



# Effect of Fluoride Concentration and pH on Corrosion Behavior of Ti–15Mo in Artificial Saliva

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## Abstract

Corrosion behavior of Ti–15Mo alloy in artificial saliva containing variation in pH (7.2, 3.5) and varying concentrations of fluoride ions (2500, 5000 and 10,000 ppm) was evaluated using potentiodynamic polarization, electrochemical impedance spectroscopy studies to ascertain its suitability for dental implant applications. The study reveals that there is a strong dependence of the corrosion resistance of Ti–15Mo alloy on the concentration of fluoride ions in the electrolyte medium. Surface morphological characterization was carried out using SEM–EDAX to investigate the corrosion resistance in artificial saliva containing sodium fluoride at various pH conditions, and it was found that the surface roughness of the specimens was highly increased at pH 3.5. In spite of the active dissolution, the Ti–15Mo alloy exhibits passivity at anodic potentials at all concentrations of the fluoride ions studied. The results suggest that Ti–15Mo alloy can be a suitable alternative for dental implant applications.

**Keywords** Ti–15Mo alloy · Corrosion resistance · Fluoride · Dental implant · pH · Artificial saliva

## 1 Introduction

In dentistry, metallic materials are used as implants in reconstructive oral surgery to replace a single or an array of teeth in the fabrication of dental prosthesis such as metal plates for complete and partial dentures, essentially inpatients requiring hypoallergenic materials. Nowadays, titanium and its alloys have been widely used for dental restorative applications such as crown, bridge, framework and dentures. These alloys are the most commonly used metal for dental implants and prosthesis owing to its excellent mechanical properties, good corrosion resistance and good adhesion, in biological fluids with good biocompatibility [1].

The stability of titanium under corrosion conditions is essentially due to the formation of a stable and tightly adherent thin protective oxide layer on its surface, and the passive film stability depends on its structure and composition. For instance, pH is known to have a strong influence on the

corrosion resistance of titanium and its alloys. For a metal to be used in oral environment, it should be biocompatible and have high corrosion resistance. Among the various types of titanium alloys, Ti–6Al–4V has become the metals of choice for endosseous parts of presently available dental implants [1–3]. Since the oral environment could involve fluoride medium, the degree of corrosion resistance offered by the Ti alloys in fluoride-containing medium becomes an important criterion in the selection of a metallic biomaterial to be used in the oral medium.

Titanium alloys developed in the early stage are mainly  $\alpha + \beta$ -type ones. In general, the biocompatibility is an important criterion for the selection of any biomaterial. Therefore, the research and development on  $\beta$ -type titanium alloys, which are considered to be advantageous in terms of biocompatibility, are increasing [4, 5]. Several  $\beta$ -phase Ti alloys, having Nb, Ta, Zr and Mo as alloying elements ( $\beta$ -stabilizer elements), were developed. Nag et al. [6, 7] have recommended it as one of the promising biocompatible Ti alloys. Daniel et al. [8] have investigated the corrosion behavior of  $\beta$ -Ti20Mo alloy in artificial saliva, while Ho et al. [9] and Oliveira et al. [10], have studied the structure and properties of a series of binary Ti–Mo alloy with Mo content ranging up to 20 wt% [11].

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The biocompatibility of Ti and its alloys for the dental implant is decided based upon the osseointegration response and cell adhesion behavior. The contact between the metallic implant and the receiving living tissues is made through the oxide layer on the implant surface, which allows the osseointegration process [12]. However, corrosion of titanium and its alloys can still occur in some clinical circumstances. The corrosion resistance of dental alloys in an acidic oral environment is surely a crucial subject when biocompatibility is of concern.

Titanium and its alloys are used in the dental field, and the oral environment is exposed to fluoride-containing medium which becomes an important property for their use in dentistry. On the other hand, when the fluoride-containing mouth rinse, toothpaste or prophylactic gel (NaF: around 0.1–2%) are used in the oral environment where proteins are naturally present in the saliva, the corrosion or electrochemical behavior of dental alloys in the oral environment with fluoride ions and proteins should be taken into account. In addition, some reports have shown the negative influence of fluoride on the corrosion resistance of titanium alloys [13, 14].

Reclaru et al. [14] have studied the effect of fluorides on titanium and other dental alloys for dentistry applications. The results revealed the development of oxide layer with one or more fluoride ion containing electrolytes. Al-Mobarak et al. [15] have investigated the effect of hydrogen peroxide on the electrochemical behavior of Ti and its alloys for dental applications and found that the presence of  $H_2O_2$  leads to decreased corrosion resistance of Ti and its alloys and an enhanced dissolution/oxidation rate and the effect of  $H_2O_2$  depends on its concentration as well as pH of the solution.

