

Impact of Fluoride on the Corrosion Pattern of Pure Titanium and Titanium Alloy Combined with Nickel-Chromium Alloy and Gold Alloy: An In-vitro Study

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ABSTRACT

Introduction: Currently, Fluoride ions are added to commercially available tooth paste, mouth washes and cariostatic gels for their prophylactic action. However, the preventive effect may also be accompanied by the corrosive activity, through infiltration of fluoride-containing saliva in the contact between crowns and bridges or into the implant supported structure.

Aim: To evaluate the corrosion action of Sodium Fluoride (NaF) on Pure Titanium (PT) and its alloy coupled with Nickel-Chromium (Ni-Cr) alloy (NC) and Gold (Au) Alloy (GA).

Materials and Methods: This in-vitro study was conducted in the Department of Prosthodontics at Central Electrochemical Research Institute, Karaikudi, Tamil Nadu, India. The duration of the study was six months, from April 2010 to September 2010. A total of 48 specimens were categorised under four groups. The four groups included for the study were pure group I (PT+GA), group II (PT+NC alloy), group III {Titanium Alloy (TA)+GA} and group IV (TA+NC alloy). The specimens under each group were divided into half and were immersed in 100 mL of either artificial saliva or artificial saliva with 1000 Parts Per Million (ppm) fluoride. The current was passed at a scanning rate of 1800 mV/hour for

60 minutes and changes in corrosion potential were observed. The elemental release analysis test was conducted by using inductively coupled plasma-mass spectrometry to quantitatively analyse the elements (metal ions) released in the test solution from the galvanic coupling alloys. In order to evaluate the corrosion behaviour, the surfaces of the specimens were examined with an optical microscope (Eclipse LV100D, Nikon, USA). Analysis of Variance (ANOVA) was done for intergroup comparison. Statistical significance was set at 5%.

Results: The mean value of corrosion of PT+GA and PT+NC was 13 $\mu\text{g}/\text{cm}^2$ and 27 $\mu\text{g}/\text{cm}^2$ respectively. The mean value of corrosion of TA+GA and TA+NC was 12 $\mu\text{g}/\text{cm}^2$ and 24 $\mu\text{g}/\text{cm}^2$ respectively, in artificial saliva without sodium fluoride. In the presence of sodium fluoride, the mean value of corrosion of PT+GA and PT+NC was 17 $\mu\text{g}/\text{cm}^2$ and 60 $\mu\text{g}/\text{cm}^2$ respectively, and the mean value of corrosion of TA+GA and TA+NC was 15 $\mu\text{g}/\text{cm}^2$ and 41 $\mu\text{g}/\text{cm}^2$. The PT and TA specimens coupled with NC alloy specimens showed more corrosion in saliva either with or without sodium fluoride compared to GA.

Conclusion: The NC alloy specimens coupled with PT and TA specimens showed severe pitting corrosion in artificial saliva containing sodium fluoride.

Keywords: Electrolyte, Elemental release, Sodium fluoride, Titanium dioxide

INTRODUCTION

Titanium and TA (Ti6Al4V) are known for its excellent corrosion resistance and biocompatibility. The ability of titanium metal to form a passive layer of Titanium dioxide (TiO_2) on exposure to air is the reason for its superior corrosion resistance. Due to its superior corrosion resistance, mechanical characteristics, and biocompatibility, PT and TAs are frequently utilised to create dental implants and orthodontic wires [1]. In the mouth, the crown (or) superstructure placed over implant is usually made up of GA or NC alloy [2]. The fluoride ion content of commercially fluoridated tooth paste, mouth washes (or) cariostatic gels is between 1000-10000 ppm [3]. Yet, this strong anion's preventive effect could be accompanied by the corrosion of metallic dental components. Corrosive attack could occur if fluoride containing saliva gets within the implant supported structure or if crowns and bridges come into contact with it [4].

Previously, studies have been done to investigate the corrosion resistance of GA and TA [5-9]. However, there are no studies have been conducted, comparing the corrosive resistance of PT and TA, when coupled with GA and NC alloy. Therefore, the present study was performed to evaluate effect of fluoride ions on the corrosion behaviour, surface roughness and elemental release of PT and TA, when coupled with GA and NC alloy.

