile, hydrogen evolution from an acidic environment is responsible for the sustenance of corrosion of metal [16]. Higher concentration of a solution will cause more hydrogen gas evolution. The stability of the halide in the surface complex determines the effect of corrosion kinetics of the metal/alloy. According to Marcus and Maurice [17], metal (M) corroding to M^{2+} ions at the anode in the presence of water would be reduced to hydroxyl ions and hydrogen at the cathode. The hydroxyl is readily oxidized by air to a hydrated compound during the corrosion process.

Metallographic examination is one of the most procedures used in failure analyses of metals. It involves microstructural inspection of corroded metallic materials or their alloys to ascertain the extent of corrosion [18]. Highly precision electron metallographic equipment, such as the scanning electron microscope (SEM), transmission electron microscope (TEM), energy disperse spectroscopy (EDS) and X-ray diffraction (XRD) are used for such analyses [18]. SEM is very useful to show surface morphology and it is widely applied in material science [19].

This work examines significantly the electrochemical corrosion behaviour of mild steel in orange, mango, grape and apple juices to determine the various rates of corrosion under given range of physical conditions; likewise to compare the corrosion mechanisms of the corroded mild steel samples in these agro fluids.

II. MATERIALS AND METHOD

The commercial mild steel used for the electrochemical corrosion studies was supplied by ArcelorMittal South Africa and the percentage chemical composition of the mild steel is presented in Table 1.

TABLE I
CHEMICAL COMPOSITION OF MILD STEEL

Thickness (t) (mm)	Fe (Max.)	C (Max.)	Mn (Max.)	P (Max.)	S (Max.)	Si (Max.)
t<4.5	98.48	0.15	1.00	0.035	0.035	0.30

Square-base mild steel test specimens (10mm x 5mm thickness) were machined from Buehler IsoMet 4000 (USA) linear precision metal cutting machine and mounted in cold-curing polyester resin to reveal flat surfaces in contact with the corrosion media. The terminals of the test specimens were linked by insulated stripes of copper wire. Mild steel specimens were abraded using 220, 600 and 1200 grit emery papers mounted on IMPTECH (20 PDVT) grinding and polishing machine at average speed/force of 300 rpm/30N over the duration of 4 minutes according to Advanced Laboratory specifications. They were later polished with diamond abrasive pastes of 3 micron, 1 micron and 50/nm grit sizes at average speed 150/rpm force of 25 N for 3/minutes.
The mild steel samples were then degreased with acetone rinsed with distilled water and dried at ambient temperature. Polarization curves at various immersion time were measured by the open circuit potential (OCP) and recorded potentiodynamically with scan rate (potential sweep) of 0.002 (V/s). Corrosion potential measurement commenced from -1.0 volts and ended at 2.0 volts. Corrosion current densities (I_{corr}) and corrosion potential (E_{corr}) were evaluated from the intersection of linear anodic and cathodic branches of the polarization curves in accordance to Tafel extrapolation method adopted by Poorsqasemi *et al*. [20] investigation. The pH values of the various agro juices were also taken before and after each exposure time using a standard portable MBI model 3D (Montreal, Canada) pH meter. Other corrosion parameters such as anodic and cathodic Tafel slopes analyses and evaluation of corrosion properties based on ASTM 59, 96 and 159 standards were also considered from the polarization curves by Tafel extrapolation.

Corrosion media investigated include fruit species of freshly harvested orange (*citussinensis*), mango (c*hok Anan*), grape (*vitisvinifera L.*), apple (*delicious*) juices and their mixture. The various agro fluid samples were prepared by extracting the juices from fresh harvested fruits using commercial blender and later kept in a refrigerator at 0°C. Organic acids are mostly responsible for corrosive effects of agro juices [21], [22]. However, there is also minute presence of phenolic content, fatty acids and amino acids in agro fluids [11]. Because of variation in the concentration and composition of organic acid in different agro fluids, it became necessary to determine the chemical composition of these acids in each medium.