Nakagawa et al. [16] have examined the effect of fluoride concentration and pH on corrosion behavior of titanium, and the results revealed that the fluoride concentrations and pH values on corrosion environments where the fluoride concentration and pH value are known. Nakagawa et al. [17] have also studied the corrosion behavior of pure titanium and titanium alloys in fluoride-containing solutions. The results proved that the corrosion resistance of Ti–0.2Pd alloy was greater than Cp–Ti, Ti–6Al–4V and Ti–6Al–7Nb alloys in the wide range of pH and fluoride concentrations. Nakagawa et al. [18] have also investigated the effect of fluoride and dissolved oxygen concentrations on the corrosion behavior of pure titanium and titanium alloys.

Huang [19] examined the effects of fluoride and albumin concentration on the corrosion behavior of Ti–6Al–4V alloy

and reported that the protectiveness of the  $TiO_2$  passive film formed on Ti–6Al–4V alloy was destroyed by fluoride ions via the formation of  $Na_2TiF_6$  when the NaF concentration was increased up to 0.1%. Various researchers have also reported on the corrosion behavior of influence of fluoride ion on the Cp–Ti and its alloys for dental implant applications [20–29].

The objective of this present investigation is to study the interaction of Ti–15Mo alloy in artificial saliva with a high concentration of fluoride ions in simulated oral environments with varying fluoride concentrations at various pH conditions to improve the bioactivity. Surface morphological studies were carried out using scanning electron microscopy to evaluate the surface roughness at various pH conditions.

## 2 Materials and Methods

### 2.1 Specimen Preparation

Ti–15Mo alloy (Kobe Steel Ltd., Japan) was used in the present study with the size of dimension  $1 \times 1 \text{ cm}^2$ , and its chemical composition is presented in Table 1. The specimens were ground using abrasive SiC paper from 600 to 1200# grade. Final polishing was done using alumina powder (0.5  $\mu\text{m}$  in size) in order to produce a mirror finish surface followed by rinsing with distilled water and degreased with acetone. Further the specimens were ultrasonicated in acetone for about 20 min. Finally the specimens were washed in distilled water and dried in an oven.

### 2.2 Test Electrolyte Media

For all the experiments, AR-grade chemicals/reagents were used and solutions were prepared in double-distilled water. The testing medium and the electrolyte solution were artificial saliva [15], and its chemical composition is presented in Table 2. Electrolyte medium used was the artificial saliva enriched with sodium fluoride (NaF) with different concentrations of 2500, 5000 and 10,000 ppm. This particular concentrations of the artificial saliva were prepared similar to that of the fluoride concentrations obtained in a commercial fluoride-containing mouth rinse and toothpaste. The pH (7.2, 3.5) was lowered by adding lactic acid. The pH was measured with an XC601-type glass electrode (Radiometer Analytical, Villeurbanne, France) connected to a PHM 220-type

**Table 1** Chemical composition of Ti–15Mo alloy in (wt%)

Alloy	Main alloying elements (wt%)						
	C	N	O	H	Fe	Mo	Ti
Ti–15Mo	0.02	0.01	0.10	0.01	0.01	15.04	Bal.

**Table 2** Chemical composition of artificial saliva

S. no.	Reagents used	Grams in 1000 mL
1	$\text{KH}_2\text{PO}_4$	0.340
2	$\text{Na}_2\text{HPO}_4 \cdot 2\text{H}_2\text{O}$	0.445
3	$\text{KHCO}_3$	1.500
4	$\text{NaCl}$	0.585
5	$\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$	0.030
6	$\text{CaCl}_2$	0.016
7	Citric acid	0.031
8	$\text{NaF}$	2500, 5000, 1000 (ppm)
9	Lactic acid	For adjusting pH (3.5 and 7.2)

