Corrosion Behavior of Annealed Iron Wires Produced in Nepal in Acidic, Alkaline, and Neutral Media

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Abstract Even though, a corrosion is an all-too-common result of electrochemical reactions between materials and substances in their environment, the corrosion of iron has captivated many researchers as it leads to a loss of structural integrity. Among many varieties of iron-based products produced in Nepal and their electrochemical corrosion studies, the annealed iron wires which are mostly used for reinforcing concrete and their tendency of undergoing corrosion are not being the subject of research interest so far. Present work is aimed at investigating corrosion/passivation behavior of the annealed iron wires in different corrosive media. As a complimentary electrochemical study, we measured the open circuit potentials (OCPs) of the two most popular and demandable annealed iron wires (named as IW1 and IW2) available in the Nepalese market in acidic, alkaline, and neutral media at 25ºC, open to air and found that both IW1 and IW2 possess more negative OCPs in neutral medium than in other two media, suggesting more corrosive in neutral and less corrosive in alkaline media. In other words, the neutral medium is found to be more aggressive leading to intensive iron wire corrosion than acidic and alkaline media. Unlike the iron wire sample IW2, the IW1 consumed longer time for the development of passive film having complete corrosion resistive capacity in neutral and acidic media indicating comparatively higher corrosion rate of IW1. We believed that the results achieved here would be highly applicable to produce better quality iron and steel products in Nepalese market.

Keywords Passivation, Iron products of Nepal, Open circuit potential (OCPs), Aggressive media

Introduction

Rusting or Corrosion is widely known as the destructive and unintentional degradation of a material caused by its environment. According to IUPAC, corrosion is an irreversible interfacial reaction of the materials (metal and its alloys, ceramic and polymer) with its environment, which results in consumption of the material or the dissolution of material into the components of the environment [1]. On the basis of the cause of the metal's chemical deterioration, there are many different types of corrosion such as general attack corrosion, localized corrosion, galvanic corrosion, environmental cracking, flow-assisted corrosion, intergranular corrosion, de-alloying, fretting corrosion, and high temperature corrosion [2]. Among them, the general attack corrosion is the most common type of corrosion caused by a chemical or electrochemical reaction that results in the deterioration of the entire exposed surface of a metal. More specifically, the general corrosion usually occurs when most or all of the atoms on the same metal surface are oxidized (tendency to lose electrons), damaging the entire surface [3].

According to electrochemical theory, corrosion process is a combination of anodic dissolution or oxidation and cathodic reduction. An acid-base reaction concept of corrosion [4] proposed to explain hydration or complexation of metal ions, however, is not widely discussed. As the metallic specimen is immersed in an electrolyte, the electrochemical reactions (two or more oxidation and reduction partial reactions) occur in such a way that the total rate of oxidation is equal to total rate of reduction as illustrated by Evans diagram in Figure 1. A metallic dissolution ($M \rightarrow M^{2+} + 2e^-$) usually occurs at anode. So, if a stable surface film is not formed, the dissolution rate measured in terms of anodic current density i_A generally increases logarithmically with increasing potential E, as shown in equation 1 and the rate of cathodic reaction involving hydrogen ions and/or oxygen dissolved in the solution ($2H^+ + 2e^- \rightarrow H_2$; $O_2 + 2H_2O + 4e^- \rightarrow 4OH^-$; $O_2 + 4H^+ + 4e^- \rightarrow H_2O$) rises logarithmically with lowering E, as shown in equation 2. At the open circuit corrosion potential, both anodic and cathodic reactions occur simultaneously with the same rate [5], as shown in equation 3.

$$i_A = i_A^o \, exp \, \alpha(E - E_m) \tag{1}$$

Where, i_A^o = Anodic exchange current density; α = constant; E_m = Equilibrium potential.

$$i_C = i_C^o \, exp[-\beta(E - E_C)] \tag{2}$$

Where, i_C^o = Cathodic exchange current density; β = constant; E_C = Equilibrium potential.
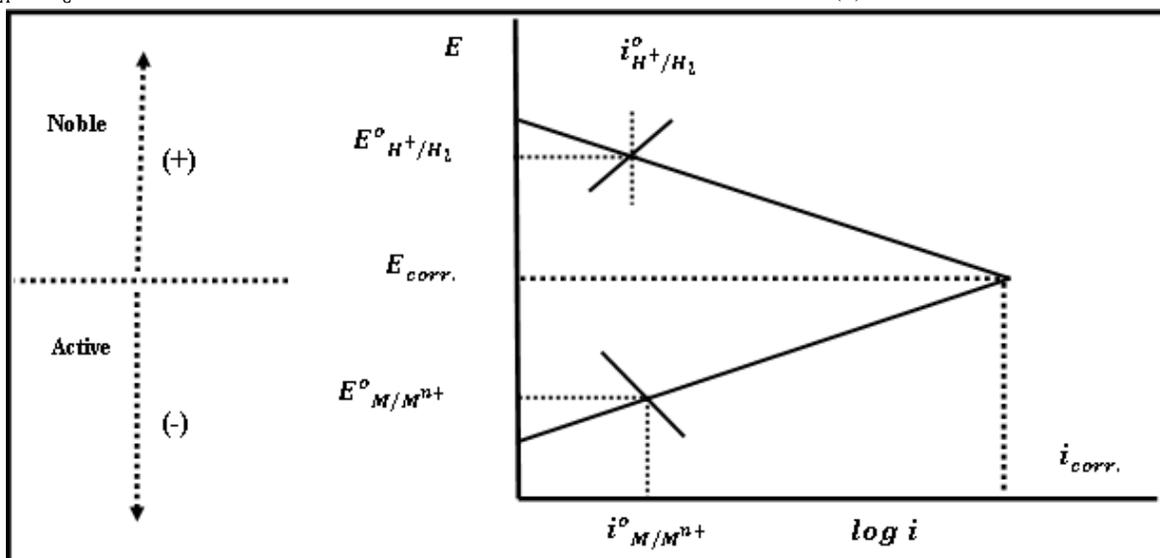
$$i_A = i_C \tag{3}$$