MATERIALS AND METHODS

The present in-vitro study was conducted in the Department of Prosthodontics at Central Electrochemical Research Institute, Karaikudi, Tamil Nadu, India. The duration of the study was six months, from April 2010 to September 2010. The Ethical clearance was obtained from Rajas Dental College and Hospital (EC no: RDCH/EC/02/10).

Study Procedure

A total of 48 specimens were categorised under four groups. The four groups included for the study were pure group I (PT+GA), group II (PT+NC alloy), group III (TA+GA) and group IV (TA+NC alloy). The composition of the specimens, are mentioned in [Table/Fig-1]. The 10x10x2 mm specimens were cut from the source metal, smoothed with silicon carbide paper Nos. 400 through Nos. 120 in sequence, degreased with trichloroethylene, and then washed with deionised water. The specimens under each group were divided into half and were immersed in 100 mL of either artificial saliva or artificial saliva with 1000 ppm fluoride contained in a beaker. The electrodes used and the different systems are mentioned in [Table/Fig-2].

The test solution (electrolyte) used was Fusayama-Meyer's artificial saliva [1] and artificial saliva with 1000 ppm of NaF (Lobachemie, India). The pH of the artificial saliva was 5.3. The pH was measured with pH meter (pH ep, Hanna, Singapore). The pH of artificial saliva

Material	Composition	Supplier
Pure titanium	99.2% metal basis	Alfa Aesar, U.S.A.
(Ti6Al4V)	Ti: base (90%), Al: 6%, V:4%	South Asia Metals Pvt., Ltd., India
Ni-Cr alloy	Ni-61.4%, Cr-25.7%, Mo- 11%, Si-1.5%, Mn <1%, Al <1%, C<1%	Ivoclar vivadent, Switzerland
Gold alloy maxigold	Au-59.5%, Ag-26.3%, Pd-2.7%, Cu-8.5%, Zn-2.7%, In <1%, Ir <1%S	Ivoclar vivadent, Switzerland

[Table/Fig-1]: Materials, composition and suppliers.

S. No.	Electrode	System
1	Pure titanium with gold alloy	PT+GA ₁ →Artificial saliva PT+GA ₂ →Artificial saliva-1000 ppm NaF
2	Pure titanium with NC alloy	PT+NC ₁ →Artificial saliva PT+NC ₂ →Artificial saliva-1000 ppm NaF
3	(Ti6Al4V) with gold alloy	TA+GA ₁ →Artificial saliva TA+GA ₂ →Artificial saliva-1000 ppm NaF
4	Ti6Al4V with NC alloy	TA+NC ₁ →Artificial saliva TA+NC ₂ →Artificial saliva-1000 ppm NaF

[Table/Fig-2]: Electrodes and systems.

was adjusted to 6.4 by adding Sodium hydroxide (Lobachemie, India). The pH was set at 6.4 based on the study done previously [10]. The second test solution was prepared by adding 1000 ppm of NaF to artificial saliva.