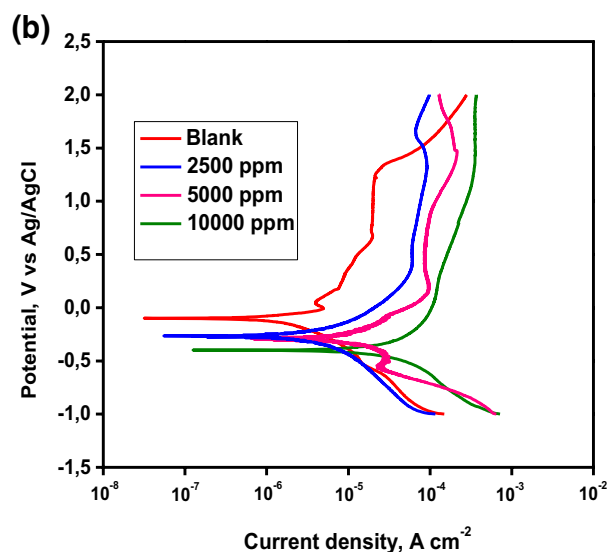
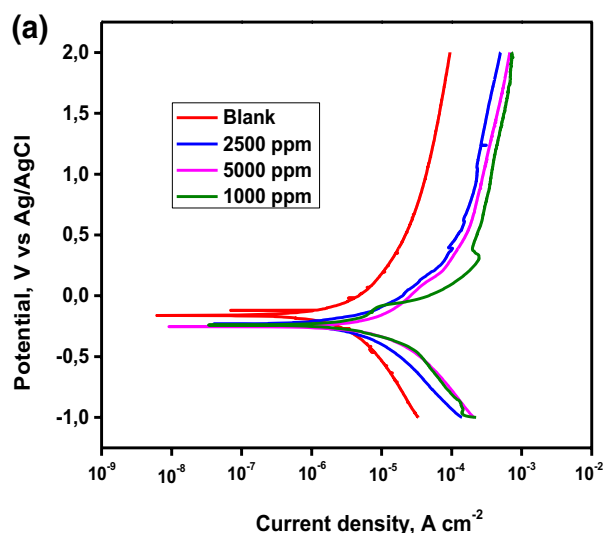
pH meter. The temperature was maintained at  $37 \pm 1$  °C for all the experiments.

### 2.3 Electrochemical Characterization

Electrochemical experiments were performed using a conventional three electrode cell assembly with potentiostat (model PGSTAT 12 with FRA, Autolab, the Netherlands B.V.) controlled by a personnel computer with dedicated software; viz., General Purpose Electrochemical System (GPES version 6.0) was used for conducting the polarization experiments. A saturated  $\text{Ag}/\text{AgCl}$  electrode served as a reference electrode, platinum sheet acted as a counter electrode and the test specimens ( $\text{Ti}-15\text{Mo}$  alloy) acted as the working electrode with the exposed area of  $1 \text{ cm}^2$ . Potentiodynamic polarization studies were carried out in the potential range of  $-1$  to  $2 \text{ V}$  at a scan rate of  $1 \text{ mV/s}$  in an aerated medium. Electrochemical impedance spectroscopic studies (EIS) were carried out using an electrochemical system frequency response analyzer (FRA), and the impedance spectra were acquired in the frequency range of  $10^4$  to  $10^{-2} \text{ Hz}$  with a  $10 \text{ mV}$  amplitude sine wave generated by FRA. The impedance spectra (Bode plots) were fitted using a nonlinear least square (NLLS) method. In order to obtain reliable results, all the experiments were triplicated for concordant values.

### 2.4 Surface Morphological Characterization

Surface morphology and elemental composition of the  $\text{Ti}-15\text{Mo}$  specimens were examined using (Hitachi model S-3400) scanning electron microscope (SEM) coupled with an energy-dispersive X-ray analysis system (EDAX).



**Fig. 1** Potentiodynamic polarization curves of  $\text{Ti}-15\text{Mo}$  alloy in artificial saliva containing various concentrations of fluoride at a pH 7.2, b pH 3.5

