**C**ORROSION INHIBITIVE EFFECT OF METAL AND ALLOY IN ARTIFICIAL URINE IN PRESENCE OF UREA

R.Nagalakshmi\(^1\), S.Rajendran\(^2\), J.Sathiyabama\(^3\)

\(^1\)Assistant Professor, Department of Chemistry, Aarupadai Veedu Institute of Technology, Chennai-603104, Tamilnadu, India.

\(^2\)Professor, PG and Research Department of Chemistry, GTN Arts College, Dindigul-624005, Tamilnadu, India.

Research Centre RVS School of Engineering and Technology, Dindigul, Tamilnadu, India

\(^3\)Associate Professor, PG and Research Department of Chemistry, GTN Arts College, Dindigul-624 005, Tamilnadu, India.

**ABSTRACT**

Corrosion resistance of two metals namely mild steel (MS), Nickel Titanium super elastic alloy has been evaluated in artificial urine in the absence and presence of urea. Potentiodynamic polarization study and SEM analysis have been used to investigate the corrosion behaviour of these metals. Nickel Titanium super elastic alloy shows a more corrosion resistance in artificial urine in the presence of 100ppm of urea than in the 50ppm of urea and in the absence of urea. The order of corrosion resistance of metals in artificial urine, in the absence and also in the presence of urea was: Nickel Titanium super elastic alloy > Mild steel.

**KEYWORDS:** Mild steel (MS), Nickel Titanium super elastic alloy, artificial urine, urea.

**I. INTRODUCTION**

Metallic biomaterials are commonly used in reconstruction in the orthopedic and dental surgery, operative cardiology and urology. Implant alloys exhibit attractive properties such as mechanical strength and biocompatibility, corrosion resistance, safety, ductility, and wear resistance. Stainless steels, titanium alloys and cobalt alloys are commonly used as biomaterials. [1] - [7] Biocompatibility of implants in tissue environment is determined by inseparable biochemical, biomechanical and bioelectronic factors. Biological reactions are analyzed with respect to metabolic, bacteriological, immunological and oncological processes [8], [10]-[13], [15]-[17], [19] – [25], [28]. Current chemical compositions of the stainless steel (Cr-Ni-Mo) should ensure good pitting corrosion resistance and monophase austenitic structure. The austenite grain size (less than 4 acc. to ISO) and non-metallic inclusions (max. 1.5 acc. to ISO) are limited. Fine grain and low level of non-metallic inclusions ensure good mechanical properties and reduce crackability, specially in implants with small cross-sections. They also increase corrosion resistance of implants [16], [17], [23]. Great number of publications is focused on generalization of corrosion failure of implants. These analyses are focused on implants commonly used in reconstruction in the orthopedic, dental surgery, operative cardiology and urology. These implants are mainly made of austenitic stainless steel [8],[9], [12], [14], [18], [26] – [28]. Long-term research on corrosion of implants made of the mentioned steel show the complexity of corrosion processes depending on the implant form, its chemical and phase composition, surface condition, surgical procedure and implantation period [16], [17], [23]. Corrosion products infiltrate tissues. This process is called metalosis [10]. Phatomorphological changes, dependent on the type and concentration of elements, occur in tissues close to implant. Histopathological changes are observed in the detoxication organs (liver, kidneys, spleen) [16]. Therefore, corrosion tests in simulated body fluids are the basis for searching optimal fields of usage and improvement of existing solutions. In general the human urine contains urea and also excreted by the human body. In this research paper, if
the person undergone implantation in the urinary tract how the metal undergoes corrosion in the presence of excess amount of urea.

The present work was undertaken

1. To study the corrosion behavior of two metals namely Mild steel (MS), Nickel Titanium super elastic alloy in artificial urine, in the absence and presence of 50ppm and 100ppm of urea by Potentiodynamic polarization study. Corrosion parameters such as corrosion potential, corrosion current and linear polarization resistance value have been derived from these studies.