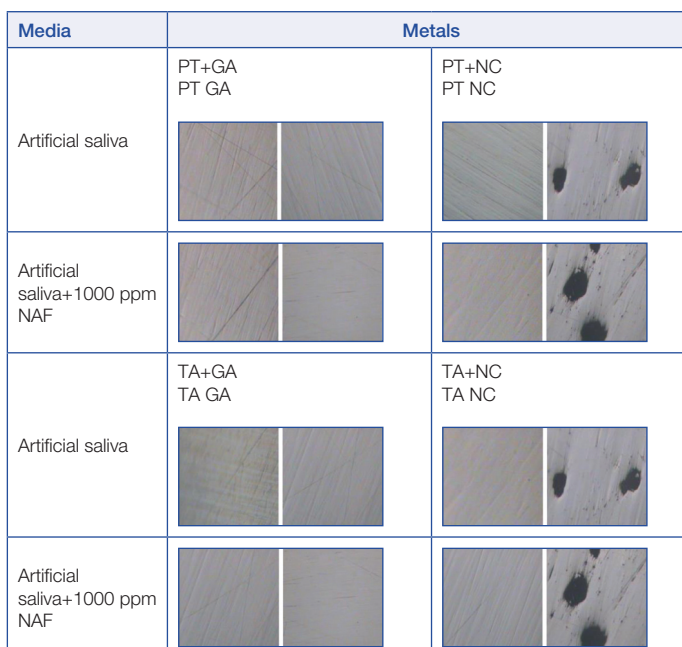
Galvanic corrosion study: By using corrosion tests, surface analyses, and elemental release analyses, the galvanic corrosion behaviour of PT and TA combined with GA and NC alloy was investigated. Platinum electrode served as the counter electrode and saturated calomel electrode served as the reference electrode [6]. The test solution, which constituted 100 mL of the test, was placed in a 250 mL beaker along with the reference electrode, counter electrode, and the specimens. The current was passed at a scanning rate of 1800 mV/hour. For 60 minutes, the corrosion potential with immersion time was measured (Potentiostat/Galvanostat, VoltaLab 21 (PGP201 and Voltmaster 4), radiometer analysis Statistical Analysis System (SAS), France). The electrodes were then taken out of the test solution, given a gentle rinse in distilled water, and allowed to air dry. In order to evaluate the corrosion behaviour, the surfaces of the specimens were examined with an optical microscope (Eclipse LV100D, Nikon, USA). The elemental release analysis test was conducted by using inductively coupled plasma-mass spectrometry (plasma quad II, VG Elemental, Canada) to quantitatively analyse the elements (metal ions) released in the test solution from the galvanic coupling alloys [8]. The amount of elements released was measured in µg/cm².

STATISTICAL ANALYSIS

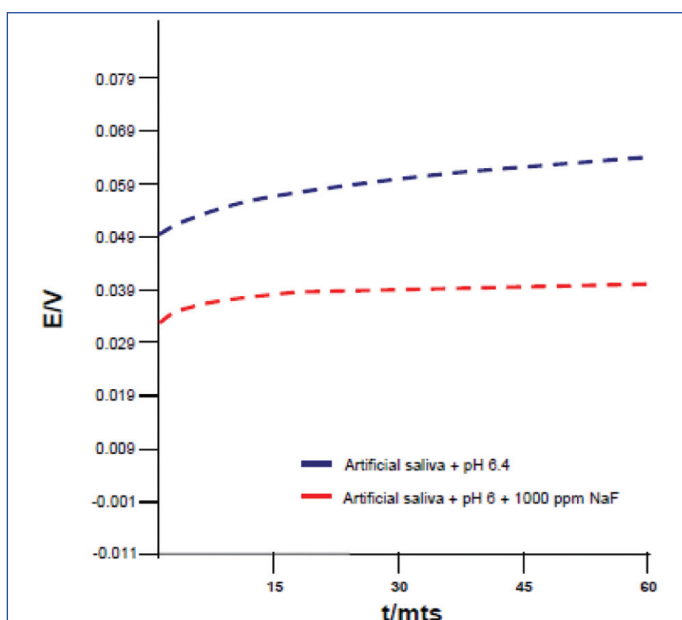
The data was analysed using Statistical Package for Social Sciences (SPSS) version 21.0 {International Business Machines (IBM), Armonk, NY, USA}. Statistical significance was set at 0.05 levels. Test of normality distribution resulted in p-value <0.05. Hence, non parametric test were used for analysis. Intergroup comparison was analysed using Kruskal-Wallis test and intragroup comparison was analysed using Mann-Whitney U test.