## 3 Results and Discussion

### 3.1 Electrochemical Characterization

#### 3.1.1 Potentiodynamic Polarization Results (PDP)

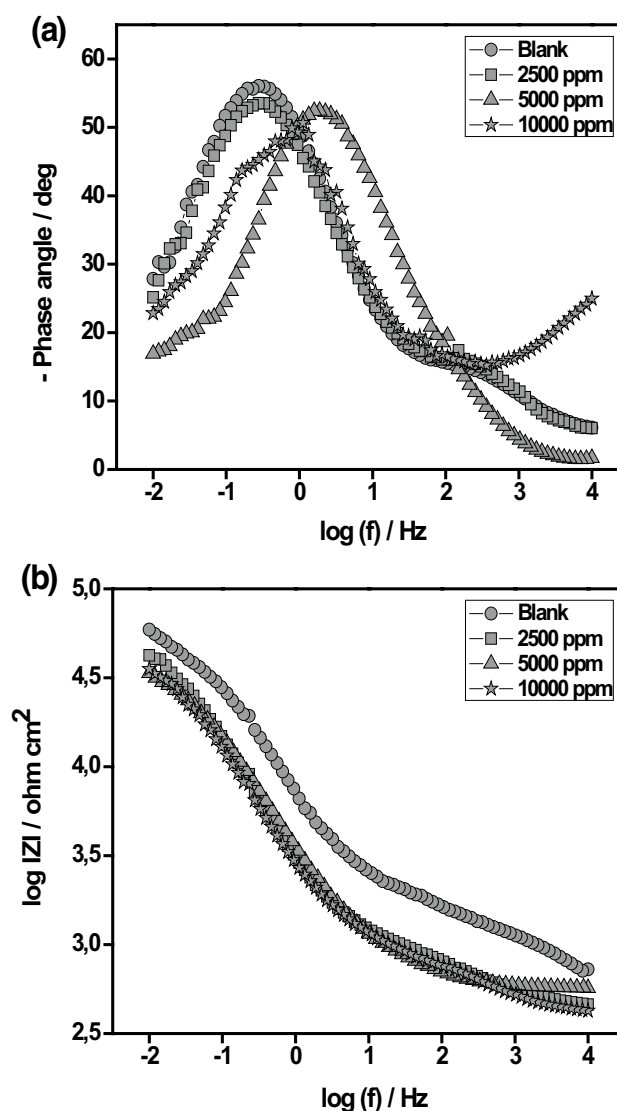
Potentiodynamic polarization studies of  $\text{Ti}-15\text{Mo}$  were conducted in the potential range of  $-1$  to  $2 \text{ V}$  at a scan rate of  $1 \text{ mV/s}$  to observe the effect of fluoride ions at various pH conditions. Figure 1 shows the polarization curves of  $\text{Ti}-15\text{Mo}$  in artificial saliva containing different fluoride concentrations (2500, 5000 and 10,000 ppm) at pH (7.2, 3.5), respectively. It can be observed that there is a cathodic shift in corrosion potential range ( $E_{\text{corr}}$ ) from  $-0.107$  to

– 0.398 V vs. SCE with increase in fluoride ion concentration from 0 to 10,000 ppm. Also the shape of the curves is quite similar; the active region of the curves is extended to higher current region in the presence of fluoride ions. For a given pH value, the corrosion current density of Ti–15Mo increases with increase in fluoride concentration and for a given fluoride concentration, and pH decrease leads to an increase in the corrosion current density. The shift in the active region of the polarization curves toward higher current region suggests the negative influence of fluoride ions on the corrosion resistance of Ti–15Mo alloy. The negative influence of fluoride ions has been confirmed by various researchers [13–20]. Nakagawa et al. [16] have also reported such occurrence at 30 ppm NaF. Hence, the increase in the active region in the presence of fluoride ions is due to the formation of a porous defective oxide layer which reduces the corrosion protection. Huang et al. [19] evaluated the active–passive transition of Ti–6Al–4V alloy in acidic artificial saliva when the NaF concentration is 0.5%. Kwon et al. [20] have also evaluated the effect of acidic fluoride solution on  $\beta$ -titanium alloy wire. Oshida et al. [21] have reported that commercially available fluoride treatment agent cause discoloration of Ti–6Al–4V alloy. Schutz and Thomas [22] evaluated that 20 ppm of NaF may destroy the protective oxide layer on Cp–Ti.

The anodic region of the polarization curve (Fig. 1b) exhibits an active–passivation transition observed for pH 3.5. The active–passive transition observed in the anodic region both in the absence and in the presence of fluoride ions suggested that the presence of fluoride ions in artificial saliva did not hinder the formation of passive oxide film on the surface of Ti–15Mo alloy. It can be identified that for pH 3.5, the  $E_{\text{corr}}$  shifts in the negative direction, the fluoride ions interfere in the titanium oxide formation which causes the changes in the protective passive layer of the metal [23, 24]. As the NaF concentration is increased from 2500 to 10,000 ppm, the higher current density was obtained due to the dissolution of protective oxide film and the influence of fluoride ions may affect the properties of the oxide layer, causing the  $E_{\text{corr}}$  to shift in the negative direction. Hence, the lower pH can enhance the activity of fluorine ions in the electrolyte medium and significantly decreased the corrosion resistance of the oxide film [25].