Figure 1: Schematic Evans diagram showing corrosion potential $E_{corr.}$ and corrosion current $i_{corr.}$

Corrosion of iron and steels have been a big problem since the beginning of the 20th century because of their wide application as structural materials. From the corrosion point of view, the discovery of stainless steels early in the 20th century was one of the incentives to study corrosion and passivity [6]. In the 1970s when instruments for surface analysis became available, many corrosion studies were performed by analyzing the surface of steels and iron products [7-9]. In the recent years, the low-scan rate cyclic voltammetry is used to study the electrochemical behavior of steel and iron products used to reinforce concrete [10]; an electrochemical impedance spectroscopy is used to measure the electrochemical behavior of carbon steel in alkaline sour environments [11]; the capillary electrode and scanning electron microscopy techniques are applied to study the galvanic corrosion behavior of aluminum alloys [12]; and the electrochemical measurements including open circuit potential (hereafter, OCPs) and potentiostatic polarization are used to study the corrosion behavior of sputter deposited nano-crystalline metallic alloys [13]. More interestingly, the theoretical methods such as semi-empirical PM6, DFT methods and UB3LYP using Gaussian software are being employed in the stationary and transient electrochemical studies where the coating behavior of steel in neutral solution is investigated [14]. Whatever advanced methodologies are being adopted in the electrochemical corrosion studies, the first and foremost step is measuring OCPs as it always provides

metallic tendencies of undergoing corrosion in different electrolytic media. As a rule, the potential below OCP is more thermodynamically stable (less tendency to take part in corrosion) whereas, the potential above OCP is considered as thermodynamically unstable and prone to corrosion. Thus, the metal with the low OCP will always dissolve/corrode faster in the electrolyte than the metal with high OCP. The further investigation for determining the passivity of the materials in acidic, alkaline, and neutral electrolytes can be carried out by studying OCPs graphs versus immersion time [15].

In Nepal, the industrialization gained momentum after the political changes in 1990. The liberal and open market policies helped the industries to grow. In 2008, the successive government focused on infrastructure development by spending national as well as international aid causing to enhance the total demand rate of iron and steel products in Nepal. At present, Nepal consumes around one million metric tons of iron and steel products annually and it has been increasing by 10 percent yearly [16]. To meet such demand rate, many iron and steel industries in Nepal are producing varieties of flat and long products types of iron and steel. The construction related products such as re-bar, TMT rods and wire products such as nails, galvanized wires, barbed wires, binding wires, and deformed wires come under long products. Among them, the binding wires (black annealed wire) are mostly used in construction for reinforcing concrete or daily used as binding material, or material for weaving wire mesh. They are supplied in the form of coil wire with 10-25 kg/roll while packaging, as depicted in chart 1a. They are soft annealed wires and can be cut into various sizes, as shown in chart 1b, for binding/bundle. They vary in diameter ranges from 0.7 to 1.4 mm [17-18].

Chart

In spite of such high production and consumption rate of iron products in Nepalese market, the domestic iron and steel industries have been facing a lot of problems with the main ones being the lack of a skilled workforce for research and development (R & D) and inadequate R & D budget. It has made the industries' pace of improvement and innovation low. The better products and innovative items can be manufactured if the government provides research opportunities and design facilities to the industries. In this context, we thought that investigating the electrochemical corrosion/passivation behavior of the major annealed iron wires produced in Nepal in different aggressive environments would be an indispensable research work. We, at first, studied a part of this work and quickly disseminated the preliminary results to Nepalese society by non-electronic method of scientific communication [19]. At present, we report in-depth findings and explanations of this work to an electronic journal. This research work is mainly aimed at studying the corrosion behavior of the most demandable and popular annealed iron wires available in Nepalese market in acidic: 1M HCl; neutral: 0.5M NaCl; and alkaline: 1M NaOH solutions at 25°C, open to air using electrochemical measurements. The structure of this paper is: Materials and Methods in section 2, Results and Discussions in section 3, and Summary and Conclusions in section 4.

