

CORROSIVE BEHAVIOR OF Al, Cu and MS IN DIFFERENT ACIDIC MEDIA

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Abstract: The corrosive behavior of some metals was studied in varying strength of two different acids by employing weight loss method. The experimentation was carried out at room temperature. The effect on corrosion behavior of metals with the change in acidic strength as well as the nature of metal was monitored at different immersion periods. The corrosion parameters were determined with the experimental data. On analysis, at room temperature, HCl (compared to H₃PO₄) was found to be more aggressive towards aluminium as well as mild steel (in comparison to copper). The kinetic parameters were evaluated and discussed. The surface analysis also carried out with the help of Scanning electron microscope (SEM), Energy dispersive X-ray (EDX), Optical microscope (OM) and X-ray fluorescence (XRF). Overall the result suggest that corrosion was observed significantly less in H₃PO₄ acidic medium as compare to HCl medium.

Keywords: Metal, Acid corrosion, Corrosion parameters, Kinetic parameters, SEM, EDX, OM, XRF.

I. INTRODUCTION

Corrosion is a prevailing destructive phenomenon in science and technology. As per literature survey, the cost due to corrosion in many countries is as high as 3-5 % of the GNP i.e. wasteful in terms of economy of any country [1-3]. This represents a huge amount of money which should have been channeled into the provision of basic social amenities in these countries. In practice corrosion can never be stopped but can be hindered to a reasonable level by analyzing of behavior of metals in different environments and by applying various inhibition methods according to their nature. Shaw Barber & A. Kelly [4] and R. R. Pierre [5] determined that metals are usually exposed to the action of bases or acids in the industries in a variety of practical applications such as acid pickling, industrial acid cleaning, cleaning of oil refinery equipment, oil well acidizing and acid descaling. The exposures can be severe to the properties of the metals as well as age of metals also, thus lead to ritual failure of materials in service.

A. Objectives

There is need to study the corrosion behavior of metals when exposed to various environments, as this is an important factor in material selection. This work examines the corrosion behaviors of some industrially used metals, such as aluminum, copper and mild steel in hydrochloric acid or phosphoric acid. These metals have been a subject of numerous studies due to their high technological value and wide range of industrial applications.

Metals: Christian Vargel, et al. [6] reported that Aluminum and its alloys, however, are reactive materials and are prone to corrosion. Copper is characterized by its high electrical and thermal conductivities and good mechanical workability [7]. Mild steel is one of the major construction materials, which is extensively used in chemical and allied industries for the handling of acid, alkali and salt solutions [8-9].

Corrosive Media: Hydrochloric acid is the most difficult of the common acids to handle from the standpoints of corrosion. Extreme care is required in the selection of materials to handle the acid by itself, even in relatively dilute concentrations or in process solutions containing appreciable amount of hydrochloric acid. This acid is very corrosive to most of the common metals and alloys. Where Phosphoric acid prepared by hydrates (wet) processes generate severe corrosion problems in containers.

B. Metal dissolution behavior

The corrosion behavior of Aluminium, Copper and Mild Steel and their dissolution mechanism in acidic media, viz., HCl and H₃PO₄ acid at two concentrations (0.5 N-2.0 N) were studied. The experimentations were carried out at room

International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

Vol. 2, Issue 10, October 2013

temperature (303±1K) by using a chemical (Weight loss) method. The morphology of these metals surface before and after immersion in HCl and H₃PO₄ solutions was examined by SEM and optical micrographs at different concentrations of the acid.

The compositions of the metals under study were determined by X-Ray fluorescence spectroscopy and the compositions are tabulated as below:

II. METHODOLOGY

A. Specimen preparation-

Corrosion tests were performed using by chemical measurements. Rectangular coupons of metals of dimensions (3cm× 2.4cm× 0.16cm) were used with a small hole of 0.12mm diameter near upper edge for hanging in test media. Each coupon was cleaned by emery paper of grade 200 to 800 to give a mirror finish and then degreased by double distilled water, then by acetone and dried in desiccators, prior to each experiment each coupon was surface treated [9].

Aluminium		Mild Steel		Copper	
Elements	Wt. %	Elements	Wt. %	Elements	Wt. %
Al	97.60	Fe	98.70	Cu	93.60
Mn	1.30	Mo	0.01	Mn	0.51
Fe	0.87	Mn	0.39	Fe	0.19
Cu	0.11	Cr	0.039	Zn	5.70
Zn	0.11				

B. Test solution-

The solutions 0.5 N and 2.0 N HCl, or H₃PO₄ was prepared by using doubly distilled water. HCl and H₃PO₄ acids used were of analytical grade, having the pKa value 1×10^{-2} for HCl and three pKa values for triprotic phosphoric acid pKa₁= 2.12, pKa₂ = 7.21, and pKa₃= 12.67.

C. Weight loss measurements-

The weight loss experiments were carried out using surface treated rectangular coupons viz. (Aluminium, Copper and Mild steel) having the above given dimensions. In this method the metal coupons were dipped in beakers containing 50 ml of respective corrosive solutions in two different concentrations ranging from 0.5 N to 2.0 N, were made after 72 h of immersion at room (303±1K) temperature. Before measurement, each coupon was surface treated dried in desiccators for 24 h and then weight was taken. From the weight loss method, the weight loss or corrosion rates of metals were calculated by helping of area and density and time periods [3, 10-11]. A is surface area taken in cm², D is density of metal in g/cm³ and T is time period in hours and

$$\Delta W = W_1 - W_2$$

Here W₁ is the initial weight of the metal coupons and W₂ is the weight at a particular immersion time. The procedure similar as reported in our earlier work, ie, by Rekha N. Nair et al. [11] and Alka Sharma et al. [12-13]. The densities for Aluminium, Copper and Mild Steel are 2.7, 8.9 and 7.8 g/cm³ respectively [14].

III RESULTS AND DISCUSSIONS

A. Qualitative Solution Characterization:

The dissolution of Aluminium, Copper and Mild steel were observed in different concentrations of aggressive media (HCl, and H₃PO₄). This was evidenced by the decrease in the original weight of the metal coupons. Experimental results reveal HCl medium to be more corrosive, than H₃PO₄ acid medium. In general the observations depend on the type of material. During initial few hours of the test, in hydrochloric acid coupons began to react vigorously as compare to phosphoric acid. The color of the solution change according to metals dipped in it; solution with Cu coupons appeared light blue which turned dark blue as the immersion time increases. It is because of the formation of cupric chloride (CuCl₂) due to reaction of copper with chloride ions of HCl media.