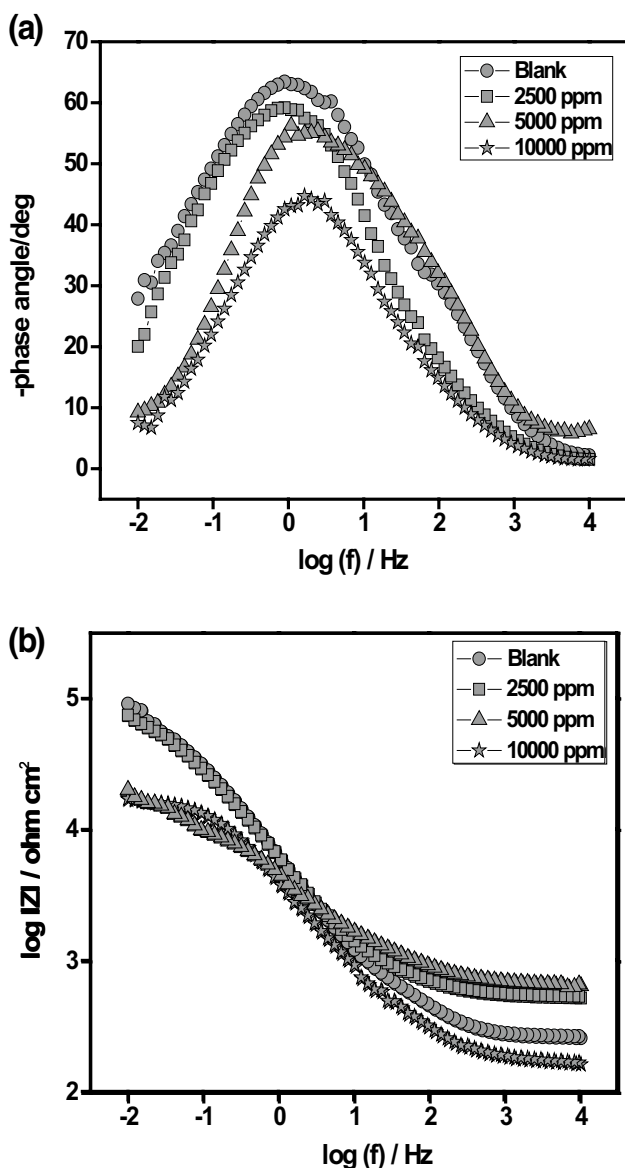
### 3.1.2 Electrochemical Impedance Spectroscopic (EIS) Studies

EIS studies of Ti–15Mo were performed in artificial saliva for various fluoride concentration/pH conditions, in order to observe the influence of both parameters with the impedance response. Figures 2 and 3 show the Bode phase angle and Bode impedance plots for Ti–15Mo in artificial saliva containing (2500, 5000 and 10,000 ppm) at pH (7.2, 3.5),



**Fig. 2** Bode phase angle and Bode impedance plots of Ti–15Mo alloy in artificial saliva containing various concentrations of fluoride at pH 7.2

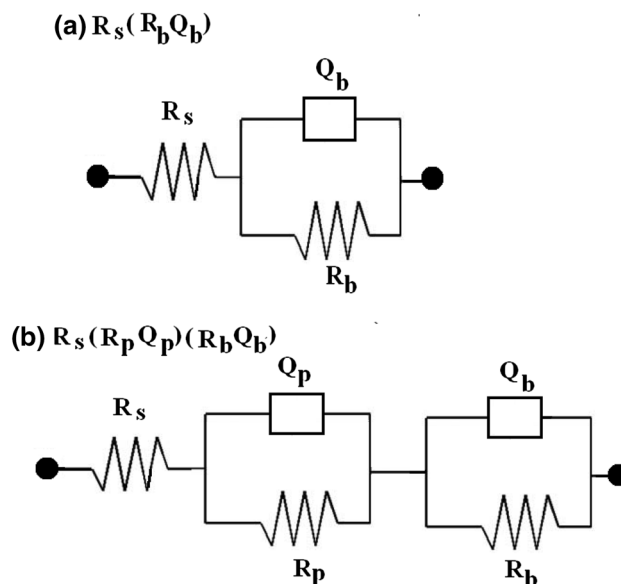
respectively. It can be noted that the impedance values measured for the various concentrations of NaF leads to an active state of Ti with a reduction in corrosion resistance of Ti–15Mo. As can be seen that, the increase in fluoride concentration and a decrease in pH, reflects the change of phase angle behavior suggested the stable passive film formation on Ti–15Mo [25]. At higher fluoride concentration (10,000 ppm), the phase angle values shifts to decrease in its values which may be due to the dissolution of passive oxide film formation consisting oxides of titanium and molybdenum. Satendra et al. [26, 27] evaluated that the passive oxide film formed above 2500 ppm of fluoride ions may not be stable for a longer time and starts dissolving due to the negative effect of higher concentration of fluoride ions. It can