2. To analyse the protective film by SEM.

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II. EXPERIMENTAL

2.1. Materials and Methods

Two metals namely mild steel (MS), Nickel Titanium super elastic alloy were chosen for the present. The composition of mild steel was (wt %): 0.026S, 0.06P, 0.4 Mn, 0.1 C and balance iron (ArockiaSelvi et al. 2009) [29]. The composition of Ni-Ti super elastic alloy was (wt %) Ni 55.5, and balance Ti [30]. The metal specimens were encapsulated in Teflon. The surface area of the exposed metal surface was 0.0785 cm$^2$. The metal specimens were polished to mirror finish and degreased with trichloroethylene. The metal specimens were immersed in artificial urine (AU) (J. Przondziono et al, 2009) [31], whose composition was: Solution A: CaCl$_2$.H$_2$O -1.765g/l, Na$_2$SO$_4$ - 4.862g/l, MgSO$_4$.7H$_2$O - 1.462g/l, NH$_4$Cl - 4.643g/l, KCl – 12.130g/l. Solution B: NaH$_2$PO$_4$.2H$_2$O - 2.660g/l, Na$_2$HPO$_4$ - 0.869 g/l, C$_6$H$_5$Na$_3$O$_7$.2H$_2$O - 1.168 g/l, NaCl - 13.545 g/l. The pH of the solution was 6.5 (W.Kajzer et al, 2006) [5].

In electrochemical studies the metal specimens were used as working electrodes. Artificial urine (AU) was used as the electrolyte (10 ml). The temperature was maintained at 37±0.1°C. Commercially available urea was used in this study. 50ppm and 100ppm of urea was used in artificial urine.

2.2. Potentiodynamic Polarization

Polarization studies were carried out in a CHI- Electrochemical workstation with impedance, Model 660A. A three electrode cell assembly was used. The working electrode was one of the three metals. A saturated calomel electrode (SCE) was the reference electrode and platinum was the counter electrode. From the polarization study, corrosion parameter such as corrosion potential ($E_{corr}$), Corrosion current ($I_{corr}$) and Tafel slopes (anodic = $b_a$ and cathodic = $b_c$) were calculated.

2.3. Surface Examination Study

The carbon steel specimens were immersed in various test solutions for a period of 1 day. After 1 day, the specimens were taken out and dried. The nature of the film formed on the surface of the metal specimen was analyzed by surface analysis techniques.

2.4. Scanning Electron Microscopic Studies (SEM)

The carbon steel immersed in blank solution and in the inhibitor solution for a period of one day was removed, rinsed with double distilled water, dried and observed in a scanning electron microscope to examine the surface morphology. The surface morphology measurements of carbon steel were examined using JEOLMODEL6390 computer controlled scanning electron microscope.

III. RESULTS AND DISCUSSION

3.1. Polarization study

3.1.1. Corrosion resistance of mild steel in au in presence of urea
It is known to everyone that MS undergoes corrosion rapidly in AU. Hence it should not be used as implant material in body fluids. However in the presence study, the corrosion resistance of MS is chosen just compare the corrosion resistance of other metals with the corrosion resistance of MS. The polarization curves of mild steel immersed in AU in the absence and presence of urea are shown in Fig.1. The corrosion parameters namely LPR, $I_{corr}$, $E_{corr}$, Tafel slopes ($b_c =$ cathodic, $b_a =$ anodic) are given in Table.1

**TABLE.1:** Corrosion Parameters of MS Immersed in AU in Absence and Presence of Urea Obtained by Polarization Study.

<table>
<thead>
<tr>
<th>System</th>
<th>$E_{corr}$</th>
<th>$b_c$</th>
<th>$b_a$</th>
<th>LPR</th>
<th>$I_{corr}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>AU</td>
<td>-0.651</td>
<td>183</td>
<td>208</td>
<td>11415</td>
<td>3.69x10^{-6}</td>
</tr>
<tr>
<td>AU+50ppm urea</td>
<td>-0.732</td>
<td>162</td>
<td>239</td>
<td>9777</td>
<td>4.29x10^{-6}</td>
</tr>
<tr>
<td>AU+100ppm urea</td>
<td>-0.719</td>
<td>160</td>
<td>245</td>
<td>8688</td>
<td>4.85x10^{-6}</td>
</tr>
</tbody>
</table>

