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Randomized study to evaluate and compare the corrosion for five different orthodontic wires surface because of prophylactic fluoride exposure and GC tooth mousse with distilled water using scanning electron microscope (SEM)

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Abstract---Maintaining proper oral hygiene and caries management is one of the most crucial aspects of effective orthodontic treatment. Enamel demineralization occurs around Orthodontic brackets as a result of direct bonding and de-bonding treatments. Many sites have been observed to be vulnerable to plaque buildup. Due to the numerous locations available for plaque collection and the difficulty in cleaning them, maintaining excellent oral hygiene is challenging. Chlorhexidine mouth rinses, Fluoride mouth rinses, and the newly available GC Tooth mousse are all common methods of maintaining oral hygiene in Orthodontic patients. Orthodontic wires have been found to corrode and discolour due to fluoride ions generated by fluoride prophylactic agents. The coefficient of friction is increased when the surface properties of the arch wires are altered by pitting and corrosion, which has clinical importance during the gap closure stage. Using a Scanning Electron Microscope (SEM), the following article attempts to examine and compare the corrosion of five distinct Orthodontic wire surfaces as a result of prophylactic fluoride exposure and GC Tooth mousse with Distilled water.

Keywords---orthodontic treatment, demineralization, fluoride prophylactic agents, corrosion, orthodontic wires.

Introduction

One of the most important requirements of successful Orthodontic treatment is the maintenance of good oral hygiene and caries control. Direct bonding and debonding procedures lead to areas of enamel demineralization around Orthodontic brackets. During these procedures many of the times more area than that covered by brackets is etched and may remain exposed for plaque accumulation. These etched areas lead to demineralization and white spot lesions around the brackets. Maintaining good oral hygiene is difficult due to multiple areas available for plaque accumulation and difficulty in cleaning them.

Common methods of maintaining oral hygiene in Orthodontic patients is the use of Ortho brush, Chlorhexidine mouth rinses, Fluoride mouth rinses and recently available GC Tooth mousse. Fluoride prophylactic agents such as, acidulated phosphate fluoride and sodium fluoride have been extensively used to prevent the demineralization and help in the remineralization of white spot lesions around Orthodontic brackets. However, the fluoride ions have been reported to cause corrosion and discoloration of Orthodontic wires. Alteration of the surface characteristics of the arch wires by the way of pitting and corrosion of the surface increase the coefficient of friction which has its clinical significance during space closure stage^{1,2}.

Orthodontic mechanotherapy encompasses the use of several arch wires during various stages of treatment. Stainless steel, Nickel titanium and TMA wires are the ones predominately used. The Titanium and its alloys are easily passivated metals with surface oxides that do not breakdown under physiologic conditions. Stainless steel and Nickel titanium arch wires normally corrode in the oral cavity by interaction with oral fluids. Topical fluoride agents further aggravate the corrosion of these arch wire. Though, β titanium arch wires have some resistance to corrosion, the passivated wire surface disintegrates and corrodes due to mechanical friction between bracket and arch wire in the presence of fluoride ions.

Objective

To evaluate and compare the corrosion for five different Orthodontic wires surface as a result of Prophylactic fluoride exposure and GC Tooth mousse with Distilled water using Scanning Electron Microscope (SEM).

Materials and Methods

The study was carried out in the Department of Orthodontics and Dentofacial Orthopedics, Sharad Pawar Dental College and Hospital, Sawangi (Meghe) Wardha in collaboration with Department of Metallurgy Engineering, Visvesvaraya National Institute of Technology (VNIT), Nagpur.

Materials

The material used for the study included five different types of wire, which are commonly used for Orthodontic treatment:

- 1) 0.019" x 0.25" Nickel Titanium Wire
- 2) 0.019" x 0.25" Beta Titanium Wire
- 3) 0.019" x 0.25" Stainless Steel Wire
- 4) 0.019" x 0.25" Copper Nickel Titanium wire
- 5) 0.016 "A.J. WILCOCK. SPECIAL PLUS Australian wire

The topical applicants selected are commonly prescribed by the Orthodontists to their patients during Orthodontic treatment, to prevent demineralization of tooth or white spot lesions. (Photograph 1a and 1b) (Plate 1)

1. FLUROVIL gel (acidulated phosphate fluoride gel (APF gel) containing A stable aqueous gel providing 1.23% fluoride ion (derived from NAF and HF) (pH-4.1)
2. PHOSFLUR mouth rinse (Sodium fluoride and acidulated phosphate topical solution) 16 FL.OZ. (1PT) (pH-5.2)
3. GC Tooth mousse (Ingredients: pure water, glycerol, CPP-ACP, D-Sorbitol, CMC-Na, propylene glycol, Silicon dioxide, Titanium dioxide, Xylitol, Phosphoric acid, Flavouing agents, Zinc oxide, Sodium saccharin, Ethyl p-hydroxybenzoate, Magnesium oxide, Guar gum, propyl p- hydroxybenzoate, Butyl p- hydroxybenzoate (pH- Neutral-7). At the time study was conducted GC Tooth Mousse with fluoride was not introduced.