RESULTS

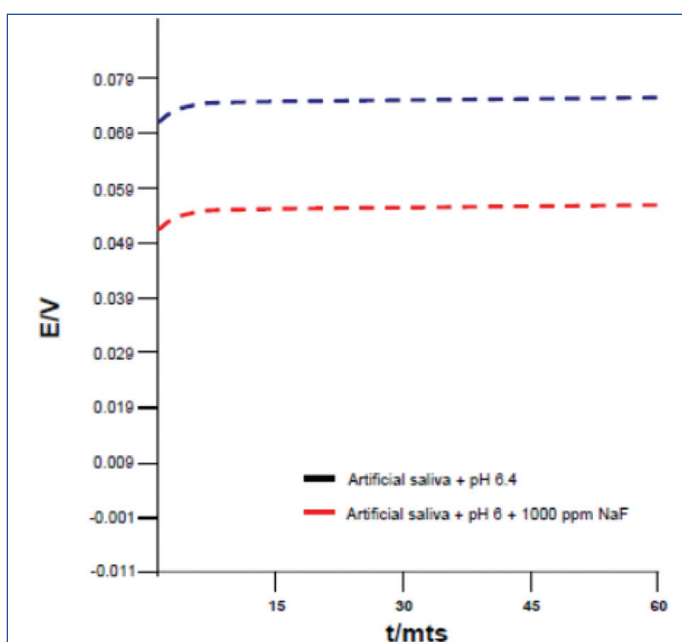
Corrosion test: In the presence of 1000 ppm fluoride, the metal surfaces of PT+GA and TA+GA did not exhibit any increase in surface roughness, according to the optical microscopic image of metals after galvanic corrosion. The PT+NC and TA+NC showed pitting corrosion in the presence of 1000 ppm fluoride and in the absence of fluoride. The size and number of pits was increased in the presence of fluoride. PT's surface did not become rough even when fluoride was present, whereas, there was a slight increase in roughening on the surface of TA in the presence of fluoride [Table/Fig-3]. The potential of PT and TA coupled with GA showed a decreased value in presence of fluoride [Table/Fig-4,5]. The corrosion behaviour of



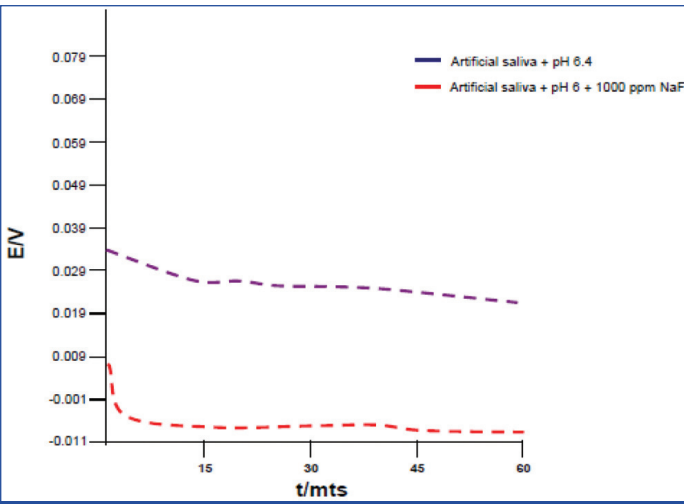
[Table/Fig-3]: Optical microscopic picture of metals after galvanic corrosion.



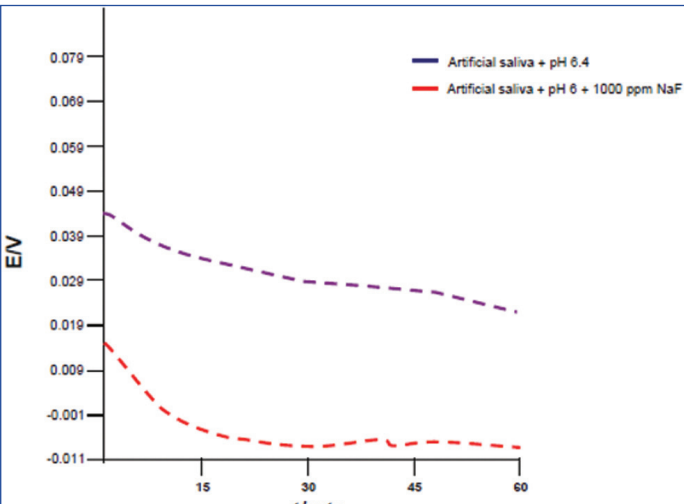
[Table/Fig-4]: Mean galvanic potential when PT coupled with GA.