Materials and Methods

Annealed iron wires

We selected the most demandable and popular soft annealed iron wires that are made available in the Nepalese market by *JAY JAGADAMBA* and *PANCHAKANYA STEEL* industries as experimental specimens. The samples of the respective iron wires were directly collected from the respective suppliers located in Kathmandu, Nepal. Most of the time, they were collected from Kantipur Hardwares and Suppliers, Sanepa, Kathmandu. They are characterized as long iron products having: diameter ~1.2 mm, minimum tensile strength 380 Mpa, yield strength 170 Mpa, maximum Rockwell hardness (HRB) 88 and Brinell hardness (HB or BHN) 179. The approximate chemical composition of the wires produced by *JAY JAGADAMBA* are disclosed as C(Max): 0.08%; Mn(Max): 1.00%; P(Max): 0.04%; S(Max): 0.01%; Si(Max):1.00%; Cr:10-11.75%; Ni:0%; Mo:0%; N:0% and Ti: 6×(C+N)% (Min.) or 0.70% (Max.) [17]. Such chemical compositions and mechanical properties of the annealed iron wires produced by *PANCHAKANYA* industry are not made available to the public. For the experimental simplicity, we randomly named these iron wire samples as IW1 and IW2 without any proper order.

Electrochemical Measurement

Chart 2

(a)   **(b)**
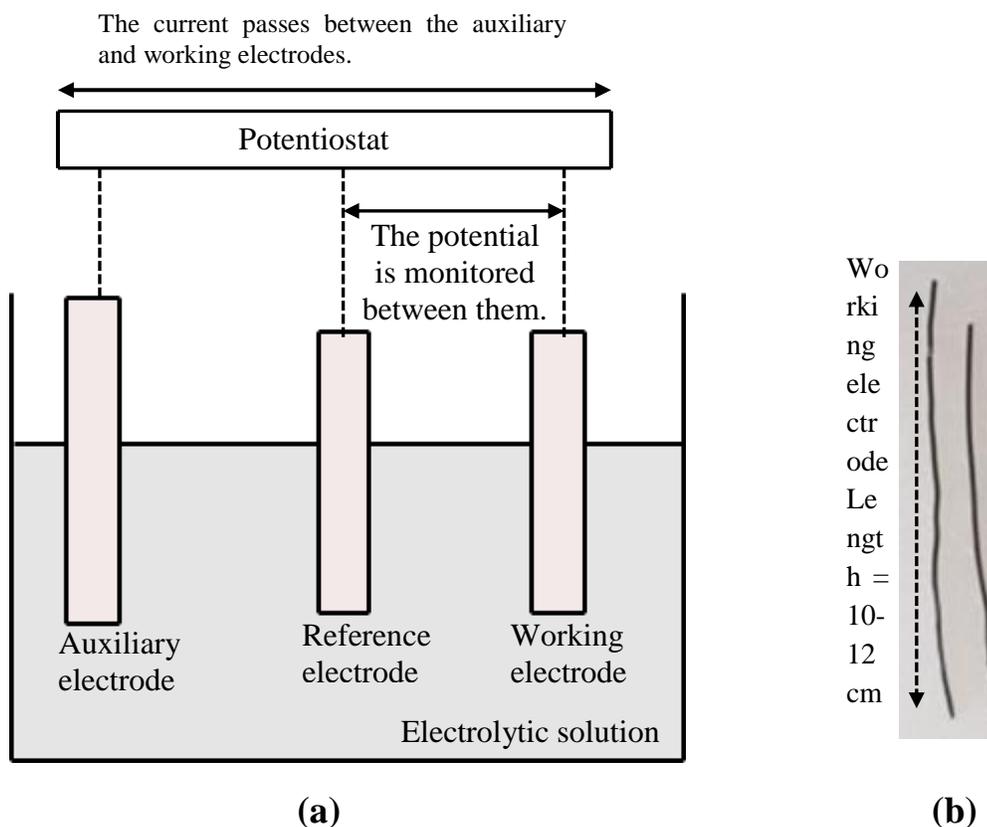
As the corrosion or metallic dissolution occurs via electrochemical reactions, electrochemical techniques are extremely essential for studying corrosion processes. In this technique, an experimental metallic specimen (in this case, iron wire) with a surface area of a few square centimeters is usually immersed in a solution (corroding system to be studied) as a working electrode along with the two additional electrodes (reference and auxiliary electrodes)

and all are connected to a device called a potentiostat. By using it, one can change the potential of the working electrode in a controlled manner and measure the current that flows as a function of the applied potential. The schematic diagram illustrating the working principle of three electrode systems is shown in chart 2a. The detailed experimental procedures are given in the following subsections.

Preparation of iron wire specimens

At first, the collected annealed iron wires, IW1 and IW2, were cut separately into short pieces of 10-12 cm length, as shown in chart 2b. The surface of each wire specimen was mechanically polished with a silicon carbide paper up to grit number 1500 in cyclohexane, rinsed with acetone and dried by an air blower. Each wire specimen was clipped by the crocodile pin welded with a stainless steel and about 3 cm length of it was immersed in a respective corroding electrolyte.