Various reviewed literatures

In the present published article, studies carried out in the past related to mechanical properties as well as related to corrosion of various Orthodontic wires by various chemical agents and oral environment, are reviewed.

- John Edie et al³ 1981 study the surface corrosion of nitinol and stainless steel under clinical conditions, the eleven nitinol and eleven stainless steel wires were retrieved after clinical service for period ranging from one month to eight months and observed in SEM, there is no reason to believe that nitinol wires are any more subject to corrosion than stainless steel wires are any more subject to corrosion than stainless steel wires in an environment that is typical of clinical service.
- M Mathew, R Kusy⁴ in 1988 conducted the effects of sterilization on the mechanical properties and the surface topography were determined on 0.017 x 0.025-inch Nitinol and Titanium arch wires. Three approved heat sterilization methods were used: dry heat, formaldehyde-alcohol vapor, and steam autoclave. Elastic moduli were obtained on 1-inch segments in 3-point bending. Laser scans of flatwise wire surfaces were conducted to detect surface alterations— whether they were caused by tarnish, corrosion, or pitting. Tensile properties were determined on 7-inch lengths: the 0.1% yield strength, the ultimate tensile strength, and the percent elongation at

break. Within the confines of the present sterilization experiments, no detrimental changes were observed for either the selected mechanical properties or the surface topography. When the mean values of the two products were compared, Nitinol was less compliant but stronger than Titanium.

- MH Kononen, and ET Lavonius⁵ at the Department of prosthetic Dentistry, University of Helsinki, Finland in 1995 made scanning electron microscope observations on stress corrosion cracking of commercially pure titanium in a topical fluoride solution. Thin electropolished titanium test specimens were previously cold rolled or cold rolled and annealed before testing. 'U' shaped specimens of both treatment types were stressed into a radius of curvature of 30 mm. The bent part was then placed in the fluoride solution at 37° c for 1, 5, 10 and 20 days. The effect of the fluoride solution on cold rolled and annealed titanium was studied using a S.E.M. In addition, mechanically fractured surfaces solutions were examined by S.E.M. Narrow cracks were observed in cold rolled specimens following exposure to the fluoride solution for 5 days. The cracks were associated with branching, a characteristic of stress corrosion cracking. The authors also found that the mode of fracture was brittle. In contrast, the fracture mode of the unexposed specimens was ductile in nature.
- Toumelin - Chemla and F Rouelle, G Burdairon⁶ in 1996 studied the corrosive properties of fluoride containing odontologic gels against Titanium. They used the linear sweep volumetric analysis, making use of a rotating disc electrode. The purpose of this study was to verify in which conditions the electrolytic medium had an influence on the corrosion of titanium. Titanium alloys were immersed in neutral Ringers' solution (pH=7), fluoridated odontologic gel (fluogel; pH - 5.5), Ringers' solution with added fluorides, Non-fluoridated Ringers* solution (pH=4) and fluoridated Ringers' solution (pH=5.5). They found that titanium suffers significant degradation in fluoridated acid media. The degradation was found only in fluoridated media and not in acidulated non fluoride media.
- Platt et al⁷ (1997) studied the corrosion behavior of 2205 duplex stainless steel was compared with that of AISI type 316L stainless steel. Both stainless steels were subjected to electrochemical and immersion (crevice) corrosion tests in 37% C, 0.9 wt% sodium chloride solution. Electrochemical testing indicates that 2205 has a longer passivation range than 316L. The corrosion rate of 2205 was 0.416 MPY (milli-inch per year), whereas 316L exhibited 0.647 MPY. When 2205 was coupled to 316L with equal surface area ratio, the corrosion rate of 2205 reduced to 0.260 MPY, indicating that 316L stainless steel behaved like a sacrificial anode. When 316L is coupled with NiTi, TMA, or stainless steel arch wire and was subjected to the immersion corrosion test, it was found that 316L suffered from crevice corrosion. On the other hand, 2205 stainless steel did not show any localized crevice corrosion, although the surface of 2205 was covered with corrosion products, formed when coupled to NiTi and stainless steel wires. This study indicates that considering corrosion resistance, 2205 duplex stainless steel is an improved alternative to 316L for Orthodontic bracket fabrication when used in conjunction with titanium, its alloys, or stainless steel arch wires