[Table/Fig-5]: Mean galvanic potential when TA coupled with GA.



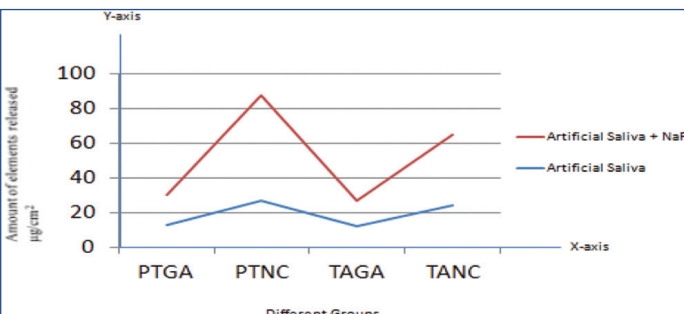
[Table/Fig-6]: Mean galvanic potential when PT coupled with NC.



[Table/Fig-7]: Mean galvanic potential when TA coupled with NC.

PT and TA coupled with NC showed a marked decrease in potential in the initial 10 minutes time irrespective of fluoride presence, then attained a static level for the remaining time tested [Table/Fig-6,7]. The very low potential of the PT and TA combined with NC in the presence of fluoride indicated that significant corrosion was taking place in this combination.

Elemental release: The mean elemental release by different groups is shown in [Table/Fig-8]. The GA released in the test solution was more in presence of fluoride, when GA was coupled with PT and TA. The released amount of PT and TA was negligible when coupled with GA. The NC content of NC alloy release was much higher in presence of fluoride when NC was coupled with PT and TA. In presence of fluoride, the titanium release was higher when PT was coupled with NC than in any other combination. In [Table/Fig-9], the amount of elements released when PT and TA were combined with GA and NC and represented in $\mu\text{g}/\text{cm}^2$ is shown. There was statistically significant difference ($p < 0.001$) when titanium was combined with GA in comparison to combination with NC alloys.



[Table/Fig-8]: The mean elemental release by different groups.

Test solutions	Groups	Mean $\mu\text{g}/\text{cm}^2$	Standard deviation	95% confidence interval for mean		Chi-square test statistic	p-value
				Lower bound	Upper bound		
Artificial saliva	PT+GA	13.00	1.265	11.67	14.33	19.244	0.001*
	PT+NC	27.00	2.000	24.90	29.10		
	TA+GA	12.00	0.894	11.06	12.94		
	TA+NC	24.00	1.414	22.52	25.48		
Artificial saliva+NaF	PT+GA	17.00	0.632	16.34	17.66	20.672	0.001*
	PT+NC	60.67	4.320	56.13	65.20		
	TA+GA	15.00	2.098	12.80	17.20		
	TA+NC	41.00	1.897	39.01	42.99		

[Table/Fig-9]: Kruskal-Wallis test comparing the different groups across the test solutions of amount of elements released. (*p-value<0.05 considered statistically significant)

Intergroup comparison: In the presence of artificial saliva alone PT+GA group when compared with PT+NC, TA+GA and TA+NC showed a p-value=0.002*, 0.180 and 0.002* respectively. Similarly, in the presence of artificial saliva and sodium fluoride, PT+GA group when compared with PT+NC, TA+GA and TA+NC showed a p-value=0.002*, 0.065 and 0.002*, respectively. It was found that, each group differ from other groups with statistical significance ($p < 0.05$) except PT+GA and TA+GA where, there were no significant differences noted ($p > 0.05$) as shown in [Table/Fig-10].

Groups	Comparison group	Artificial saliva p-value	Artificial saliva+NaF p-value
PT+GA	PT+NC	0.002*	0.002*
	TA+GA	0.180	0.065
	TA+NC	0.002*	0.002*
PT+NC	TA+GA	0.002*	0.002*
	TA+NC	0.026*	0.002*
TA+GA	TA+NC	0.002*	0.002*

[Table/Fig-10]: Mann-Whitney U test showing the multiple comparison of amount of elements released. (*p-value<0.05 considered statistically significant)

Intragroup comparison: It was found that, there were significant statistical differences seen between the two test solutions across all the groups ($p < 0.05$). Mean elemental release was observed to be more in artificial saliva+NaF test solution when compared to only artificial saliva test solution [Table/Fig-11].