The aggressive solution containing mild steel coupons gained color (light brown) which later on darkened with increasing immersion period. This change in color is clearly due to dissolution of Fe to form ferric chloride (FeCl₃).

The HCl medium with aluminium coupons does not show any considerable change while in H₃PO₄ medium turbidity appears. At the bottom of the test solution in the MS Coupon of 2.0 N phosphoric acid solutions, a dark and rusty solids in appearance. Literature suggests that the solid product is formed due to Iron phosphate FePO₄ [15 - 16].

International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

Vol. 2, Issue 10, October 2013

B. Weight loss Measurements:

The experimental data obtained by weight loss measurements were evaluated to determine various corrosion parameters, viz., corrosion rate and activation energy. These have been summarized in Tables 1 and 2. These tables (1 and 2) and figures (1-4) represents the variation in HCl acid and H₃PO₄ acid as a function of two different concentration 0.5 N and 2.0 N. All experimental results obtained from measurements are interpreted as described under.

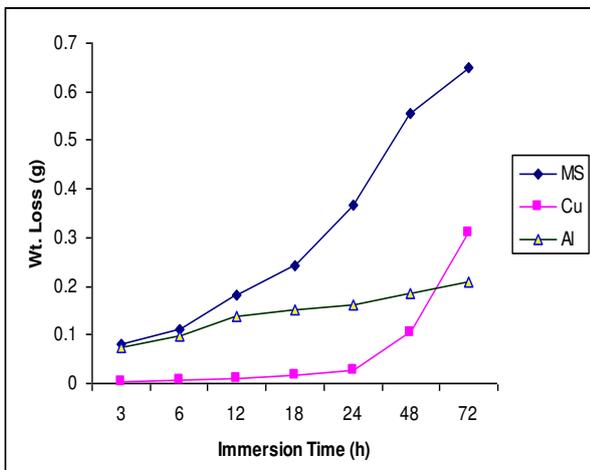


Fig 1: Weight Loss Vs time for Al, Cu, and MS in 0.5 N HCl

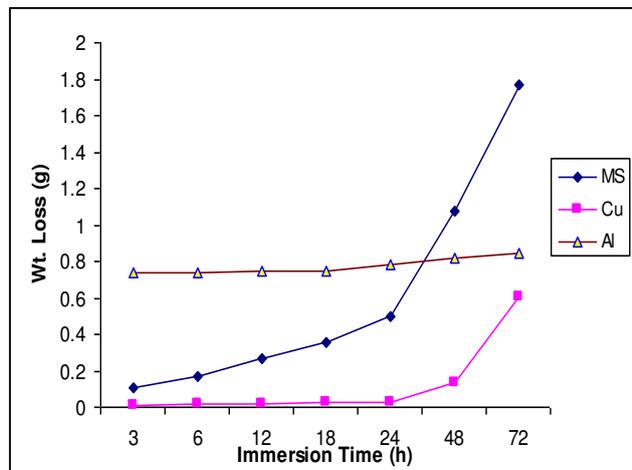


Fig 2: Weight Loss Vs time for Al, Cu, and MS in 2.0 N HCl

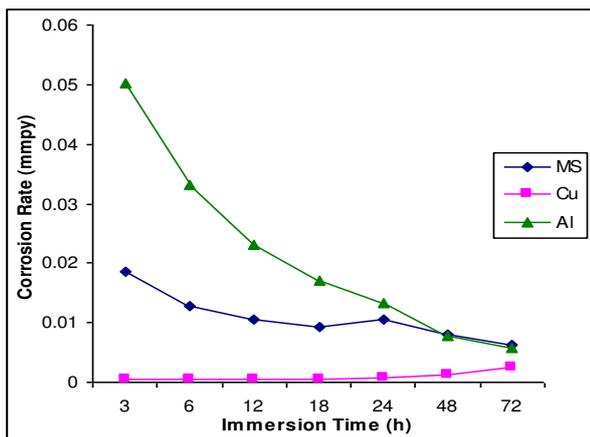


Fig 3: Corrosion Rate Vs time for Al, Cu, and MS in 0.5 N HCl

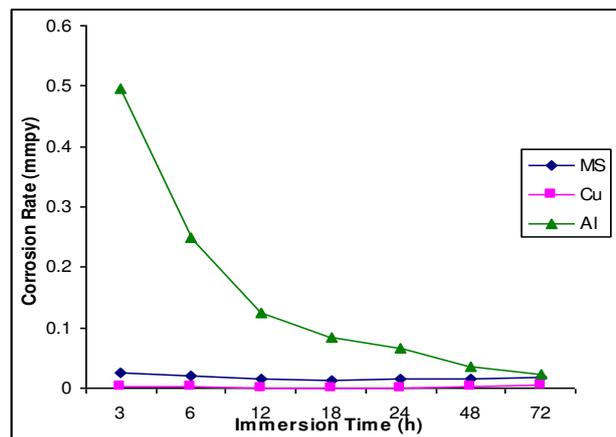


Fig 4: Corrosion Rate Vs time for Al, Cu, and MS in 2.0 N HCl

Figures (1- 8) and tables (1 and 2) reveals that aluminium is found to be more corroded in the HCl media as compare to H₃PO₄ media while in H₃PO₄ mild steel is found to be more corroded in as compared to aluminium and copper. This is because of the anion effect, the presence of aggressive anions like chloride (Cl⁻) accelerates the process of corrosion and form a soluble aluminium complex i.e. AlCl₃. Aluminium is more reactive in HCl acid solution as compare to Phosphoric acid solution as reported by Peter R. Kosting et al. [17]. This is because aluminium is more reactive than the MS and Cu according to standard Electrochemical potential series and initially aluminium being react violently in initial hours, in HCl medium, as immersion time is increased the film of aluminium chloride formed and considerable weight loss is smaller observed, so Al is active than copper and MS and considerably copper doesn't frequently

**International Journal of Innovative Research in Science,
Engineering and Technology**

(An ISO 3297: 2007 Certified Organization)

Vol. 2, Issue 10, October 2013

affected by these varying acidic strengths. Thus the following trends were observed from weight loss and corrosion rate data in HCl acid and in H₃PO₄ acid solutions.