- Kim Hera, J Johnson⁸ (1999) conduct a study to determine if there is a significance difference in the corrosive potential of stainless steel, Nickel titanium, and nitride coated Nickel titanium, epoxy coated Nickel titanium and titanium Orthodontic wire. Which were subjected to potentio static anodic dissolution in 0.9% sodium chloride solution with neutral ph at room temp. SEM photograph revealed that corrosion occur readily in stainless steel as compare to Nickel titanium.
- Nakagawa et al⁹ (1999) studied the corrosion of titanium in prophylactic fluoride environment. The results of this study revealed that HF concentration higher than 30ppm caused destruction of the passivation film of the titanium which predisposed the alloy for corrosion.
- T Eliades, G Eliades and A Athanasiou¹⁰ (2000) studied surface characterization of retrieved NiTi Orthodontic wires. Structure and morphological condition of retrieved NiTi Orthodontic archwires were evaluated for alterations in surface composition of the alloy following 1-6 months in the oral cavity. Optical microscopy revealed islands of amorphous precipitants and accumulated microcrystalline particles. Micro MIR-FTIR investigation demonstrated the presence of a proteinaceous biofilm containing amide, alcohol and carbonate. SEM and x-ray microanalysis showed precipitation of Na, K, Cl, Ca and P forming NaCl, KCl, Ca-P precipitates. Increased intraoral exposure resulted in alloy delamination, pitting and crevice corrosion.
- HH Huang¹¹ (2002), studied the corrosion resistance of commercially pure titanium in 1% NaCl solution and approximately 1% NaF solution (PH-6) under different elastic tensile strains using electrochemical impedance spectroscopy (EIS) measurement technique. Polarization resistance (RP) which is inversely proportional to the corrosion rate for the commercially pure titanium was obtained using the E.I.S. data. Different elastic tensile strains, namely 0%, 1%, 2% and 4% were applied on the test specimens during the corrosion test. In addition, corrosion morphology was characterized using a S.E.M. and surface chemical analysis was done using energy dispersive spectroscopy and x-ray photoelectron spectroscopy. The results showed that, when the NaF concentration was lower than 0.1%, the Rp value was increased due to formation of protective titanium dioxide layer on the metal surface regardless of the tensile strain applied. However, when the NaF concentration was higher than 0.1%, the protectiveness of TiO₂ was destroyed by fluoride ions, leading to severe corrosion of titanium.

Method

Total 120 wire samples of 50mm length were divided in following 4 groups:

GROUP	TREATMENT MATERIAL	WIRE USED	SAMPLE NO.	TOTAL SAMPLE
Group A (control)	Distilled water	Nickel Titanium Wire	06	30
		Beta Titanium Wire	06	
		Stainless Steel Wire	06	
		Copper Nickel Titanium wire	06	

		0.016 " A.J. WILCOCK ss	06	
Group B (experimental group)	Phosflur	Nickel Titanium Wire	06	30
		Beta Titanium Wire	06	
		Stainless Steel Wire	06	
		Copper Nickel Titanium wire	06	
		0.016 " A.J. WILCOCK ss	06	
Group C (experimental group)	Flurovil	Nickel Titanium Wire	06	30
		Beta Titanium Wire	06	
		Stainless Steel Wire	06	
		Copper Nickel Titanium wire	06	
		0.016 " A.J. WILCOCK ss	06	
Group D (experimental group)	GC Tooth mousse	Nickel Titanium Wire	06	30
		Beta Titanium Wire	06	
		Stainless Steel Wire	06	
		Copper Nickel Titanium wire	06	
		0.016 " A.J. WILCOCK ss	06	

Control Group

Group A: Thirty (30) wire samples consisting of six (6) samples of each wire type were immersed in 10 ml DISTILLED WATER at 37⁰ C in 5 test tube for 90 minutes.

Experimental Groups

Group B : Thirty (30) wire samples consisting of six (6) samples of each wire type was immersed in 10ml PHOSFLUR mouth rinse at 37⁰ C in 5 test tubes as per for 90 minutes.