Analysis of the organic acid content in the agro fluids was studied using high pressure liquid chromatography (HPLC) method. The organic acids were identified and quantified with the aid of ultra violet (UV) detector with a wavelength of 250 nm attached to a model K-2502 KNAUER equipment by comparing their retention times and peak heights with standard organic acid solutions. Potentials of hydrogen (pH) values of the agro fluids were recorded before and after the exposure time to establish differences in their acidic level. Table 2 shows the analysis of the agro fluids used for the study.

TABLE II
CHEMICAL COMPOSITION OF AGRO JUICES

Apple (*delicious*) (g/l)		Grape (*vitisvinifera*) (g/l)		Mango (*chokAnan*) (g/l)		Orange (*citussinensis*) (g/l)	
Citric	0.064	Citric	0.072	Citric	2.940	Ascorbic	0.652
Malic	2.840	Malic	3.500	Malic	7.520	Citric	14.012
Shikimic	0.021	Succinic	0.002	Tartari	0.980	Malic	1.525
Succinic	0.210	Tartaric	7.140	Succinic	2.090	Lactic	1.913
Tartaric	0.019					Tartaric	0.382
Quinic	0.611					Oxalic	0.105

Microscopic analysis of the electrochemical corrosion results was performed on the corroded metallic samples. Comparisons were drawn from surface morphologies to determine the extent of corrosion on the metallic alloy in the various agro fluids. The techniques used to evaluate corrosion products after electrochemical studies were scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS). SEM analysis of the steel specimens was examined on the corroded surfaces considering the surface exposed to the air side. The SEM reading was taken at magnification of 1500X/20μm to expose wider corroded region with more distinguishable characteristics of the metal. The spectrum processor of the EDS was set to depict distinction in the composition of the surface elements.

III. RESULTS AND DISCUSSION

Tafel slope analysis presented in Fig. 1 shows relative polarization behaviour of mild steel in various agro fluids. It is observed from the nature of various curves that the concentration of the environments differs completely. Fig. 1 reveals that all the agro fluids have insignificant passivation of their oxide layers on the 60th day.

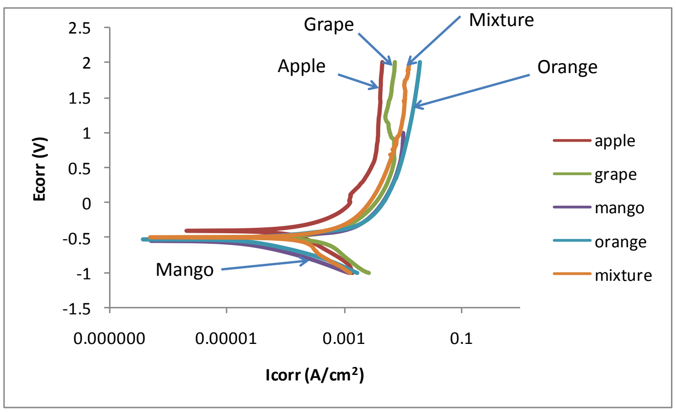

Fig. 1 Polarization curves of mild steel specimens on 60th day immersion period

Comparison of electrochemical corrosion parameters (Table 3) shows that E_{corr} of the corroded mild steel is highest in apple juice (-402 mV) while mango has the least in the value of -530 mV. The highest I_{corr} emerge from mango medium (6.667 A/cm^2) as observed from the data. Grape juice exhibits the least I_{corr} value of 1.034 A/cm^2. The maximum anodic Tafel slope (ba) is exhibited in apple juice (0.169 V dec^{-1}) while the least is shown in grape juice (0.115 Vdec^{-1}). Apple medium maintains highest cathodic Tafel slope of 0.153 Vdec^{-1} while orange medium exhibits the least (0.062 Vdec^{-1}).