In HCl medium: Al > MS > Cu ; whereas in H₃PO₄ medium, the dissolution trend observed was: MS > Al > Cu

There are also some final products of reaction of these three metals in HCl as well as H₃PO₄ acids as explained in literature by Atkins P. W. et al. [18].

Elboujdaini et al. [19] elucidated that the electrochemical reaction of iron in HCl acid than the corrosion reaction involves transfer of two electrons, and then there are two steps, each one represents transfer of one electron and one of them may be considered the rate determining step. This result is in good agreement with the mechanisms that are suggested for iron dissolution.

Table1. Table showing various corrosion parameters in Hydrochloric acid media:

Metal	Time (h)	HCl medium					
		0.5 N			2.0 N		
		Weight Loss (g) $\times 10^{-2}$	ρ_{corr} (mmy^{-1}) $\times 10^{-3}$	Activation Energy (Ea) KJ/mol	Weight Loss (g) $\times 10^{-2}$	ρ_{corr} (mmy^{-1}) $\times 10^{-3}$	Activation Energy (Ea) KJ/mol
Al	3	7.51	5.03	1.03012	7.4	0.50	0.65675
	6	7.42	3.3193		9.9	2.48778	
	12	7.46	2.3134		13.8	1.2506	
	18	6.75	1.7099		15.3	4.382	
	24	7.81	1.3411		16.0	6.5464	
	48	8.22	0.07753		18.5	3.445	
	72	8.44	0.05867		21.0	2.3581	
Cu	3	0.3	0.609	1.87	0.13	0.2639	0.59
	6	0.6	0.609		0.15	0.1522	
	12	0.9	0.457		0.19	0.0964	
	18	0.17	0.575		0.24	0.0812	
	24	0.26	0.66		0.03	0.0761	
	48	0.104	1.319		1.35	0.01713	
	72	0.31	2.622		6.08	0.05142	
MS	3	81.0	18.658	2.21341	1.08	24.877	1.26563
	6	112.0	12.899		1.66	19.119	
	12	183.0	20.538		2.64	15.203	
	18	241.0	29.252		3.6	13.821	
	24	368.0	20.596		4.95	14.253	
	48	555.0	13.799		10.75	15.476	
	72	649.0	16.229		17.66	16.95	

International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

Vol. 2, Issue 10, October 2013

Table 2: Table showing various corrosion parameters in Phosphoric acid medium:

Metal	Time (h)	H ₃ PO ₄ medium					
		0.5 N			2.0 N		
		Weight Loss (g) ×10 ⁻²	ρ_{corr} (mmy ⁻¹) ×10 ⁻³	Activation Energy (Ea) KJ/mol	Weight Loss (g) ×10 ⁻²	ρ_{corr} (mmy ⁻¹) ×10 ⁻³	Activation Energy (Ea) KJ/mol
Al	3	0.07	4.694	4.08	0.15	10.058	2.94
	6	0.13	4.359		0.3	10.068	
	12	0.23	3.856		5.2	8.717	
	18	0.29	3.241		7.9	8.829	
	24	0.37	3.101		9.7	8.131	
	48	0.55	2.305		17.1	7.167	
	72	0.87	2.431		22.1	6.175	
Cu	3	0	0	8.279224	0.02	0.0407	6.53492
	6	0.02	0.0203		0.03	0.0305	
	12	0.04	0.0202		0.04	0.0203	
	18	0.06	0.0203		0.04	0.0136	
	24	0.08	0.0204		0.12	0.0305	
	48	0.12	0.0153		0.23	0.0292	
	72	0.21	0.0178		0.32	0.0271	
MS	3	95.0	2.2051	1.25988	96	371.39	0.48634
	6	126.0	146.23		128.40	329.61	
	12	214.0	124.18		253.0	307.56	
	18	265.0	102.52		273.2	283.18	
	24	323.0	093.72		387.0	252.43	
	48	564.0	052.81		538	194.11	
	72	712.0	039.85		741	136.37	

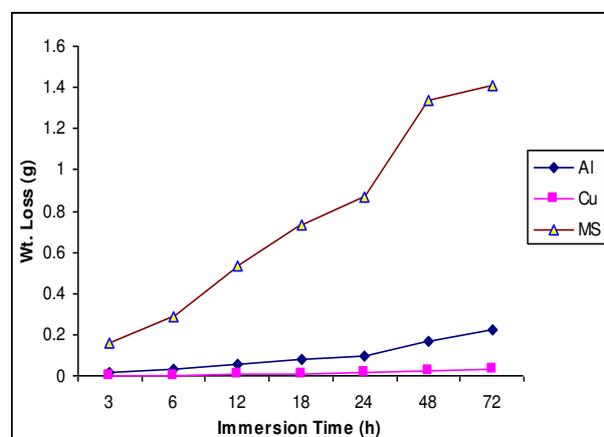
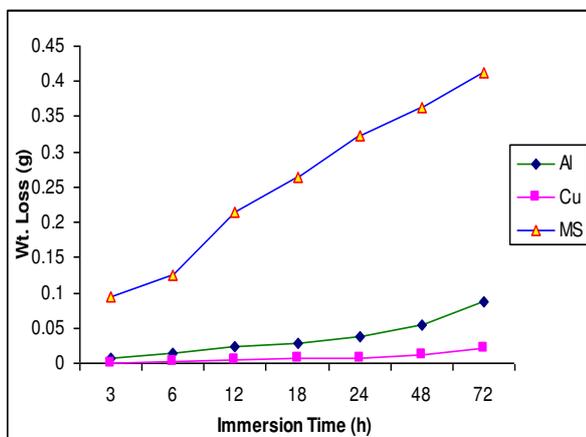


Fig 5: Weight Loss Vs time for Al, Cu, and MS in 0.5 N H₃PO₄ Fig 6: Weight Loss Vs time for Al, Cu, and MS in 2.0 N H₃PO₄

