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Evaluation of Surface Cleaning Regimens on Bond Strength of Resin Cement to Zirconia reinforced Lithium Silicate Ceramic – An in Vitro Study

INTRODUCTION

With various advancements in dental adhesives and material science, there has been an increased demand and preference to opt for all - ceramic restorations which are considered as an excellent substitute to traditional metal - ceramic crowns due to their durability and good aesthetics.

As a result of extensive research in novel restorative materials, Zirconia - Reinforced Lithium Silicate (ZLS) ceramics have been introduced with the aim of achieving excellent mechanical and aesthetic properties. [1] This ceramic contains tetragonal zirconia fillers that are added to the crystalline component consisting of a homogenous glassy matrix with round and submicrometric elongated grains of lithium metasilicates and lithium orthophosphates. [1] The grain formation of lithium disilicate follows after a crystallization process.

Apart from material properties, it is the appropriate surface treatment of the intaglio surface that will dictate the longetivity of any bonded restoration. The manufacturer recommends the use of hydrofluoric acid followed by silane application, since ZLS ceramics are acid sensitive. [2] However in regard to the cementation of ZLS ceramics, it is still not known as to which surface treatment will offer sufficient bond strength. [3] Further, it is known that the contamination of the intaglio surface of the prosthesis with saliva during the intraoral try in can compromise the bond strength due to components such as proteins, enzymes, microorganisms and food debris, present in the saliva. [4,5] Although conventionally, the intra-oral try-in of the restoration is performed before applying the Hydrofluoric (HF) acid and silane coupling agent, the restoration may occasionally be etched in the laboratory. [4] This is done in order to make it easier for the practitioner or because chairside use of HF is prohibited in some countries due to its potential hazardous effects. [6] Due to the potentially toxic nature of HF acid and its technique sensitivity, its chairside use should be performed with caution or avoided. [2,5,6] It is therefore necessary to have an efficient alternative to HF acid. [4]

Many alternative cleaning methods have been proposed, such as 37% phosphoric acid and alkaline based cleaning agents such as Ivoclean (Ivoclar Vivadent, Schaan, Liechtenstein), and have shown good results when tested on lithium disilicate ceramics. [4,7] But the cleaning efficiency of these materials has not been tested on ZLS ceramics.

Another material that has been used for the surface treatment of glass ceramics is a self-etching ceramic primer (SECP), available commercially as Monobond Etch & Prime (Ivoclar Vivadent, Schaan, Liechtenstein), that has shown to combine the conditioning effects of hydrofluoric acid and silane, while at the same time offering a safer and less technique sensitive surface treatment compared to HF etching. [2] The manufacturer also claims that the SECP can clear salivary residues from the surface. [8] However, very scarce evidence exists regarding its efficacy. The current study aims to assess how different surface cleaning techniques affected the resin cement's ability to adhere to zirconia-reinforced lithium silicate ceramic.

According to the null hypothesis, ZLS and the resin cement have no difference in shear bond strength across all of the variables examined in the study.

METHODOLOGY

Preparation of ceramic specimens

ZLS CAD/CAM blanks "(Vita Suprinity PC; Vita Zahnfabrik, Germany)" of dimensions 12mm x 14mm x 18mm were used in the study. The blocks were sectioned into slices of 1.5mm thickness to obtain dimensions of 12mm x 14mm x 1.5mm per specimen which was done with the help of a precision cutting saw (Streurs SECOTOM 60; Streurs, USA) under constant water cooling [Figure 1]. A total of 50 specimens were used. Silicon Carbide abrasive papers of #600, #800, and #1200 grits were used to polish the specimens, to obtain standardized smooth surfaces. After that, they were then placed in an ultrasonic immersion with distilled water for ten minutes. The specimens were immersed with their polished surfaces exposed in an autopolymerizing resin after being crystallized in accordance with the manufacturer's instructions. For ten minutes, the specimens underwent additional ultrasonic cleaning.