Electrolytes and Electrodes

The three different electrolytes used for all the electrochemical measurements are of very low concentrated: 1M HCl, 0.5M NaCl, and 1M NaOH. The analytical grade reagent of HCl, NaCl, and NaOH was used to prepare respective aqueous solutions in distilled water, open to air and their temperature was controlled to 25 ±1°C by using water bath. Thus, all of these aqueous electrolytes are well aerated. A saturated calomel electrode (hereafter, SCE) and a platinum mesh were used as the reference and auxiliary electrode respectively. Each wire specimen was used as a working electrode in the electrochemical corrosion experiments. All the potentials were recorded with respect to SCE.

Open Circuit Potential

The open circuit potential (also referred to as the equilibrium potential, the rest potential, or the corrosion potential) is the potential at which there is no current; i.e., experiments based on the measurement of the OCPs are potentiometric experiments. Although such measurements are quite common, they have many important applications including identification of metallic tendencies for undergoing more/less corrosion. In this study, we have employed a potentiometric methodology to measure the OCPs of the annealed iron wires in different aggressive media. The OCPs for each iron wire specimen (IW1 and IW2) were recorded by using a digital potentiometer (DT-2101, HI-TECK INSTRUMENTS ENGLAND) at 25°C, open to air, with respect to SCE at different time intervals. The potentiometer reading was noted immediately after immersing each wire specimen in each electrolyte solution for about 2 h. We observed maximum fluctuation of the OCPs within 1.5 h and then almost attained steady state. That is why, we set 2 h as maximum time period while measuring OCPs for each IW1 and IW2 in different electrolytes. All the OCP measurements were repeated two times or more.

Results and Discussion

It is obvious that the change in corrosion potentials or OCPs always depends on the change in one or both of the anodic and cathodic reactions. For example, an increase/decrease in corrosion potential can be attributed to a decrease/increase in the anodic reaction or the increase/decrease in the cathodic reaction respectively. The anodic reaction usually decreases after the growth of a passive film on the anodic surface and the cathodic reaction increases due to increase in dissolved oxygen in the electrolyte solution. Thus, measuring OCPs at a regular time interval in electrochemical experiments is extremely necessary to unveil the dissolution/corrosion and passivation behavior of the metallic specimens. In this work, we recorded the OCPs of the iron wires IW1 and IW2 at different time interval in each 1M NaOH, 1M HCl, and 0.5M NaCl solution separately at 25 °C, open to air for 2 h and plotted the OCPs graph as a function of immersion time as shown in Figure 2, Figure 3, and Figure 4 respectively. For both iron wires IW1 and IW2, the OCPs in acidic (1M HCl) and neutral (0.5M NaCl) solutions after about 30 min. immersion are almost same except slight fluctuations in alkaline (1M NaOH) solution. However, the nature of the changes of the OCPs with immersion time in all three media are dissimilar. In 1M HCl and 0.5M NaCl solutions, the OCPs for both wire samples are shifted to more negative (or less noble) direction and in 1M NaOH solution, the

OCPs are shifted to more positive (or more noble) direction with immersion time. More specifically, the OCPs for the iron wires IW1 and IW2 in 0.5M NaCl solution, are more negative value (i.e., about -580 mV, SCE) than that in a 1M HCl solution and in 1M NaOH solution, it is more positive value (i.e., about -150 mV, SCE) than in 0.5M NaCl solution as shown in Figure 5(a) and Figure 5(b).

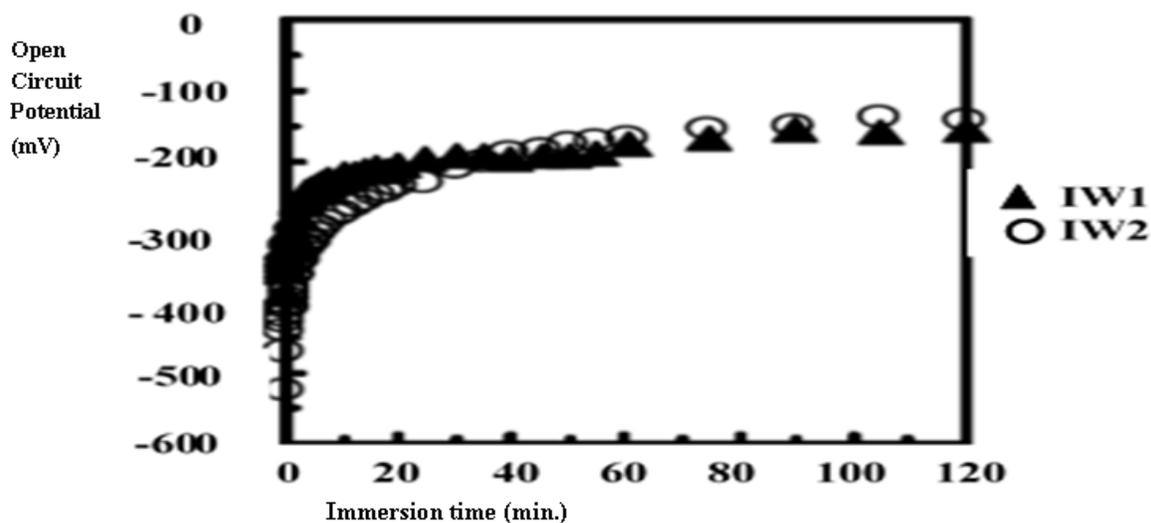**Annealed iron wires of Nepal in 1M NaOH solution at 25°C, open to air.**