**Fig. 3** Bode phase angle and Bode impedance plots of Ti–15Mo alloy in artificial saliva containing various concentrations of fluoride at pH 3.5

also be observed that the width of the phase angle decreased significantly with the increase in the fluoride concentration. The resistance value of the passive film was initially high, and then, it decreased with decrease in pH values which may be due to the dissolution of barrier layer formed on the metal surface. Further the phase angle value drops slightly toward the lower values in the low-frequency range indicating the contribution of surface film resistance to the impedance, i.e., the resistance of the surface film decreases [28].

The results obtained were in good agreement with the impedance values obtained on fitting curve using the non-linear least square fit developed by boukamp. Figure 4 shows



**Fig. 4** Equivalent circuit diagrams fitted for Ti–15Mo alloy in artificial saliva. **a** Blank. **b** Fluoride concentrations/pH conditions

the equivalent circuit diagram used to evaluate the electrochemical impedance parameters for Ti–15Mo alloy in artificial saliva. The proposed equivalent circuit model  $R_s(R_bQ_b)$  was used to fit the spectra obtained for Ti–15Mo alloy for the blank (Fig. 4a) where  $R_s$  represents the ohmic or solution resistance, and  $R_b$  and  $Q_b$  represent the charge transfer resistance and double-layer capacitance of the barrier layer. This represents the presence of a single layer on the metal surface possessing resistance as well as capacitance. The surface film is considered to be a parallel circuit of a resistor due to the ionic conduction through the film and a capacitor due to its dielectric properties [29, 30]. The equivalent circuit proposed for the Ti–15Mo alloy with the variation of pH and fluoride concentrations is  $R_s(R_pQ_p)(R_bQ_b)$  (as shown in Fig. 4b) where  $R_p$  and  $Q_p$  represents the charge transfer resistance and double-layer capacitance of the porous layer and is characterized by two parallel combination of resistance and capacitance in series with the solution resistance. It indicated the formation of two layers, viz. inner barrier and outer porous layers, respectively [31].

The electrochemical impedance parameters by fitting with the equivalent circuit in artificial saliva with different fluoride concentrations at various pH values are shown in Table 3. It was observed that the resistance of the barrier film decreased with increase in concentration of fluoride and the variation of pH, whereas the capacitance increased. The  $R_p$  and  $Q_p$  values were determined from the Bode plots which are presented in Table 4. The changes in the charge transfer resistance and double-layer capacitance of the porous layer of Ti–15Mo in artificial saliva with varying fluoride and pH concentrations are presented in Fig. 5. It can be observed

**Table 3** Electrochemical impedance parameters of Ti–15Mo alloy by fitting with  $R_s(R_bQ_b)$  and  $R_s(R_pQ_p)$  ( $R_bQ_b$ ) in artificial saliva with various fluoride concentrations/pH conditions

Concentration of F (in ppm)	$R_s$ (k $\Omega$ cm $^2$ )	$R_p$ (k $\Omega$ cm $^2$ )	$Q_p$ ( $\mu$ F cm $^{-2}$ )	$n$	$R_b$ (k $\Omega$ cm $^2$ )	$Q_b$ ( $\mu$ F cm $^{-2}$ )	$n$
<i>pH 7.2</i>							
Blank	1.581	–	–	–	4773	6.37	0.87
2500	0.786	4632	4.64	0.79	2669	7.27	0.85
5000	0.653	4512	4.81	0.82	2786	7.58	0.84
10,000	0.584	4678	5.27	0.80	2678	7.92	0.82
<i>pH 3.5</i>							
Blank	1.581	–	–	–	4773	6.37	0.87
2500	0.622	4878	3.26	0.71	2725	5.28	0.74
5000	0.492	4286	3.58	0.69	2878	6.42	0.73
10,000	0.230	4184	4.23	0.68	2214	6.94	0.69

**Table 4** Charge transfer resistance obtained from impedance measurements for various fluoride concentrations/pH conditions