Groups	Mean (SD)		Z-score test statistics	p-value
	Artificial saliva $\mu\text{g}/\text{cm}^2$	Artificial saliva+NaF $\mu\text{g}/\text{cm}^2$		
PT+GA	13 (1.26)	17 (0.63)	-2.956	0.002*
PT+NC	27 (2)	60.67 (4.32)	-2.908	0.002*
TA+GA	12 (0.89)	15 (2.1)	-2.282	0.026*
TA+NC	24 (1.41)	41 (1.9)	-2.898	0.002*

[Table/Fig-11]: Mann-Whitney U test showing the intragroup comparisons of amount of elements released. (*p-value<0.05 considered statistically significant)

DISCUSSION

In the current study, corrosive resistance of PT and TA was compared after combining the materials with either GA or nickel chromium alloy. In the present study, titanium has been taken into consideration, because titanium has been shown to be a suitable biocompatible material for crown restoration [1,2,6]. The biocompatibility of titanium implants is significantly influenced by the chemical characteristics of the oxide layer [2]. Previous studies done on investigating the corrosion resistance of gold and TA is shown in [Table/Fig-12] [5-9]. Fluoride containing dental gels and

S. no.	Author's name and year	Place of study	Parameters studied	Conclusion
1	Ward LP et al., (2013) [5]	Australia	Quaternary gold-silver-copper-zinc alloys	Increased zinc and reduced copper promoted increased corrosion resistance to the alloys
2	Wang ZB et al., (2016) [6]	China	Titanium and titanium alloys were investigated for corrosive resistance	Titanium alloys exhibited higher corrosive resistance than pure titanium
3	Li Y and Xu J (2017) [7]	China	Commercially pure titanium compared with niobium for its corrosive resistance	Niobium exhibits a significant corrosive resistance
4	Klotz U and Heiss T (2021) [8]	Oregon	Platinum and gold alloys for its wear resistance	Platinum shows higher corrosive resistance compared to gold alloys
5	Su B et al., (2021) [9]	China	Ti-6Al-3Nb-2Zr-1Mo (Ti80) alloy	Beta phase of Ti-6Al-3Nb-2Zr-1Mo (Ti80) alloy exhibited higher corrosive resistance when compared to alpha phase
6	Present study	India	Corrosion resistance of pure titanium and titanium alloys coupled with gold alloy and nickel chromium	Pure titanium and titanium alloys coupled with gold alloy was better in corrosion resistance when compared to nickel chromium alloy combination.

[Table/Fig-12]: Studies done on investigating the corrosion resistance of gold and titanium alloys [5-9].

mouthwashes are being used more frequently to prevent dental cavities. Dental implants, especially those, that rely on low pH values for greater effectiveness, should be taken into account before using fluoride preparation in dental applications. In the place of NaF, fluoride-titanium (or) fluoride-hydrogen compounds were deposited on the titanium surface [11,12]. In the solution, sodium fluoride breaks down into sodium ions and fluoride ions. Depending on the pH of the medium, the fluoride ion can partially transform into hydrofluoric acid. The passive films on the titanium surface are damaged by the hydrofluoric acid [4].