It is observed from Table.1 that when 50ppm of urea is added to AU, the LPR value decreases from 11415 to 9777 ohmcm$^2$. Correspondingly the corrosion current ($I_{corr}$) increases from 3.69x10$^{-6}$ to 4.29 x10$^{-6}$A/cm$^2$.When 100ppm of urea is added to AU, the LPR value further decreases to 8688 ohmcm$^2$ and the corrosion current ($I_{corr}$) increases to 4.85 x10$^{-6}$A/cm$^2$. In general it is observed that the corrosion resistance of MS in AU decreases in the presence of urea[32] – [36]. It is observed from the table that the corrosion potential shifts to cathodic side (more negative) in the presence of urea. Hence it is concluded that in presence of urea, the cathodic reaction is controlled predominantly.

**Fig.1.** Polarization curves of MS in various test solutions. 

a) AU  b) AU+ 50ppm of urea  c) AU+ 100ppm of urea

### 3.1.2. Corrosion Resistance of Ni-Ti Superelastic in AU in Presence of Urea:

The polarization curves of Ni-Ti superelastic alloy immersed in AU in the absence and presence of urea are shown in Fig.2. The corrosion parameters namely LPR, $I_{corr}$, $E_{corr}$, Tafel slopes ($b_c =$ cathodic, $b_a =$ anodic) are given in Table.2

**TABLE.2:** Corrosion Parameters of Ni-Ti Superelastic Alloy Immersed in AU in Absence and Presence of Urea Obtained by Polarization Study.

<table>
<thead>
<tr>
<th>System</th>
<th>$E_{corr}$</th>
<th>$b_c$</th>
<th>$b_a$</th>
<th>LPR</th>
<th>$I_{corr}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>AU</td>
<td>-0.432</td>
<td>124</td>
<td>208</td>
<td>1.84x10^{-9}</td>
<td>1.84x10^{-9}</td>
</tr>
<tr>
<td>AU+50ppm urea</td>
<td>-0.451</td>
<td>126</td>
<td>195</td>
<td>1.21x10^{-9}</td>
<td>2.76x10^{-9}</td>
</tr>
<tr>
<td>AU+100ppm urea</td>
<td>-0.415</td>
<td>127</td>
<td>190</td>
<td>1.17x10^{-9}</td>
<td>2.82x10^{-9}</td>
</tr>
</tbody>
</table>
It is observed from Table 2 that when 50ppm of urea is added to AU, the LPR value decreases from $1.84 \times 10^7$ to $1.20 \times 10^7 \text{ohmcm}^2$. Correspondingly the corrosion current ($I_{corr}$) increases from $1.84 \times 10^{-9}$ to $2.76 \times 10^{-9} \text{A/cm}^2$. When 100ppm of urea is added to AU, the LPR value further decreases to $1.17 \times 10^7 \text{ohmcm}^2$ and the corrosion current ($I_{corr}$) increases to $2.82 \times 10^{-9} \text{A/cm}^2$. In general it is observed that the corrosion resistance of Ni-Ti superelastic alloy in AU decreases in the presence of 50ppm of urea but in 100ppm of urea slightly decreases. It is observed from the table that the corrosion potential shifts to cathodic side (more negative) in the presence of urea. Hence it is concluded that in presence of urea, the cathodic reaction is not controlled predominantly. [32] – [36]. These data reveals that the corrosion resistance is increased as the quantity of urea increased in artificial urine.