International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

Vol. 2, Issue 10, October 2013

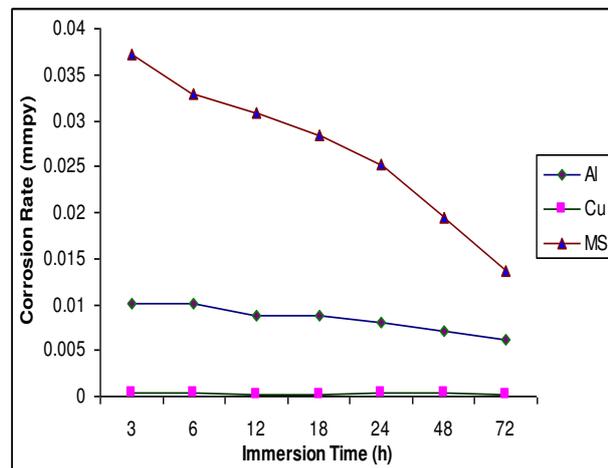
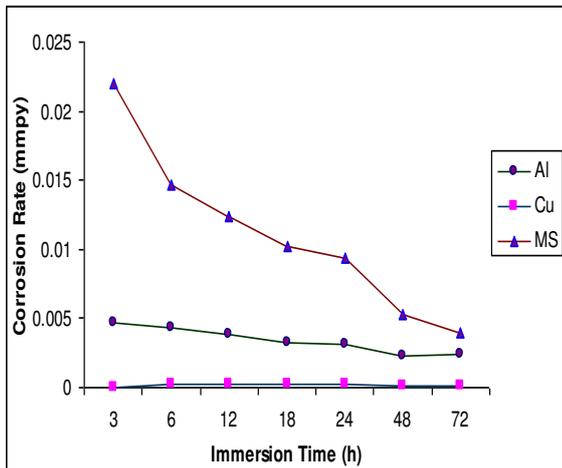
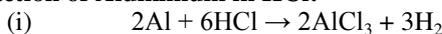


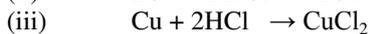
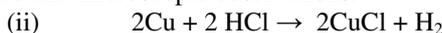
Fig 7: Corrosion Rate Vs time (Al, Cu & MS in 0.5 N H₃PO₄) Fig 8: Corrosion Rate Vs time (Al, Cu, and MS in 2.0 N H₃PO₄)

Reaction of Aluminium in HCl:

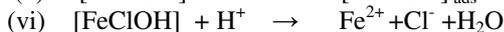
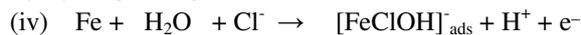


Reaction of Copper in HCl:

Literature suggest that usually copper does not react with HCl acid due to oxide layer formation on it. Initial value of corrosion rate and higher value of activation energy also proves the above view. but with the increase in immersion times copper starts reacting with HCl acid and form cuprous and cupric chloride, these result can also be explained by macro and microscopic observations.

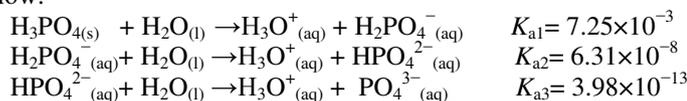


Reaction of MS in HCl:

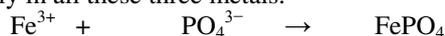


Reactions in H₃PO₄ medium:

In aqueous solution following reaction take place which complete in H₃PO₄ acid medium such as: orthophosphoric acid molecules dissociate up to three times, giving up an H⁺ ion each time, which typically combines with a water molecule, as shown below:



The anion formed after the third dissociation i.e. PO₄³⁻, is known as the phosphate or orthophosphate ion, which react with Al³⁺, Cu²⁺, Fe³⁺ ion and form Aluminium phosphate (AlPO₄), Copper phosphate Cu₃(PO₄)₂ and Ferric phosphate (FePO₄) respectively in all these three metals.



C. Kinetic parameters (Activation energy):

The activation energy of different chemical reactions is expressed by the Arrhenius law:

$$K = A \cdot e^{-E_a/RT} \dots\dots\dots (1)$$

Here k is the rate constant of any chemical reaction. A is the pre-exponential factor or simply the prefactor and R is the gas constant. The units of the pre-exponential factor are identical to those of the rate constant. Above equation shows the dependence of the rate constant k of chemical reactions on the temperature (T) and activation energy (E_a) attributed by Khalid Al-malahyet al. [20].

International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

Vol. 2, Issue 10, October 2013

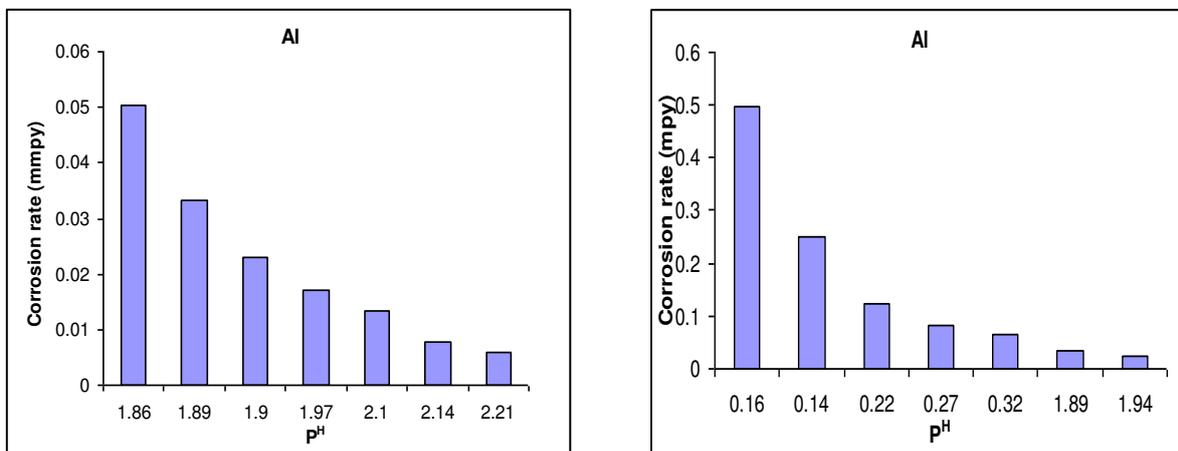
Arrhenius law predicts that corrosion rate increases with increase in the temperature and decrease with increases in E_a and A may vary with temperature (Eq.1). Nowadays it is best seen as below, empirical relationship. The activation energy may be calculated as a straight line of the logarithm of the corrosion rate (mm^{-1}) and $1/T$ according to the following relation:

$$\ln k = -E_a/RT + \ln A$$

The different value of activation energy in different acidic strength of two aggressive media were calculated and summarized in tables 1 and 2. The experimental values obtained clearly indicate that E_a for aluminium is lower in HCl than H_3PO_4 acid; this reveals that the corrosion rate is higher in HCl media compared to H_3PO_4 acid media. In other case when we compared the E_a value of Cu and MS in both acidic media Cu corrodes more in HCl while MS more corrode in H_3PO_4 acid. These results clearly explained by the higher and lower value of activation energy.