In the current study, it was reported that, PT and TA showed decreased potential, which was in accordance to the study done by Mayouf AM et al., where, sodium fluoride concentration increased upto 0.1%, the fluoride ions attack on the titanium-dioxide passive layer resulted in its destruction by the development of the soluble titanium-fluoride complex combination [13]. According to predictions, titanium will not corrode in an acidic solution with fluoride concentrations below 1000 ppm. TA have shown to exhibit higher corrosion resistance when compared to PT alloy [2,4,6]. The severity of fluoride ion attack on titanium-di-oxide depends on its concentration and the pH level of saliva [11]. Corrosion resistance of titanium was maintained in 2% sodium fluoride solution at pH>6.2. The oxide film on titanium metal and its alloys has two layers, an outer layer, that is porous and a dense interior layer. The corrosion resistance of titanium and its alloys are controlled by an acidic solution and the total fluoride concentration in the solution [4]. GA coupled with titanium was an excellent couple with negligible corrosion. NC alloy showed unstable galvanic corrosion behaviour when coupled with titanium implant [14]. Similarly, in the present study titanium combined with NC showed more corrosion when compared to Ti-Au group. This was in accordance with the study done by Reclaru L and Meyer JM, titanium coupled with GA cause negligible galvanism [15].

In an electrolytic environment, fluoride ions cause titanium to be aggressively attacked. This is because complex titanium-fluoride molecules are formed, and these molecules are extremely stable and soluble in an electrolytic solution. The titanium-tri and tetra halides frequently combine with oxidisable atoms to produce complexes.

Some halide complexes are $(TiF_6)^{2-}$ and $(TiF_6)^{3-}$. The dissolution of titanium in the presence of fluorides was explained by the creation of these types of salts [16,17]. Saliva infiltration causes galvanic cells between dental alloys, which are followed by corrosion, between implants and implant supported structures [18]. Titanium is resistant against corrosion and is used as a constructive material in corrosive environments. Its stability is due to the formation of passive surface oxide layer, which is absent in an acidic solution. PT and (Ti6Al4V) exhibited high resistances to the onset of localised corrosion [18]. During the longer implantation time, the fluorine content of the titanium modified layer rose noticeably, and the surface elements were TiF_3 and titanium-dioxide [19]. The rate of passive oxide layer regeneration is a linear function of the dissolved oxygen concentration once the passive layer on titanium has been damaged by fluoride assault [20,21]. The addition of small amount of platinum (or) palladium to titanium is very effective in improving the corrosion resistance of titanium in sodium fluoride solution of various concentrations upto 2%. Surface alloying of titanium with palladium improved the corrosion resistance of titanium in highly acidic solution [22]. Because of the low oxygen content, the regeneration of the passive oxide layer was delayed [1]. The addition of platinum (or) palladium to titanium promoted the formation of titanium-di-oxide on the titanium surface resulted in the high corrosion resistance of titanium-platinum (or) titanium-palladium alloys. While the surfaces of TA containing palladium (or) platinum were undamaged, the acidic fluoride-containing saliva roughened the surfaces of PT and the (Ti6Al4V). This was supported by a study done by Klotz U and Heiss T revealed that, platinum showed more corrosion resistance [8]. In low dissolved oxygen environment, the surfaces of PT and (Ti6Al4V) were damaged by corrosion. This increases the possibility in the stress corrosion cracking of a titanium implant [1]. The protectiveness of titanium-di-oxide passive film formed on (Ti6Al4V) was destroyed by fluoride ions via the formation of Na_2TiF_6 when the sodium fluoride concentration was increased upto 0.1% [21]. The concentration of fluoride affects how quickly the titanium-dioxide layer grows. At the oxide layer, internal pressures causing blister formation were found when fluoride was present [22]. The composition of saliva including fluoride concentration and imperfections in the titanium surface influence the titanium-di-oxide formation and titanium passivity. The oxide film's thickness has an impact on the procedure as well [23].

Limitation(s)

The limitations of the study were inability to analyse the biocompatibility of the metals used. Since, short time was implicated for assessing the corrosion behaviour, actual corrosion resistance values was not attached. Being an electropolarisation study, the ion analysis value couldn't be detected accurately.

CONCLUSION(S)

The PT and TA specimens coupled with NC alloy specimens showed more corrosion in saliva compared to GA indicating the use of GA crowns for superstructure with PT and TA implants. The NC alloy specimens coupled with PT and TA specimens showed severe pitting corrosion in artificial saliva containing sodium fluoride. Further research can be carried out on evaluating the biocompatibility, the application of PT and TA coupled with NC alloys in patients mouth and follow-up of patients to determine the success of these materials clinically.

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