**Fig.2.** Polarization curves of Ni-Ti Super elastic in various test solution.
- a) AU
- b) AU+ 50ppm of urea
- c) AU+ 100ppm of urea

### 3.2. Scanning Electron Microscopic Studies (SEM)

#### 3.2.1. SEM Analysis of Mild steel Surface:

SEM provides a pictorial representation of the surface. To understand the nature of the surface film in the absence and presence of inhibitors and the extent of corrosion of Mild steel, the SEM micrographs of the surface are examined. The SEM micrographs (X 5000, X 10000, X 50000) of polished Mild steel surface (control) in **Fig. 3. (a, b, c)** shows the smooth surface of the metal. This shows the absence of any corrosion products or inhibitor complex formed on the metal surface. The SEM micrographs (X 5000, X 10000, X 50000) of Mild steel specimen immersed in the AU media for one day in the absence and presence of inhibitor system are shown in **Fig.3.(d, e, f)** and **Fig.3.(g, h, i)** respectively. The SEM micrographs of Mild steel surface immersed in AU media in **Fig.3. (d, e, f)** shows the roughness of the metal surface which indicates the corrosion of Mild steel in AU media. **Fig.3. (g, h, i)** indicates that in the presence of 100 ppm urea in AU media, the surface coverage increases which in turn results in the formation of insoluble complex on the surface of the metal. In the presence of urea, the surface is covered by a thin layer of inhibitors which effectively control the dissolution of Mild steel.37, 38

**Fig.6(a, b, c):** SEM micrographs of Mild steel (control)

- (a) Mild Steel immersed control Magnification X-5000
- (b) Magnification X-10,000
- (c) Magnification X-50,000
(h) Mild Steel immersed control Magnification X-10000
(i) Mild Steel immersed control Magnification X-50000

Fig .3(d, e, f): SEM micrographs of Mild Steel immersed in AU

(g) Mild Steel immersed in AU Magnification X-5000
(h) Mild Steel immersed in AU Magnification X-10000
(i) Mild Steel immersed in AU Magnification X-50000

Fig .3(g, h, i): SEM micrographs of Mild Steel immersed in AU containing urea

(g) Mild Steel immersed in AU containing urea Magnification X-5000
(h) Mild Steel immersed in AU containing urea Magnification X-10000
(i) Mild Steel immersed in AU containing urea Magnification X-50000

3.2.1. SEM Analysis of NiTi super elastic alloy Surface:

SEM provides a pictorial representation of the surface. To understand the nature of the surface film in the absence and presence of inhibitors and the extent of corrosion of NiTi super elastic alloy, the SEM micrographs of the surface are examined. The SEM micrographs ((X 5000, X 10000, X 50000) of polished NiTi super elastic alloy surface (control) in Fig. 6. (a, b, c) shows the smooth surface of the metal. This shows the absence of any corrosion products or inhibitor complex formed on the metal surface. The SEM micrographs (X 5000, X 10000, X 50000) of NiTi super elastic alloy specimen immersed in the AU media for one day in the absence and presence of inhibitor system are shown in Fig.6. (d, e, f) and Fig.6. (g, h, i) respectively. The SEM micrographs of NiTi super elastic alloy surface immersed in AU media in Fig.6. (d, e, f) shows the roughness of the metal surface which indicates the corrosion of NiTi super elastic alloy in marine media. Fig.6. (g, h, i) indicates that in the presence of 100 ppm urea in AU media, the surface coverage increases which in turn results in the formation of insoluble complex on the surface of the metal. In the presence of urea, the surface is covered by a thin layer of inhibitors which effectively control the dissolution of NiTi super elastic alloy.32, 33, 37, 38
IV. CONCLUSION

The corrosion behavior of two metals namely mild steel (MS), Nickel Titanium super elastic alloy have been studied in artificial urine in the absence and presence of urea. AC impedance spectra have led to the following conclusions: In the absence of urea the order of corrosion resistance was: Ni-Ti super elastic > Mild steel. In the presence of 50ppm urea, the order of corrosion resistance was: Ni-Ti super elastic > Mild steel. In the presence of 100ppm urea, the order of corrosion resistance was: Ni-Ti super elastic > Mild steel. Ni-Ti super elastic was more corrosion resistant in the absence of urea than in the presence of urea. Mild steel was less corrosion resistant in the presence of urea and in the...
absence of urea. The SEM micrographs confirm the formation of protective layer on the metal surface. The formation of protective layer on the metal surface was more in NiTi super elastic alloy than mild steel. It reveals that NiTi super elastic alloy is best suited for implantation.

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