D. The pH effect on Corrosion behavior:

Pourbaix M.et al. [21] explained that the acidity or alkalinity of medium affects the corrosion behavior of metals and alloys, and it accelerates strongly where two dissimilar metals are contact with each other and oxide coating is not present. A slightly higher pH about (5-6) was observed in phosphoric acid solutions that than of Hydrochloric acid (0.1-2.5). The pH of both the solutions was found increased significantly compared to their initial pH values and similar a slight increase in the pH for the 0.5 N HCl acid solutions was also observed. Thus suggesting that a significant amount of hydrogen consumption occurs in these acids solutions. When these metals are dipped for different immersion time period to study the effect of pH, it reveals that corrosion rates are significantly greater at lower pH for Al and as the pH increases, a slight decrease in corrosion rate was observed. Whereas, for copper where the dissolution potential was not significantly affected by a variation in pH. The results for mild steel indicate that both uniform corrosion and localized corrosion are affected by a decrease in pH. It also reveals by reference figures (9-10) for Aluminium as given below:



Figures 9-10: Variation in Corrosion rate of Al with pH in (left) HCl and (right) H_2PO_4

E. Microscopic Surface Morphology of Coupons:

The microscopic surface morphological analysis was undertaken by Optical micrographs and Scanning Electron Microscope (SEM). Optical micrographs were taken with the help of (LABOMED) microscope and SEM was taken with the help of ZEISS special edition instrument. Several conclusions may be drawn from the results of these scoping experiments that could apply to these different acids.

Optical Micrographs (OM):

Figures 11-13 are optical micrographs of the all three metal coupons i.e. (Al, Cu, Ms) that reveal the morphology of the non-corroded polished metals prior to immersions in aggressive media.

**International Journal of Innovative Research in Science,
Engineering and Technology**

(An ISO 3297: 2007 Certified Organization)

Vol. 2, Issue 10, October 2013

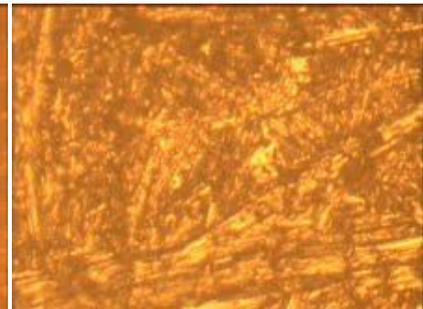
Fig. 11 Aluminium



Fig 12: Copper



Fig. 13: Mild Steel



Figures 11-13: shows the non-corroded surfaces of Al, Cu, MS coupons.

Figure 14-19 shows the corroded surface of all three metal coupons that had been exposed to 0.5 N and 2.0 N hydrochloric acids. The surface is rough and bumpy, indicative of general corrosion. All three metal coupons that were immersed in 0.5 N and 2.0 N H_3PO_4 acids were very similar in appearance to that of coupons immersed in the 2.0 N HCl solutions. The only exception was that a significant destruction of the MS coupons surface has been found when exposed for 72 h (figures 22 and 25).

Fig 14: Aluminium (0.5 N HCl)



Fig. 15: Copper (0.5 N HCl)

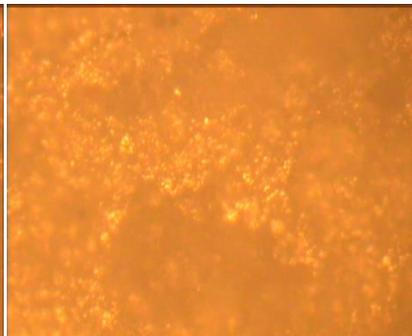


Fig. 16: Mild Steel (0.5 N HCl)



Fig. 17: Aluminium (2.0 N HCl)



Fig. 18: Copper (2.0 N HCl)



Fig. 19: Mild Steel (2.0 N HCl)



Figures: 14-19 shows the corroded metal surface of Al, Cu, MS coupons in 0.5 N and 2.0 N HCl acid solutions

**International Journal of Innovative Research in Science,
Engineering and Technology**

(An ISO 3297: 2007 Certified Organization)

Vol. 2, Issue 10, October 2013

Several areas of these coupons were damaged which was evident by their surface appearance, the film present on the surface was cracked and flaked off. There was an insignificant amount of corrosion product on these coupons which exposed to the acid. All coupons exhibited severe general attack and crevice attack where the glass string had been inserted into the hole. In all cases, the corrosive attack had eaten through the top edge of the coupon and the coupon had fallen to the bottom of the bottle. Further crevice attack ensued on the edge of Cu and MS Coupons (figures 15 & 16 and 18 & 19 respectively) in 2.0 N HCl acid. The surface of MS coupon shows highly dissolution and also the deposition of ferric chloride was observable. Thus, in general, the HCl acidic medium is more aggressive than H₃PO₄ acidic medium, wherever Cu coupons are less infected by these media, as compared to Al and MS Coupons.

