Available online at http://www.journalcra.com

International Journal of Current Research
Vol. 11, Issue, 04, pp.3130-3132, April, 2019

DOI: https://doi.org/10.24941/ijcr.35105.04.2019

INTERNATIONAL JOURNAL OF CURRENT RESEARCH

ISSN: 0975-833X

RESEARCH ARTICLE

EFFECT OF HYDROFLUORIC ACID TREATMENT ON SHEAR BOND STRENGTH AND SURFACE CHARACTERSTICS OF TITANIUM ALLOYS: AN IN-VITRO STUDY

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ARTICLE INFO

Article History:
Received 15th January, 2019
Received in revised form
20th February, 2019
Accepted 27th March, 2019
Published online 30th April, 2019

Key Words:
Hydrofluoric acid; Shear bond strength; Surface characteristics; Titanium

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ABSTRACT

Purpose: The purpose of this in-vitro study was to evaluate the effect of Hydrofluoric acid treatment on shear bond strength and surface characteristics of Titanium alloys. **Methods:** 3 dental luting agents representative of different compositional classes (Polycarboxylate, Glass ionomer, and Zinc oxide based cements) were used to evaluate their effect on Titanium-6Aluminum-4 Vanadium (Ti-6Al-4V) alloy surfaces. Samples were divided according to their surface etching with 9.6% Hydrofluoric acid (n=15 for each group). Group (1) Titanium alloy cylinders with acid etching. Group (2) Titanium alloy cylinders without acid etching. 30 pairs of cylinders were cemented together. Shear Bond strength was measured using a Universal testing machine at a crosshead speed of 5mm/min after incubation in a water bath at 37°C for 7 days. SEM was used to observe the representative specimens for the surface characteristics under 500X magnification. **Results:** Polycarboxylate cement showed significantly greater retention than all other groups ($P<0.05$).SEM examination showed surface pits on the Hydrofluoric acid etched Titanium alloy cemented with Polycarboxylate cement. **Conclusion:** Higher Shear bond strength and some chemical surface changes were observed in acid etched Ti alloy group cemented with Polycarboxylate cement.

***Citation: Dr. Priyanka Rai and Dr. Shilpa Shetty.* 2019.** "Effect of Hydrofluoric acid treatment on shear bond strength and surface characterstics of Titanium alloys: An in-vitro study", *International Journal of Current Research*, 11, (04), 3130-3132.

INTRODUCTION

Ti and its alloys are considered one of the best and useful dental restorative material because of their remarkable biocompatibility, passivity, high mechanical strength and relatively lower cost as compared with the noble metal alloys (Wadhwani, 2015). Biocompatibility and passivity of Ti alloys is due to the presence of the highly protective oxide layer which is formed when Ti reacts with Oxygen (Wadhwani, 2015; Turpin *et al.,* 2000).This property has made Ti as one of the material of choice for implant components (Turpin *et al.,* 2000). Metal frameworks which are used for porcelain application are etched with fluoride gels for strong and durable bonds (Turpin *et al.,* 2000). Or implant superstructures are cemented with a variety of dental luting agents. However, no luting agent is considered most appropriate for implants (Wadhwani, 2015). Also, it has been postulated that restoration can be modified by surface treatment with fluoride acid gel to increase surface roughness and surface area to improve their retentive strength with the implant abutment (de Campos, 2010). Therefore, physical and chemical interactions of Ti alloys with dental luting agents and acid fluoride gels must be studied as various reports indicate that several dental luting cements alter the passivity of Ti alloy surfaces (Wadhwani, 2015) and acidic fluoride gels have reported the risk of corrosion of Ti (Turpin *et al.,* 2000) which may lead to implant failure (Wadhwani, 2015). The aim of this in-vitro study was to evaluate the effect of Hydrofluoric acid treatment on shear bond strength and surface characteristics of Ti alloys.

MATERIALS AND METHODS

30 pairs of cylinders made up of Ti-6Al-4V alloy of dimensions 6.0 mm in diameter and 15 cylinders represented the implant abutment component and the remaining half represented the alloy copings. 10.0 mm length was cut from stock of Ti-6Al-4V alloy rods (ASTM Grade IV). Specimens were divided into 2 groups: Etched Group (E) where etching of Ti alloys with 9.6% HF acid (Deor Deo Cera Etch) for 1 min (n=15) and Non-Etched Group (NE) where Ti alloys were not etching (n=15). 3 dental luting agents representative of different compositional classes Polycarboxylate PF (Poly- F Plus; Dentsply DeTrey), Glass ionomer GC (GC Fuji, GC corporation Tokyo, Japan) and Zinc oxide-based cements K (Kerr Temp Bond NE) were used. The dental cements were mixed according to the manufacturer's instructions and was

applied on the surface of cylinder using micro brush (Benda Micro applicators; Centrix Inc) and the excess were removed. 2 joined cylinders were axially loaded with 49N force for 10 mins at room temperature. Cylinder pairs were incubated in a water bath at 37°C for 7 days when once they were set. Using the Universal testing machine (Mecmesin Multi-Test 10-i) shear Bond strength test was subjected on each cemented cylinder pair test specimens at a cross-head speed of 5 mm/min and the peak shear force was recorded. Representative specimens were selected and observed under 500X magnification after sputtering gold conductive layer of appx. 10nm with a scanning electron microscope (Zeiss Ultra 55).