Corrosion rates (CR) of the mild steel specimens in the various agro fluids decreased over the duration of the study as seen from Table 4. According to Tran *et al*. [23], organic acids enhance corrosion rates of mild steel samples by accelerating cathodic reaction either through direct reduction at the metal surface or by means of buffering effect which involves dissociation of the hydrogen ions near the corroding surface.

TABLE III
COMPARISON OF ELECTROCHEMICAL CORROSION PARAMETERS ON THE 60TH DAY OF IMMERSION

Electrolyte	Parameter			
	ba (V dec^{-1})	bc (V dec^{-1})	I_{corr} (μAcm^{-2})	E_{corr}(mV)
Apple	0.169	0.153	5.969	-402
Grape	0.115	0.103	1.034	-499
Mango	0.123	0.065	6.667	-530
Orange	0.157	0.062	1.460	-515
Mixture	0.118	0.084	5.624	-490

TABLE IV
ELECTROCHEMICAL CORROSION RATES OF MILD STEEL IN THE AGRO FLUIDS

Duration (days)	Corrosion Rate: CR (mm/year)				
	Apple	Grape	Mango	Orange	Mixture
5	6.946	9.684	7.759	8.056	9.224
10	5.435	7.278	7.705	7.435	7.571
15	3.131	5.809	6.750	7.014	7.430
20	2.447	5.068	6.080	5.154	7.296
25	2.275	3.632	5.706	4.562	6.545
30	2.069	3.469	4.215	2.972	6.031
35	1.825	2.275	3.063	2.352	5.944
40	1.741	1.933	2.387	2.347	3.578
45	1.567	1.752	2.305	1.700	3.090
50	1.383	1.560	1.669	1.593	2.307
55	1.344	1.451	1.356	1.558	1.899
60	1.301	1.403	1.339	1.530	1.672

The highest electrochemical corrosion rate was obtained from the mixture medium. It maintained the lead from 15th day (7.430 mm/yr) till 60th day with 1.672 mm/yr corrosion rate. Hence, it is possible that orange juice contains more acidic ions with lower pH value than mango, apple and grape lately. This of course may have great impact in the dissolution of passivating oxide layers on the metallic sample leading to more rapid corrosion rate. The mixture of these fluids however, has more profound corrosion rate than any of the individual agro fluids as noted. Possibly presence of more acidic group in the fluid may have given rise to greater corrosive attack on the metallic specimen than other agro juices. There was significant decline in the potential of hydrogen (pH) values in the various corrosion fluids on the 60th day of immersion as indicated: apple 5.82, grape 5.45, mango 5.60, orange 5.31, and mixture 5.08, respectively.

Fig. 2(a)-(e) shows the microscopic structural examination of the corroded mild steel specimens in the different agro fluids taken after 60th day exposure period at approximately 1500X magnification and distance of 20μm.