Fig. 20: Aluminium (0.5 N H₃PO₄) Fig. 21: Copper (0.5 N H₃PO₄) Fig. 22: Mild Steel (0.5 N H₃PO₄)

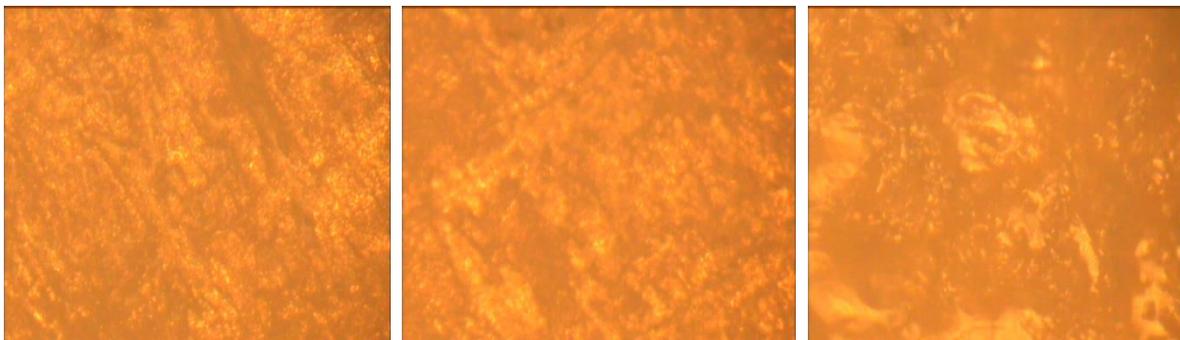
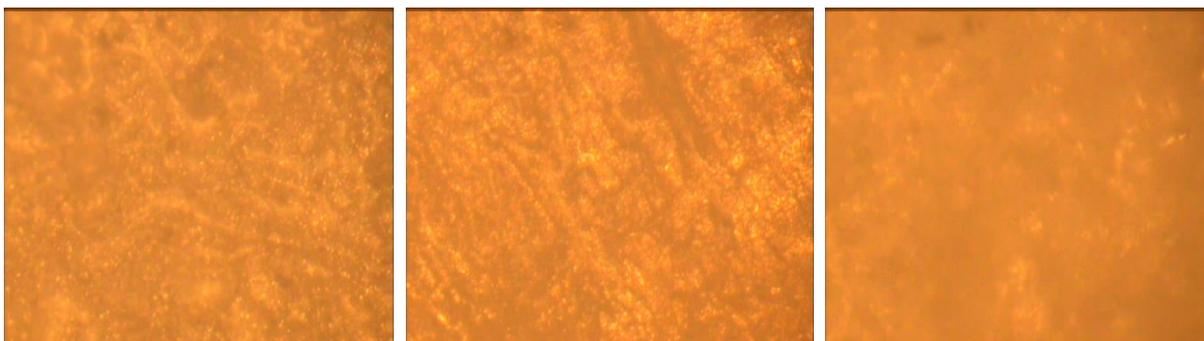


Fig. 23: Aluminium (2.0 N H₃PO₄) Fig. 24: Copper (2.0 N H₃PO₄) Fig. 25: Mild Steel (2.0 N H₃PO₄)



Figures: (20-25) shows the corroded metal surface of Al, Cu and MS coupons in 0.5 N and 2.0 N H₃PO₄ acid solutions.

Scanning Electron Microscopy (SEM):

The surface morphology of polished coupons of metal viz aluminium, copper and mild steel was studied in various concentrations of HCl and H₃PO₄ using scanning electron microscopy (figures 26 – 34). SEM was taken by the help of ZEISS Scanning Electron Microscope.

Figures 26-28 shows the micrographs of polished metal coupons which are non-corroded prior to immersion in aggressive media.

**International Journal of Innovative Research in Science,
Engineering and Technology**

(An ISO 3297: 2007 Certified Organization)

Vol. 2, Issue 10, October 2013

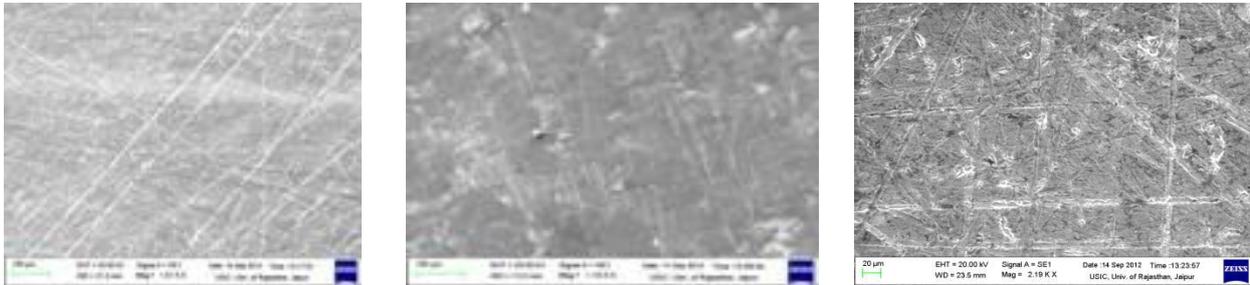


Figure: 26-28 SEM shows the polished surface of non-corroded Al, Cu and MS coupons.

From figures 29-34, the micro-flakes are clearly visible depicting dissolution of metal surfaces. Also indicate that the surface is strongly and uniformly damaged by corrosion due to aggressive media. From the figures it can be observed that the pattern of corrosion is different in both the acid media.

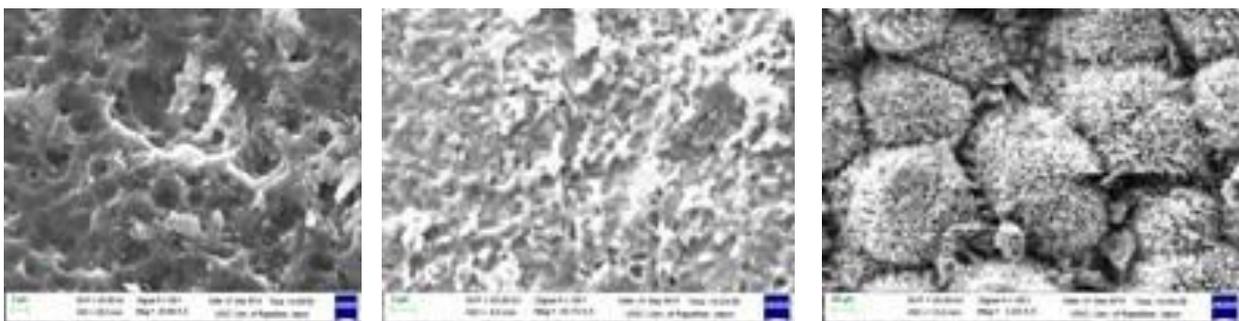


Figure: 29-31 SEM of surfaces of metal coupons viz. Al, Cu, and MS after 72 hours of immersion in HCl media