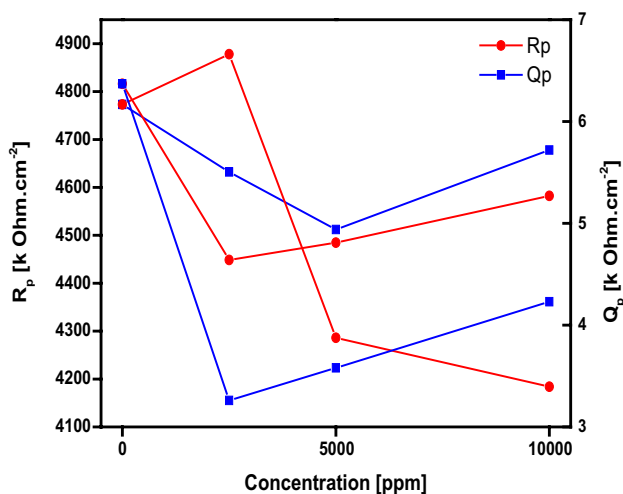
Alloy	Concentration of F (in ppm)	$R_p$ (k $\Omega$ cm $^2$ )	$Q_p$ ( $\mu$ F cm $^{-2}$ )
<i>pH 7.2</i>			
Ti–15Mo	Blank	4773	6.37
	2500	4632	4.64
	5000	4512	4.81
	10,000	4678	5.27
<i>pH 3.5</i>			
Ti–15Mo	Blank	4773	6.37
	2500	4878	3.26
	5000	4286	3.58
	10,000	4184	4.23

that the  $R_p$  values decreased from 4773 to 4632 k $\Omega$  cm $^2$  for pH 7.2 and from 4773 to 4184 k $\Omega$  cm $^2$  for pH 3.5, whereas the  $Q_p$  values increased from 4.64 to 5.27  $\mu$ F cm $^{-2}$  for pH 7.2 and from 3.26 to 4.23  $\mu$ F cm $^{-2}$  for pH 3.5, respectively, with increase in fluoride ion concentration from 0 to 10,000 ppm.

As from the electrochemical results, it can be inferred that the active–passive transition is observed in the presence of all concentrations of fluoride ions at different pH values. In spite of the active dissolution in the presence of fluoride ions, the Ti–15Mo exhibits passivity at anodic potentials. Increase in fluoride ion concentration increases the  $i_{corr}$ ,  $Q_p$  and  $Q_b$  values, and causes a cathodic shift in  $E_{corr}$  and a decrease in  $R_p$  and  $R_b$  values, suggesting the negative influence of fluoride ion and a decrease in corrosion protective ability of Ti–15Mo alloy.