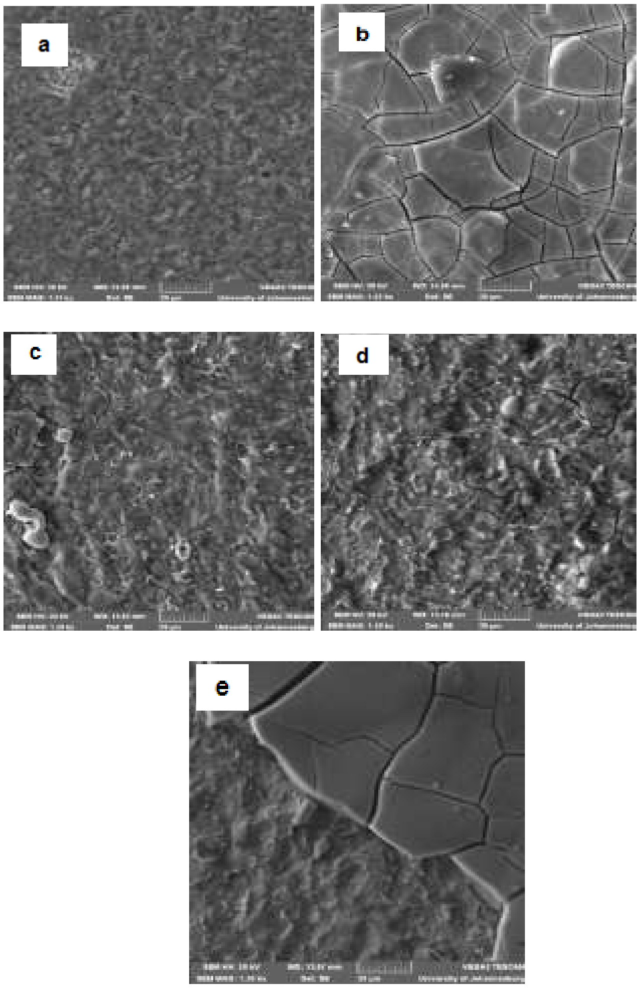

Fig. 2 Comparison of SEM surface morphologies of mild steel specimens immersed in (a) apple (b) grape (c) mango (d) orange (e) mixture juices at 20µm

SEM surface micrographs of the corroded samples indeed clearly depict varying degrees of oxide scales on the metallic samples. Corrosion mechanism and formation of crystal morphology on the metal specimens depend on corrosion products formed within the period of immersion [18], [24]. The various SEM surface morphologies of the corroded mild steel specimens show different degrees or sizes of passivating oxide films deposition. Intense deposited oxide layers were formed on the surfaces of mild steel specimens immersed in grape juice (Fig. 2b) and that of mixture juice (Fig. 2e), while specimen from mango juice (Fig. 2c) shows different structure of oxide formation across the surface. The micrograph of metal sample immersed in orange medium (Fig. 2d) shows multiple film scales due to the nature of deposited oxide layers. The identified signs of rupture that occurred on the deposited oxides scales (Fig. 2d) may arise because of greater corrosiveness of the orange environment. Smaller sizes of oxide films with closed structures are observed across the surface of samples from apple (Fig. 2a) and mango juices (Fig. 2d) environments. The oxide film in Fig. 2(a) is much finer than Fig. 2(d), possibly indicating better wear resistant to corrosion in apple juice than in mango juice.

The elemental characterization of the corroded mild steel specimens in the agro juices after 60 days is viewed by energy dispersive X-ray spectroscopy (EDS). The results are presented in Fig. 3 (a)-(e).