Group C : Thirty (30) wire samples consisting of six (6) samples of each wire type was immersed in 10ml FLUROVIL gel at 37⁰ C in 5 test tubes as per for 90 minutes.

Group D: Thirty (30) wire samples consisting of six (6) samples of each wire type was immersed in 10ml GC Tooth mousse at 37⁰ C in 5 test tubes for 90 minutes.

The exposure time of 90 minutes is equivalent to three months of 1 minute daily topical fluoride application or fluoride rinse as stated by Mary Walker and Richard White in 2005.¹² The wires from control and experimental groups were scanned under Scanning Electron Microscopy (S.E.M.) observed (Philips XL30 field emission SEM, Phillips Electron Optics, Hillsboro, Ore). The sample were mounted directly on the metal stubs and scanned at a depth of 10 micro. The

SEM microphotograph was made at $2000 \times$ magnification and the images thus obtained were evaluated for corrosion.

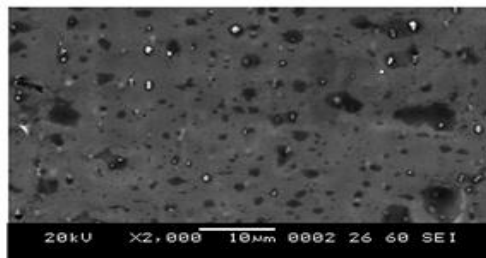
Scanning Electron Microscope (SEM) Observation

Ni- Ti wire

Significant variation was observed in the amount of corrosion seen in the NiTi wires dipped in the various solutions as compared to segment of wire dipped in the Distilled water, as shown in Microphotograph 1A to 1D. NiTi wire segments dipped in Flurovil showed a significant level of corrosion. The highest corrosion was seen in the sample dipped in Flurovil. There were increased areas of defects and also increase in the number of white inclusion bodies seen when Flurovil was compared with control group. These white inclusion bodies were exposed after the alloy was corrode due to the fluoride. (1C) There was also a significant level of corrosion seen in Phosflur with increase in white inclusion bodies. But in comparison with Flurovil it was significantly less. (1B) No significant change in surface topography was observed when NiTi wire segment immersed in GC Tooth mousse was scanned. The SEM images showed similar surface topography with inherent defect of wire as seen in the control group.

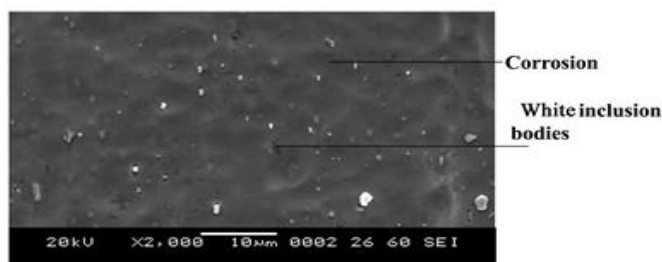
Ni-Ti wire sample :

Ni-Ti wire aged in Distilled water



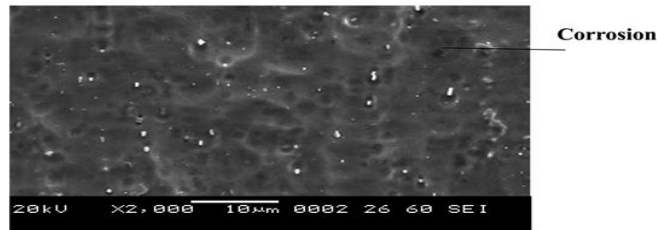
Microphotograph 1A. Showing that the black spot of NiTi sample aged in Distilled water

Ni-Ti wire aged in phosflur



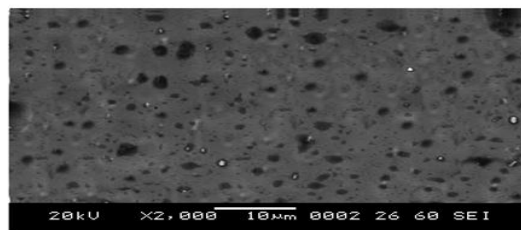
Microphotograph 1B. Showing Location of metal distortion Ni-Ti sample aged in phosflur

NiTi wire aged in Flurovil



Microphotograph 1C. Showing Location of chemical attack on NiTi sample aged in Flurovil

NiTi wire aged in GC Tooth mousse



Microphotograph 1D. Showing that the black spot of NiTi sample aged in GC Tooth mousse

Cu NiTi wire

When Cu-NiTi wire was scanned and compared, it also exhibited similar findings. Increase in the size and number of defect were observed in the sample treated with Flurovil gel as compared to Distilled water (control). The sample treated with Phosflur solution also exhibited the corrosion but the amount of defect due to corrosion was much less than Flurovil. The sample treated with GC Tooth mousse did not show any change in surface corrosion which was similar to control group. We observed comparatively lesser corrosion in the sample of Cu-NiTi when compared with corrosion in the samples of NiTi, as shown in Microphotograph 2A to 2D.