Figure 2: Changes in OCPs of iron wires in 1 M NaOH solution at 25 ºC, open to air as a function of immersion time

Annealed iron wires of Nepal in 1M HCl solution at 25°C, open to air.
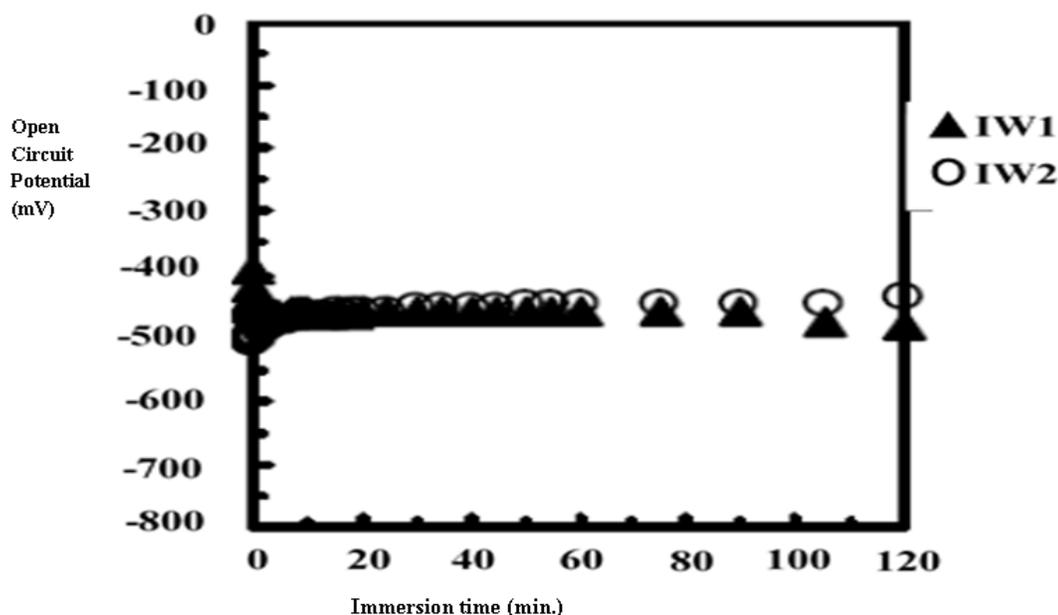
Figure 3: Changes in OCPs of iron wires in 1M HCl solution at 25 ºC, open to air as a function of immersion time

In 0.5M NaCl and 1M HCl solutions, the initial potential of the iron wires IW1 and IW2 increases towards more negative value due to the dissolution of a preformed air oxide film on the iron wire surface. The potential then

slightly shift in more negative direction with increasing time till the end of the experiment. This negative shifting is resulted from the dissolution of iron wires by the attack of aqueous chloride ions (Cl⁻ (aq.)) [20] present in the aqueous solutions of 0.5M NaCl and 1M HCl. The corrosion of iron wires in 1M HCl solution can be simply understood by the electrochemical reactions: Fe (s) + 2HCl (aq.) → FeCl$_2$ (aq.) + H$_2$ (g) i.e. Fe(s) → Fe^{2+} (aq.) + 2e⁻, $E°$ = −0.44 V (oxidation reaction) and O$_2$ (aq.) + 4H$^+$ + 4e⁻→ 2H$_2$O, $E°$ = +1.23 V (reduction reaction) and, the overall redox reaction is 2Fe(s) + O$_2$ (aq.) + 4H$^+$ → 2Fe^{2+} (aq.) + 2H$_2$O, $E°$ = +1.67 V. One more possible reduction in acidic medium is 2H$^+$ + 2e⁻ → H$_2$, $E°$ = 0V.