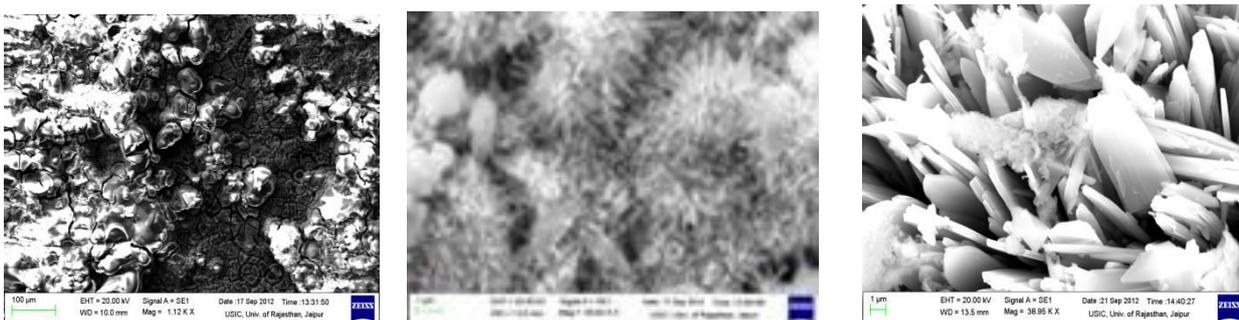


Figure 32-34 SEM of surfaces of metal coupons viz Al, Cu, and MS after 72 hours of immersion in H₃PO₄ media

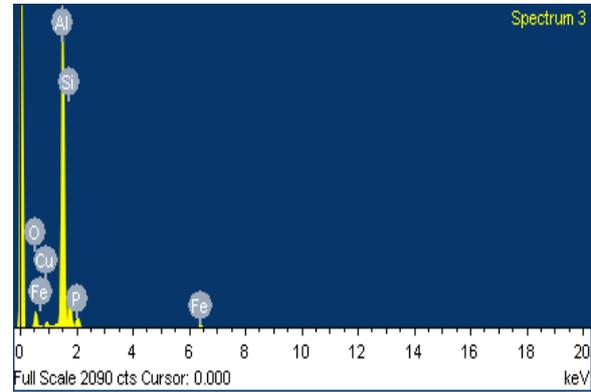
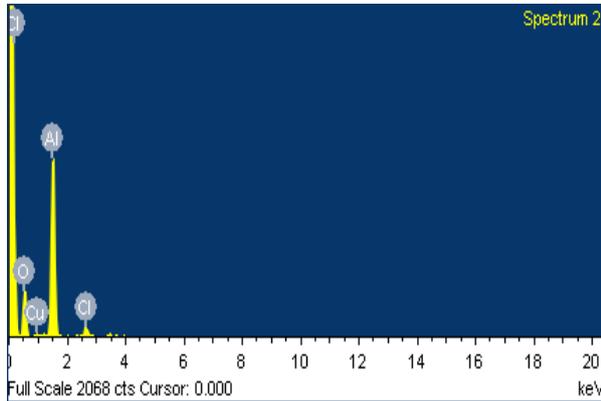
Energy Dispersive X-RAY (EDX):

The EDX technique used to identify the elemental composition of as small as cubic micron of material (figures 35-40). EDX was taken by the help of ZEISS Sanning Electron Microscope equipped with AN-10000 link EDX spectrometer. EDX spectrum of the sample indicated that corrosion product layer has been formed over the surface of the metal samples in different acid media.

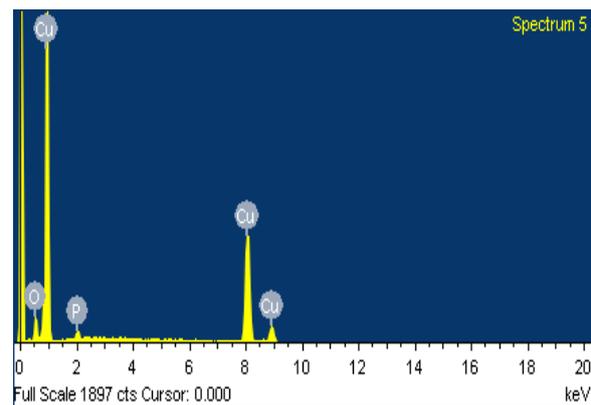
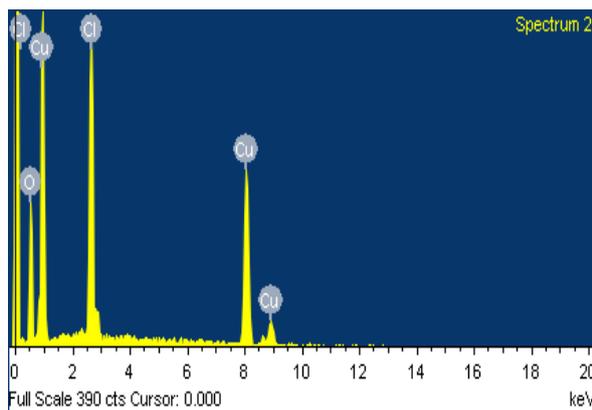
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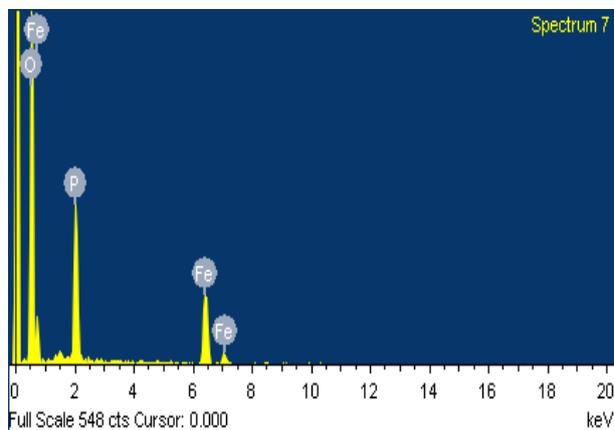
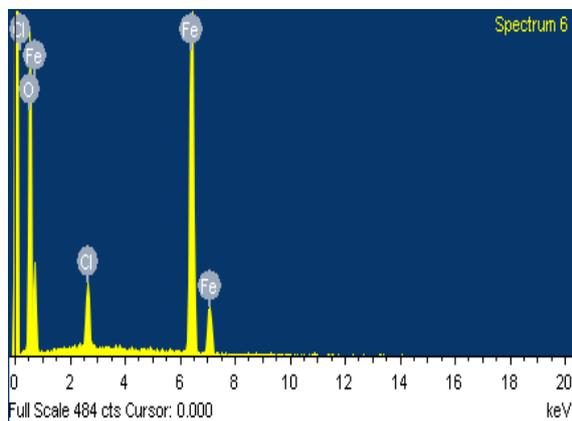
Vol. 2, Issue 10, October 2013



Figures 35-36: EDX of Aluminium in HCl and H₃PO₄ acid media.