Beta Titanium wire

When Beta titanium wire immersed in Distilled water was scanned the inherent defect such as cracks were observed. The wire sample exposed to the Flurovil showed increase in the length of crack, widening of cracks and increase surface roughness. Lesser increase and widening of cracks were seen more with Phosflur as compared to the Flurovil. No significant variation in the surface topography was observed in the scanned image of the wire dipped in GC Tooth mousse as compared to control group (Microphotograph 3A to 3D).

Stainless Steel wire

When Stainless steel wire was scanned and compared it was found that the wire samples exposed to Distilled water and GC Tooth mousse had no corrosive effect. Occasional gray and black areas were seen which may be by-products of the manufacturing process. When the wire sample exposed to the Flurovil was scanned, chemical segregation of black areas and white inclusion bodies in the metal surface was seen along with corrosion of the metal surface. In the wire sample exposed to Phosflur there is less segregation of black areas and white inclusion bodies in the metal surface were seen along with corrosion of the metal surface as compare to wire sample dipped in Flurovil (Microphotograph 4A to 4D).

Australian A. J. Wilcock

When Australian stainless steel wire was scanned and compared it was found that the wire samples exposed to Distilled water and GC Tooth mousse had no corrosive effect on the surface area of the metal matrix. When the wire sample exposed to the Flurovil was scanned, white inclusion bodies in the metal surface was seen along with corrosion of the metal surface, with increase surface roughness. In the wire sample exposed to Phosflur was seen less white inclusion bodies and less corrosion on the metal surface as compared to Flurovil (Microphotograph 5A to 5D).

Discussion

The history of technical development in Orthodontics has been marked by a continuing search to improve Orthodontic treatment and also to provide best health care protocol. Orthodontic wires have progressed from Nickel-Chromium-Iron, Stainless steel alloy to Nickel-Titanium, Beta-Titanium, Copper-NiTi, Elgiloy, Chinese NiTi etc. Corrosion resistance of wires is equally important just like modulus of elasticity and yield strength as corrosion may induce friction and affect the mechanical properties of the wire. The corrosion occurs because of the disintegration of oxide layer i.e., Chromium oxide in Stainless steel and elgiloy or Chromium based wires and Titanium oxide in Titanium based wires ^{13,14,15,16,17}.

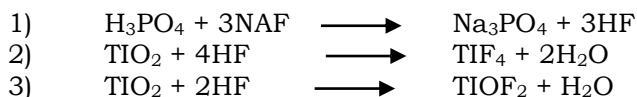
This oxide layer is thin and stable as a result of passivation. Titanium based alloys are known to exhibit good corrosion resistance in artificial saliva, Ringer's solutions and NaCl due to the presence of this protective layer which usually does not break down and thus prevent further corrosion of underneath material¹⁸. But this thin protective layer is known to break down in the presence of fluoride ¹⁹. Mouthwash regime is usually prescribed in Orthodontic patients to maintain good oral hygiene and to prevent enamel decalcification from the plaque deposited around the bracket.

Several species of bacteria like S.Mutans, and H.Casei reside in the film of dental plaque which fragments carbohydrate to produce lactic acid, propionic acid and acetic acid. It also causes release of hydrogen ion. If small quantity of fluoride is present in the oral cavity as a part of oral hygiene regime, it will react with hydrogen ion to form hydrofluoric acid (HF) This HF reacts with oral bacteria and

inactivates them. It also brings about remineralization of enamel thereby reducing the white spot lesions of the hard tissue.²⁰

But the drawback is, HF (hydrogen fluoride) also reacts with the Titanium oxide and Chromium oxide dissolving the protective thin covering of outer layer, forming Titanium fluoride (TiOF) and Chromium fluoride on the surface which leads to surface discoloration and corrosion.