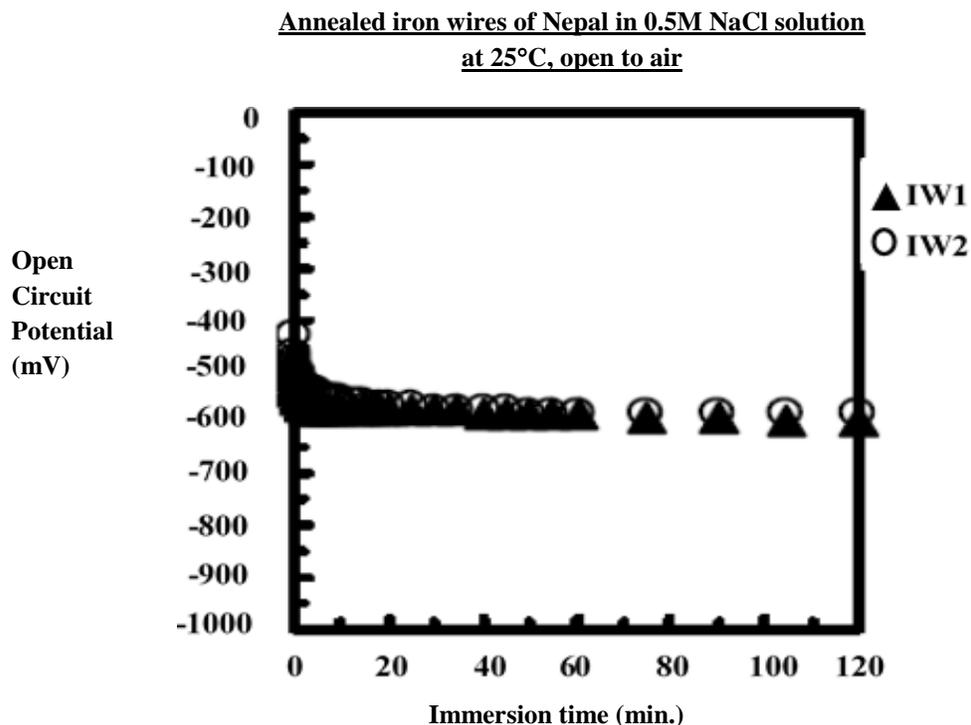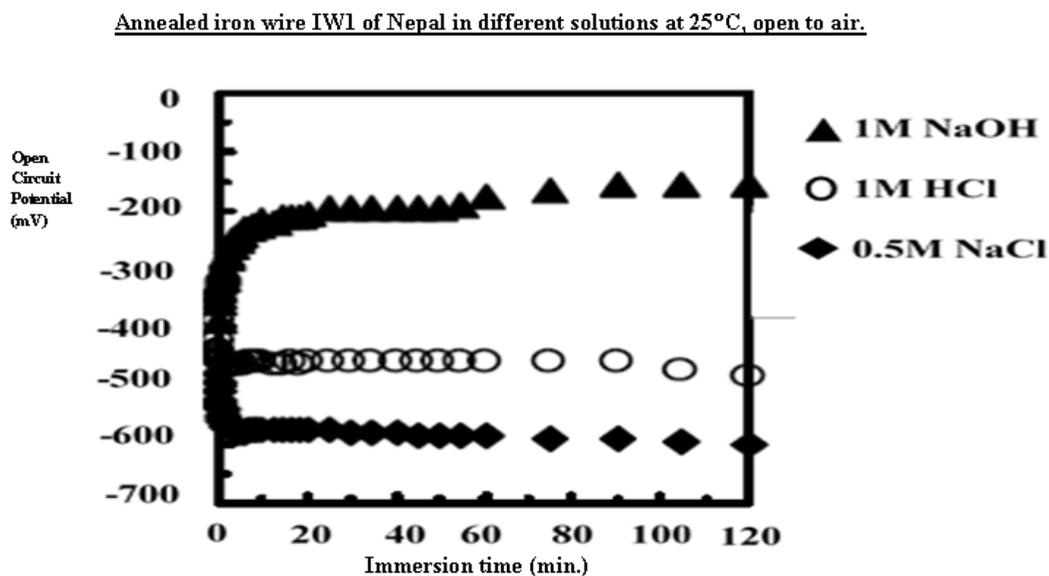**Annealed iron wires of Nepal in 0.5M NaCl solution at 25°C, open to air**

Figure 4: Changes in OCPs of iron wires in 0.5M NaCl solution at 25ºC, open to air as a function of immersion time

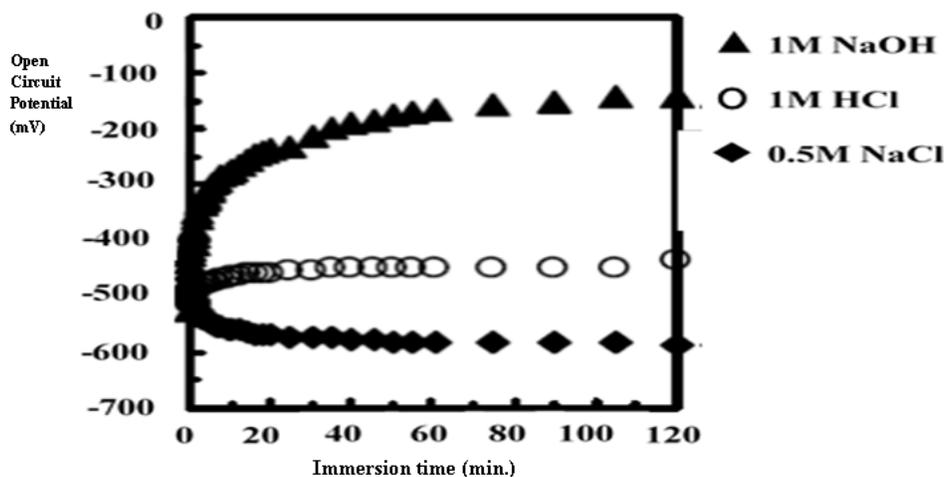**Annealed iron wire IW1 of Nepal in different solutions at 25°C, open to air.**