Statistical Analysis: Data were expressed as the mean ± standard deviation (SD). The results were analyzed using software SPSS, version 19 (SPSS Inc. Chicago, IL, USA). Student t- test was used to compare the mean rank between the groups. The level of statistical significance was defined as $P<0.05$.

RESULTS

The mean ± standard deviation (SD) Shear Bond strength values for Group PF ranged from 4.93±1.42 MPa for subgroup E and 1.41±0.50 MPa for Group NE (Table 1).

Table 1. Mean comparison of shear Bond strength (MPa) in Polycarboxylate cement

Group	Mean	SD	T value	P value
Etch	4.93	1.42	5.204	.001*
Non –etch	1.41	0.50		

*Statistically significant

For Group GC ranged from 2.88±0.72 MPa for subgroup E and 1.59±0.41 MPa for subgroup NE (Table 2).

Table 2. Mean comparison of shear Bond strength (Mpa) in Glass ionomer cement

Group	Mean	SD	T value	P value
Etch	2.88	0.72	3.453	.009*
Non –etch	1.59	0.41		

***Statistically significant**

For Group K ranged from 1.95±0.44 MPa for subgroup E and 1.20±0.24 MPa for subgroup NE (Table 3)

Table 3. Mean comparison of shear Bond strength (Mpa) in Zinc oxide based cement

Group	Mean	SD	T value	P value
Etch	1.95	0.44	3.295	.011*
Non –etch	1.20	0.24		

***Statistically significant**

Significantly lowest shear Bond strength was observed in Group Zinc oxide based cement(K) than other test groups ($P<0.05$).In contrast, Group Polycarboxylate cement(PF) showed significantly the highest shear Bond strength than all other test groups ($P<0.05$) (Figure 1). Representative debonded surfaces were examined under SEM where no surface detail changes were observed for Group K (Figure 2) and GC (Figure 3). Whereas, PF group specimens showed micropits on Ti alloy surface which were acid etched with HF acid (Figure 4).

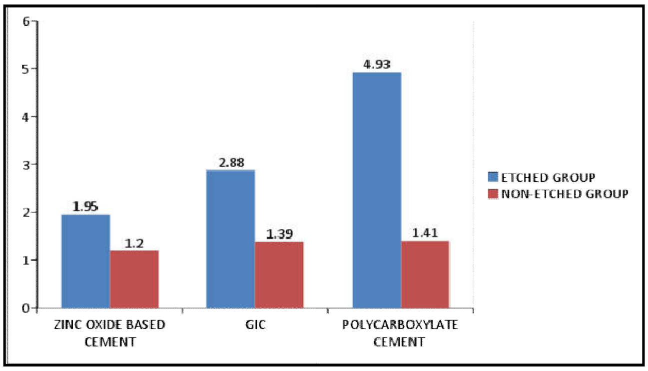

Fig. 1. Mean comparison of shear bond strength among the groups

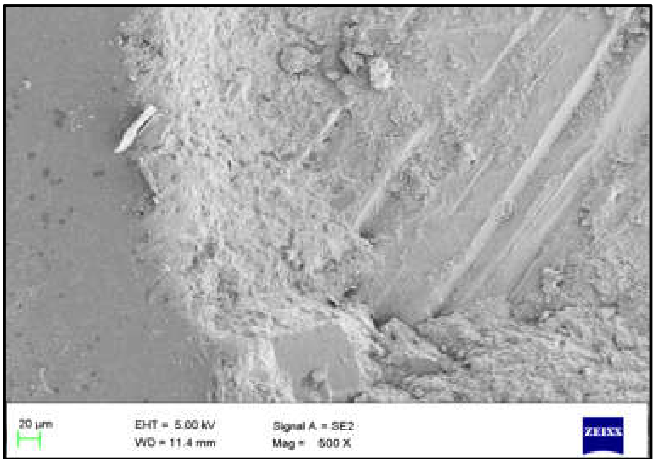

Fig. 2. Representative of K Group. (500X)