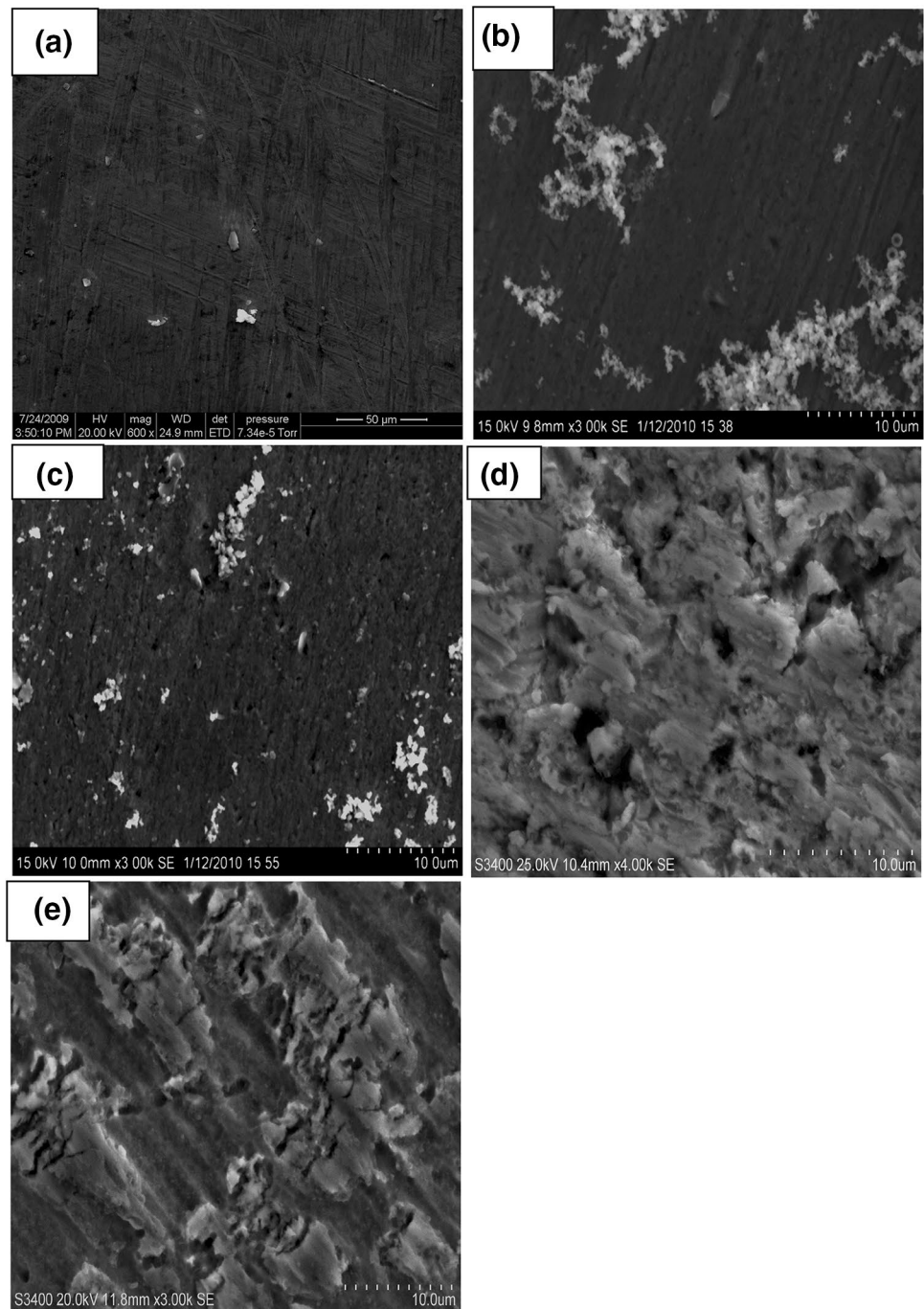
### 3.2 Surface Morphological Characterization

Surface morphological behavior on Ti–15Mo in artificial saliva with varying fluoride concentrations at various pH conditions was examined, and the obtained images are presented in Fig. 6. The surface morphology for the blank surface (BARE) at pH 7.2 showed a smooth uniform surface (Fig. 6a). A smooth surface with white crystal deposits (Fig. 6b) was observed for the 2500 ppm of fluoride concentration on Ti–15Mo alloy at pH 7.2. When there is an increase in the concentration of fluoride ions of 5000 ppm, a rough surface of white particles with small blisters (Fig. 6c) was exhibited due to the fluoride ions attack over the surface of Ti–15Mo. The aggressive actions of fluoride anions are hindered when a thick oxide layer is formed after long exposure period with a very rough surface of pores (Fig. 6d) was observed for 2500 ppm of fluoride at pH 3.5 and (Fig. 6e) for 5000 ppm of fluoride at pH 3.5 which evinced that the rate of fluoride ions can cause more corrosive attack on Ti–15Mo surfaces.



**Fig. 5** Changes in charge transfer resistance and double-layer capacitance of Ti–15Mo alloy in artificial saliva containing various fluoride concentrations/pH conditions

**Fig. 6** SEM Micrographs of Ti–15Mo alloy in artificial saliva containing various concentrations of fluoride. **a** Blank, **b** 2500 ppm at pH 7.2, **c** 5000 ppm at pH 7.2, **d** 2500 ppm at pH 3.5, **e** 5000 ppm at pH 3.5



## 4 Conclusions

Corrosion behavior of Ti–15Mo alloy in artificial saliva by varying with fluoride ions and pH conditions was evaluated. Electrochemical results revealed that the increase in fluoride ion concentration increases the  $i_{\text{corr}}$ ,  $Q_p$  and  $Q_b$  values, which causes a cathodic shift in  $E_{\text{corr}}$  and a decrease in  $R_p$  and  $R_b$  values, suggesting the negative influence of fluoride

ion to decrease the corrosion protective ability of surface of Ti–15Mo. Morphological studies exhibited a smooth uniform surface for blank, whereas a rough surface with blisters, indicates the increase in surface roughness of the oxide layer was observed at acidic condition (pH 3.5). The surface also gets damaged due to the increase of the fluoride ions. Hence, based on the results obtained, Ti–15Mo alloy can be used for dental implants.

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