Figures 37-38: EDX of Copper in HCl and H₃PO₄ acid media.



Figures 39-40: EDX of Mild Steel in HCl and H₃PO₄ acid media.

The Cl and P peaks in above spectra clearly indicate the reaction of metal with both the acid media. As the aluminium corroding more in HCl media the sharp peaks of chloride ions appears in the spectra similarly fine peaks are seen of

International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

Vol. 2, Issue 10, October 2013

mild steel in phosphoric acid which is more corroded in H₃PO₄ media. Hence it can be revealed that the above results are in good agreement with the results suggested by the weight loss.

XRF graphs of Metal Coupons:

The XRF studies were also carried out by the help of Pay Analytical X-ray fluorescence instrument in the frequency range of 25 KV. The study shows the elemental compositions of these coupons (figures 41-43). The highest peak in each graph shows the purity of Al, Cu and Fe for these metals respectively.

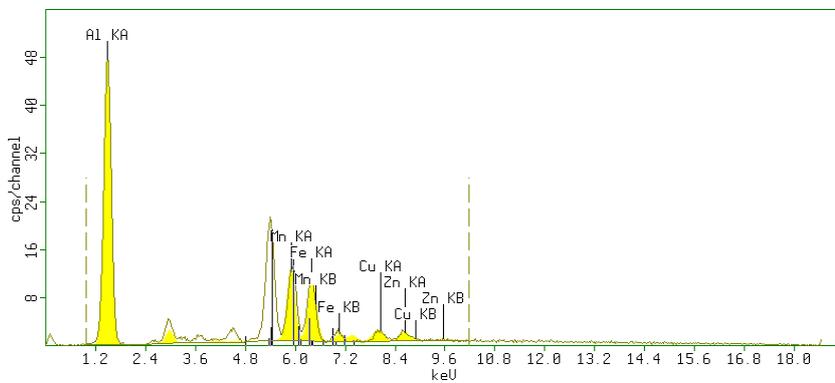


Fig. 41: XRF graph of Aluminium coupon

Compound	Conc (%)
Al	97.6
Mn	1.3
Fe	0.87

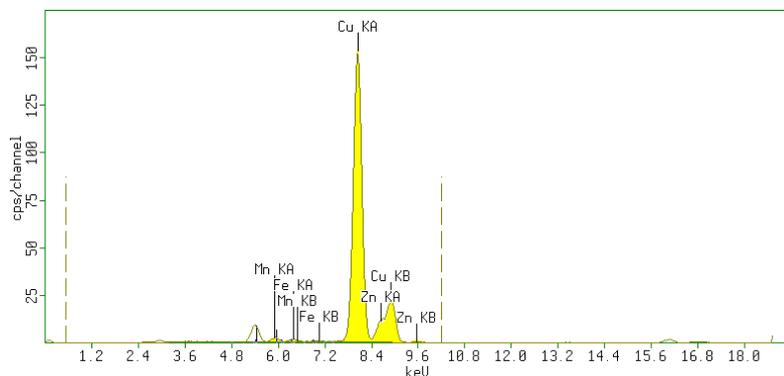


Fig. 42: XRF graph of Copper coupon

Compound	Conc (%)
Mn	0.51
Fe	0.19
Cu	93.6
Zn	5.7

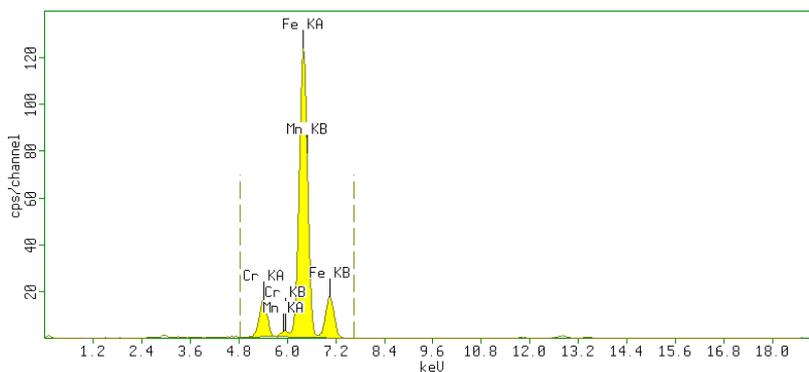


Fig. 43: XRF graph of Mild Steel coupon

Compound	Conc (%)
Mn	0.39
Fe	98.7

International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

Vol. 2, Issue 10, October 2013

IV. CONCLUSIONS

The dissolution behavior of Aluminium, Copper and Mild Steel in 0.5 N and 2.0 N of HCl and H₃PO₄ solutions have been investigated using weight loss (chemical) method. The dissolution was further endorsed by carrying out surface morphological studies employing micro spectroscopic techniques. The following conclusions can be drawn on the basis of the results:

- * Weight-loss data and corrosion rate graphs reveals that the Al coupons immersed in HCl acid is more corrode compare to H₃PO₄ acid solutions, this is due to formation of Aluminium chloride, and the value of activation energy shows the reaction feasibility.
- * Mild steel coupons shown the insignificant dissolution in H₃PO₄ acidic medium in 0.5 N as well as 2.0 N concentrations of acids compare to HCl acids due to formation of unstable FePO₄ compound. This effect increases with increases of their immersion time.
- * Dissolution of Copper is less in HCl as well as in H₃PO₄ acidic medium compare to other two metals, but when compared in different acidic strength than Cu is more reactive in HCl acid than H₃PO₄ acidic medium.
- * Scanning electron microscopy (SEM), and Optical micrographs, EDX was used to observe surface defects and corrosion. SEM observations showed that defects or pitting corrosion occurred on the surfaces of the all coupons in tested media. EDX spectrum recorded on the area of dispersed pits and grooves shows the peaks of chloride and phosphate acidic media.

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