Figure 5: Changes in OCPs of (a) IW1 and (b) IW2 iron wires in different aggressive media at 25°C, open to air as a function of immersion time
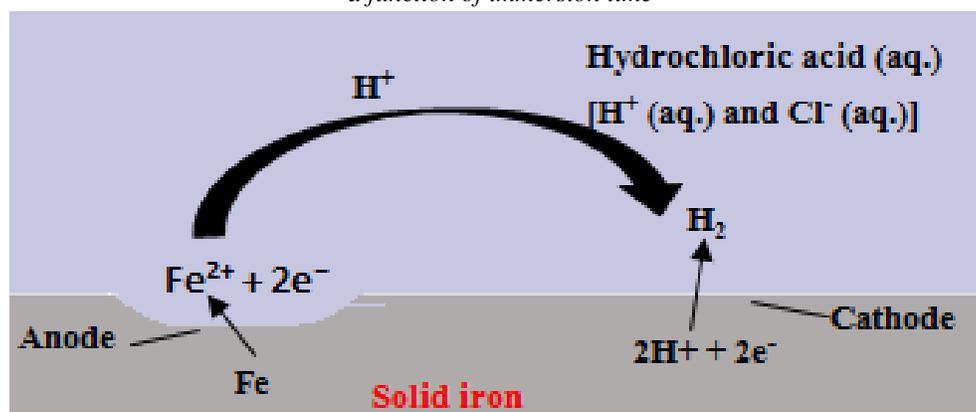

Figure 6: Mechanism of iron corrosion in acidic medium.

It is clarified in Figure 6 as well. Whereas, in 1M NaCl solution, the corrosion rate is usually hastened by improving the efficiency (conductivity) of the electrolyte. The ions Na^+ (aq.) and Cl^- (aq.) present in the 1M NaCl (aq.) solution assist in transferring respective ions through the electrolyte which finally speeds up the corrosion rate of iron that is initiated by losing electrons (oxidation). The electrons produced from the oxidation of iron wire are transferred to oxygen present in aerated neutral solution for reduction i.e. $Fe(s) \rightarrow Fe^{2+}$ (aq.) $+ 2e^-$, $E° = -0.44$ V (oxidation reaction) and O_2 (g)$\leftrightarrow O_2$ (aq.); O_2 (aq.) $+ 2H_2O$ (l) $+ 4e^- \rightarrow 4OH^-$ (aq.), $E° = +0.40$ V (reduction reaction), and the overall redox reaction is $2Fe(s) + O_2$ (aq.) $+ 2H_2O$ (aq.) $\rightarrow 2Fe^{2+}$ (aq.) $+ 4OH^-$, $E° = +0.84$ V. In either of these two electrolyte solutions, the magnitude of the difference in voltage is the driving force for the oxidation-reduction reaction which in turn be for the corrosion process. Similarly, as $E°_{Fe^{2+}/Fe} < 0$ and $E°_{cell} > 0$, it makes $\Delta G° < 0$, which further acts as the net driving force for the corrosion. The slight positive shift in the potential of iron wire IW2 with time is observed in 0.5M NaCl and 1M HCl solutions after about 110 min. immersion time. This might be due to the formation of corrosion products and/or an oxide film having corrosion resistive capacity on the IW2 surface. This passivation is caused by the presence of Cr in IW2 whose percentage composition always determines the earlier or later development of chromium-rich transparent oxide protective film. As a rule, the percentage composition of Cr

must be not less than 10% for acting as a good facilitator in developing corrosion resistive film [21]. In contrary, the slight negative shift in the OCPs of iron wire IW1 with time is observed in the same solutions (0.5M NaCl and 1M HCl) even after 110 min. immersion time. It indicates that: IW1 is still being attacked by the aqueous chloride ions, IW1 is unable to develop protective film having complete corrosion resistive capacity yet on its surface, and IW1 needs comparatively longer time to reach at the passivation state. Unlike in the case of IW2, the delay in growing protective film on the IW1 surface might be either due to not containing required percentage composition of Cr or containing comparatively less percentage of Cr than in IW2. This is why, the IW1 wire sample is found to be dissolved (corroded) in the anodic solutions (0.5M NaCl or 1M HCl) for quite longer time than IW2 sample. This explanation obviously tells us that the corrosion/passivation rates of IW1 sample is comparatively higher/lower than that of the IW2 sample in 0.5M NaCl and 1M HCl solutions. Such variation in passivation/corrosion rates of IW1 and IW2 wire samples are acceptable because they are produced by two different well established iron industries which may follow different manufacturing principles along with variations in metallurgical operations. In summary, the more negative OCPs always signifies the more chemically reactive in the given electrolyte that leads to intensive corrosion and thus, the less passivation occurs.