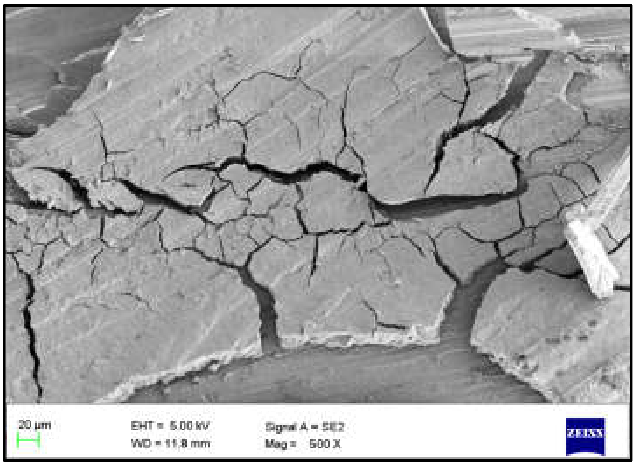

Fig 3. Representative of GC Group. (500X)

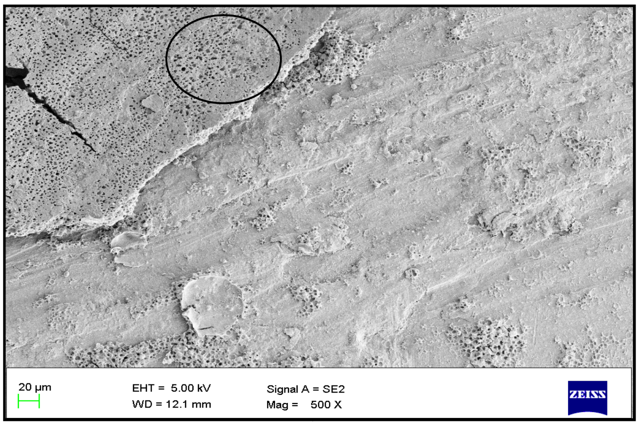

Fig 4. Representative of PF Group. (X500). Surface micropitting indicative of corrosion

DISCUSSION

Results in the present study showed a significant difference in the shear bond strength within the groups. Shear Bond strength test results showed that the retentive capabilities of E group were greater than NE group. Ti alloy surfaces are roughened after etching with HF acid which should increase the actual bonding surface area and the mechanical interlocking (Taira, 2013). Significantly higher shear Bond strength values were observed in Polycarboxylate cement than the other 2 cement groups. Christian Mehl *et al.* concluded from his study that restoration retention is increased when Polycarboxylate cements are used to cement them with Ti abutments (Mehl, 2008). Garg *et al.* also stated that Polycarboxylate cement shows chemical interaction with the Ti alloys thus, improving the retention (Garg, 2013). In the current study SEM images revealed that the surface of Ti alloy was altered physically and chemically with the Polycarboxylate cements and showed pitting corrosion. According to Yohsuke Taira *et al.,* Hydrofluoric acid which are commonly used to clean and etch Ti contain fluorides which act as a corrosive agent (Taira, 2013). Acidic or fluorinated dental cements have also been reported by Y.L Turpin et al to increase the corrosion susceptibility of Ti leading to the failure of implants (Turpin *et al.,* 2000). In the present study Polycarboxylate cement group (PF) have shown the surface pitting corrosion on Ti alloys. This chemical reaction could be due to the presence of Stannous fluoride. Fluoride ions are also present in GIC group (GC) but no such surface changes were observed with them on Ti alloy surfaces. However, some studies have shown contrasting results from the present study. The release of low concentration of fluoride ions and other elements from GIC interact with Ti alloy resulting in tarnishing and discoloration of the surfaces (Turpin *et al.,* 2000). As stated by Wadhwani *et al.* cement selection for implant restorations should be carefully considered because diseases related to periimplant can be an issue (Wadhwani, 2015). Rodrigues *et al.* supported the above statement by concluding in their study that corrosion caused by the cement may lead to implant loss (Rodrigues, 2013).

Conclusion

Within the limitations of this study, the following conclusions may be drawn:

- Higher shear Bond strength was observed after cementation with Polycarboxylate cement.
- Shear Bond strength was higher in Ti alloys cemented after etching with 9.6% HF acid as compared to the control group.
- Cementation of Ti alloys with fluorinated acid cement with Polycarboxylate cement such as Poly F revealed pitting corrosion on the surface of alloy. But no evidence was there for fluorinated GIC to depassivate Ti.

Conflicts of Interest: None

Abbreviations

American Society For Testing Materials**: ASTM**
Etched group**: E**
Glass ionomer cement**: GIC**
Hydrofluoric acid**: HF**
Mega Pascals**: MPa**
Non-etched group**: NE**
Scanning electron microscope**: SEM**
Standard deviation**: SD**
Titanium**: Ti**
Titanium-6 Aluminium-4Vandium**: Ti-6Al-4V**

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