According to the equation ^{21,2}:



This process is known as Hydrogen embrittlement.²² The most commonly advocated oral hygiene procedure uses fluoride agents. They are commonly used as gels, solutions, varnishes, mouth rinses etc. They come in various concentrations of fluoride and have varying pH. On the basis of our SEM findings the Titanium based Orthodontic wires gets corroded in the presence of fluoride. There is comparably less corrosion of β -titanium as compared to that of NiTi and Cu-NiTi, the reason may be increased concentration of Titanium in β -titanium.

More corrosion occurs in the gel form (Flurovil) of fluoride as compared to solution form (Phosflur). This, can be attributed to the difference in pH of both the agents and more availability of fluoride ions. More corrosion occurs in acidic pH-4.1 of gel (Flurovil) as compared to pH-5.2 of solution (Phosflur). Corrosion or breakdown of Titanium oxide occurs readily in acidic solution ^{6,7,8,9,10,20,22}. The NiTi and Cu-NiTi absorb hydrogen ion due to the high affinity of titanium to hydrogen which disturbs the pattern of interstitial atomic arrangement. This absorbed hydrogen atom is known to prevent martensitic transformation as this transformation is sensitive to interstitial atomic arrangement. ^{13,14,21}

When the Chromium based archwires were evaluated [Rigid Stainless Steel (0.019 x 0.025) and flexible 0.016 Australian AJ Wilcock heat-treated Stainless-steel wires], Corrosion was seen associated with the inherent defects in the alloy. More corrosion and presence of inclusion bodies were observed in both the Stainless-steel wires dipped in Flurovil as compared to control sample. The corrosion of rigid Stainless-steel wire was less as compared to flexible Stainless-steel wire (4A to 5D). Large corroded areas were seen in 0.016 Australian AJ Wilcock stainless steel wire treated with Flurovil as compared to Rigid Stainless-Steel wires.

May be difference in thickness and method of manufacturing is responsible for increased corrosion observed in AJ Wilcock wire as compared to Rigid Stainless-Steel wire.

- Maximum Scanning Electron Microscope studies have been carried out on Titanium wires and the results of the present study are in accordance with those studies conducted by:

- Kaneko K and Yokoyama K found roughened surface due to corrosion in NiTi, Beta –titanium and stainless steel wires when they were immersed in 2% APF solution for 60 minute.
- F .Toumelin – Chemla et al found that the prophylactic fluoridated odontological gel showed acidic ph value, which caused corrosion in titanium alloys such as branemark (commercially pure titanium i.e. impurities < 0.25%) using Electrochemical device.
- Ikuya and Etsuko Watanabe studied beta titanium wire immersed in 2.59% Sodium fluoride. The TMA wire showed less tarnish agents than NiTi in 24 hrs. They compared the SEM observation on the basis of color changes.
- Nicolas Schiff, Boinet, Morgan investigated NiTi and Cu-NiTi, immersed in Meridol and Elmex mouthwash. They observed highest corrosion risk with Cu-NiTi wire.
- Mary P. Walker; Ries; Kula found corrosive changes in surface topography with topical fluorides (phos-flur gel and prevident). They also studied mechanical properties which showed statistically significant decrease in unloading mechanical properties.

Conclusion

The following conclusions were drawn from the above study:

In Scanning Electron Microscope

- 1) Corrosion and surface roughness were observed in all the wires immersed in Flurovil and Phosflur i.e., fluoride containing oral prophylactic agents.
- 2) No Corrosion and surface roughness was observed in all the wires immersed in GC Tooth mousse and the distilled water.
- 3) The Flurovil appeared to cause greater corrosive effects and surface roughness as compared to Phosflur in each wire sample.
- 4) Maximum corrosion and surface roughness was observed in NiTi, CuNiTi and AJ Wilcock SS wires.

This study suggests that fluoride prophylactic agents, in spite of their established caries preventing activity, must be used with caution in patients undergoing Orthodontic treatment. Extensive use of these agents may cause corrosive effects on the Orthodontic wire surfaces with deterioration of their mechanical properties. This may possibly result in an increase in the overall treatment time. Also, on the basis of the findings of this study it is suggested to use G C Tooth Mousse as a prophylactic agent against the use of fluoride containing agents because of its negligible side effects on the properties of the wire surface. There is scope beyond the preview of this study to evaluate the phenomenon of hydrogen embrittlement of Orthodontic wires which could possibly be responsible for the deterioration of the mechanical properties, via the method of hydrogen thermal desorption analysis. Also, Nickel sensitivity in patients has been documented. Hence the amount of Nickel leaching out due to surface corrosion of NiTi and CuNiTi wires caused by fluoride agents can be evaluated in further studies.

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