Furthermore, the passivity of iron wires IW1 and IW2 in 1M NaOH solution (i.e. OCPs shifted to more noble direction) can be explained by the formation of $Fe(OH)_2$ film on the metal surface due to chemical reaction between Fe^{2+} and OH^- ions in aqueous medium: $Fe^{2+}(aq.) + OH^-(aq.) \rightarrow Fe(OH)_2$ (green layer), which does not re-dissolve in excess NaOH (aq.) solution. Growth of such film helps passivating the iron surface by creating conditions for oxygen diffusion into the metal without iron dissolution into the electrolyte. This $Fe(OH)_2$ film is composed of multiple oxide layers: Fe_2O_3 in outer layers, Fe_3O_4 in the middle layers, and FeO in the innermost layers [22]. After a certain time of immersion (~80 min.), the OCPs for IW1 and IW2 start to stabilize with time in the same alkaline solution, as shown in Figure 5(a) and Figure 5(b) respectively. This steady state is due to decrease in the reactions area ratio caused by domination of the concentration polarization on the cathode reaction side. It is also notable that the time consumed for stabilizing the OCPs also depends on the porosity percent, the geometry of the pores and defects on the surface of the working electrodes (in this case: iron wires). The detailed instrumental surface analysis of the iron wires must be studied to further understand the stability of OCPs.

From the above results and discussions, it can be said that both iron wires IW1 and IW2 show more passivation behavior in alkaline medium, less in acidic medium, and the least in neutral medium i.e. the neutral medium corrodes IW1 and IW2 iron wires more aggressively than acidic and alkaline media. Again, their corrosion and passivation behaviors in acidic medium are highly applicable to understand the spontaneous corrosion process occurring on the iron products while exposing to rain water. The rain water has a pH between five and seven, making it slightly acidic due to the effect of carbon dioxide (CO_2), sulfur oxides (SO_x), and nitrogen oxides (NO_x) and its combination with other substances in the atmosphere such as suspended soil dust etc. In daily life, exposing iron products to rain water is very common as they are used for outdoor purposes and therefore, undergoing their intensive corrosion day to day in acidic rain or in humid weather or in high-temperature has become a natural phenomenon. This uncontrolled scenario of corrosion ultimately causes wire damage, building foundation weak, engineering structures breakage/collapse, and other significant financial loss. Thus, this study is able to raise public awareness of the effect of acid rain on corroding iron and iron-based products.

Conclusions

We successfully measured the open circuit potentials (OCPs) of two different iron wires, IW1 and IW2, produced by two different iron industries in Nepal in acidic (1M HCl), alkaline (1M NaOH), and neutral (0.5M NaCl) media at 25° C, open to air as a function of immersion time. The OCPs of them are found to be shifted to more negative (or less noble) direction in acidic and neutral solutions, and to more positive (or more noble) direction in alkaline solution, indicating both of them are more passive in alkaline and then in acidic than in neutral media. The remarkable passivity of both iron wires in alkaline solution is due to the fastest growth of $Fe(OH)_2$ film having complete corrosion resistive capacity on the metal surface. In contrary, the IW1 wire sample is found to be dissolved (corroded) in aggressive media (0.5M NaCl or 1M HCl) for quite longer time than IW2 wire sample. This dissimilar

electrochemical behavior of IW1 and IW2 wires leads to their non-identical corrosion/passivation rates in the aggressive environment. The higher corrosion rate of IW1 sample is due to its inability to develop protective film earlier on its surface (unlike in the case of IW2). This delay in developing protective film on the surface of IW1 wire is either due to not containing required percentage composition of Cr (requirement is not less than 10 % Cr for fairly acting as a facilitator in developing corrosion resistive film) or containing comparatively less percentage of Cr than in IW2 wire and it is acceptable, because the IW1 and IW2 are produced by two different iron industries which might follow dissimilar metallurgical operations and manufacturing principles.

Even though we have not explicitly disclosed the name of the iron and steel industries manufacturing iron wires IW1 and IW2 due to their commercial reason, both iron wires have shown good corrosion resistive capacity in neutral solution, poor in acidic solution, and the poorest in neutral solution. It would be very much applicable and challenging research work if we could collect all the iron wire samples available in the Nepalese market. Due to the time and resource limitations, we only presented here the corrosion behavior of two mostly demandable and popular annealed iron wires produced in Nepal. We claim that the methodologies adopted in this study are equally valid to explore the electrochemical behavior of all varieties of iron/steel rods/wires/alloys in different media. This sort of research work not only puts many needful impacts in the research and development (R & D) sectors of the iron and steel industries functioning currently in the developing country like Nepal but also plays vital roles in the production as well as in consumption sides. The same type of research work and analysis in future would be indispensable to the public, and Nepalese iron and steel industries.

Acknowledgement

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