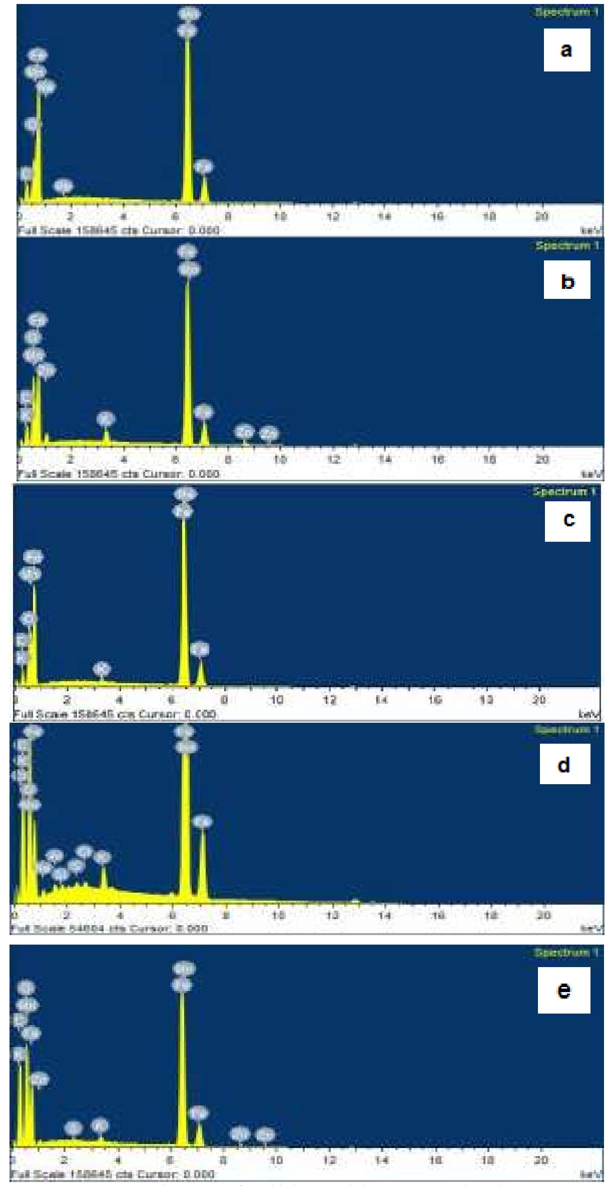

Fig. 3 EDS analyses of mild steel in (a) apple (b) grape (c) mango (d) orange (e) mixture

EDS spectra in Fig. 3(a)-(e) show the various elemental composition shown on the surfaces of corroded mild steel specimens after sixty days of immersion period. From the results, oxygen constitutes one of the major elements present in all the corroded samples. Oxygen may have been the main oxidizing agent in the agro juices environments which induced corrosive effect on the metallic specimens from the perspective of the study as supported by Porcayo-Calderon *et al*. [25]. The oxide content of the surface analysis, suggests that percentage weight of oxygen composition on the corroded specimen is proportional to the corrosion rate (CR) of the metallic alloy (Table 5).

TABLE V
CORRELATION OF CR AND % WEIGHT OF OXYGEN

Corrosion medium	mixture	orange	grape	mango	apple
% Weight of oxygen	24.29	20.01	19.47	14.44	11.25
CR (mm/yr)	1.672	1.530	1.403	1.339	1.301

Hence the amount of oxygen deposited on each corroded specimens differs almost entirely. EDS analysis shows that percent weight composition of oxygen on the metallic surface from mixture medium is greatest (24.29%), followed by metal sample immersed in orange juice with 20.01% of oxygen deposit (Fig. 3d). Corroded metals in grape medium (Fig. 3b) and mango (Fig. 3c) contain 19.11% and 14.44% weights of oxygen respectively. The least oxide deposit is observed on metal specimen in apple juice medium (Fig. 3a) with 11.25% oxygen deposit.

IV. CONCLUSION

Accelerated electrochemical corrosion behaviour of mild steel in some selected agro fluids has been demonstrated. Corrosion rate (CR) of the mild steel in the agro fluids decreased progressively over the entire duration of the study. This was associated with greater formation of passivating oxide films. Reduction of hydrogen ions from the acid media and dissolved oxygen gas were also responsible for the decline in the rate of corrosion. Corrosion rate of the mild steel has greatest impact in mixture medium (1.672 mm/year). But on individual basis orange juice corroded most (1.530 mm/year) followed by grape medium (1.403 mm/year), mango medium (1.339 mm/year) and apple medium (1.301 mm/year), respectively at the last day of the immersion.

The significant increase in the pH values of the various agro fluids was a confirmation of reduction in the acidity of all the test environments due to near or complete evolution of dissociable hydrogen gas from the corrosion media. The average pH values of various juices on the 60th day of immersion confirmed orange juice might have exhibited highest corrosive effect than other individual agro fluids as a result of the additional acids present in the orange. Also, it can be associated with the greater amount of dissociable hydrogen ion in citric acid more than in malic and tartaric acids except for mixture juice which might have combined corrosive effect and subsequently corroding at greatest rate while apple juice had least corrosive effect. The result of microscopic analysis was proportionate to that of corrosion rates which showed that corrosion rate of the mild steel is greatest in mixture medium followed by orange juice, grape juice and mango juice while apple juice had the least corrosion rate.

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