Saliva Contamination

The samples were distributed arbitrarily among five groups (n=10). Apart from the specimens in group 1, the rest of the groups were subjected to saliva contamination. Groups 4 and 5 were subjected to pre-treatment conditioning prior to contamination. In order to achieve contamination, a protocol similar to the one used in a study conducted by Yoshida et al (2020) was followed, where saliva was collected from a healthy male donor (the principal investigator) who refrained from drinking or eating for two hours prior to the collection. [5] The samples to be contaminated were then kept in the saliva for one minute, followed by rinsing with water-spray for 15 s, and air drying for 10 s.

Surface treatments

The experimental groups were cleaned using one of the following cleaning regimens, either after or prior to saliva contamination:

Group 1: Positive Control. No saliva contamination. Next, for 20 seconds, etching with 10% HF acid, tracked by 20 seconds of water rinsing. The samples were then air-dried for 5 seconds. Silane coupling agent (Silano; Angelus, Brazil) was applied onto the surface for 60 seconds with a microapplicator tip and air-dried for 5 seconds.

Group 2: Saliva contamination followed by etching with 10% HF acid for 20 seconds, followed by 20 seconds of water rinsing. The samples were then air-dried for 5 seconds. Silane coupling agent (Silano; Angelus, Brazil) was applied onto the surface for 60 seconds with a microapplicator tip and air-dried for 5 seconds.

Group 3: Saliva contamination followed by application of self-etching ceramic primer "(Monobond Etch & Prime; Ivoclar Vivadent, Schaan, Liechtenstein)". Using a microbrush, primer was applied onto the ceramic surface for 20 seconds. After a reaction time of 40 seconds, it was rinsed and dried for 10 seconds.

Group 4: Pre-treatment with 10% HF acid for 20 seconds, followed by 20 seconds of water rinsing. The samples were then air-dried for 5 seconds. Samples were then subjected to saliva contamination and subsequent cleaning for 20 seconds with 37% phosphoric acid, followed by water rinsing for 20 seconds. After that, the samples were air-dried for 5 seconds. Silane coupling agent (Silano; Angelus, Brazil) was applied onto the surface for 60 seconds with a microapplicator tip and air-dried for 5 seconds.

Group 5: Pre-treatment with 10% HF acid for 20 seconds, followed by 20 seconds of water rinsing. The samples were then air-dried for 5 seconds. After that, the samples were then subjected to saliva contamination and subsequent cleaning with Ivoclean "(IvocleanTM; Ivoclar Vivadent, Schaan, Liechtenstein)" for 20 seconds, followed by water rinsing for 20 seconds. The samples were then air-dried for 5 seconds. Silane coupling agent (Silano; Angelus, Brazil) was applied onto the surface for 60 seconds with a microapplicator tip and air-dried for 5 seconds.

Adhesive cementation

The composite resin cylinders "(Filtek Z350, 3M ESPE, Saint Paul, USA)" with a diameter of 4mm and a height of 3 mm were constructed using a silicone mold. After that, self-adhesive resin cement "(RelyX U200; 3M ESPE, Saint Paul, USA)" was utilized to attach the cylinders to the specimens. The excessive cement was removed and light polymerized from two lateral directions.

Bond strength test

With a universal testing machine (Tec-Sol, India) at a crossheading speed of 0.5mm/min, the Shear bond strength (SBS) test was carried out after 24 hours. A knife-shaped metal tip was utilized for this purpose [Figure 2]. The SBS standards were recorded in MPa by dividing the failure load (N) by the bonding area (mm2).

RESULTS

The collected data were evaluated using SPSS version 27. For comparing the five groups, One-way ANOVA was utilized ($P < 0.05$ was considered significant). The Continuous variables were defined in terms of mean and standard deviation.

Table 1 shows the mean SBS values. The highest bond strength was shown by Group 5 (20.87 ± 3.13 MPa), followed by Group 3 (17.49 ± 4.50 MPa). The lowest bond strength was seen in Group 4 (15.15 ± 3.08 MPa).

The p-value of the ANOVA test was 0.003, which is significant. Hence, the null hypothesis was rejected. For pairwise comparison to check the difference in mean in different pairs of groups, the Bonferroni numerous evaluation test was used.

Multiple evaluations among the different groups can be seen in Table 2. There was a substantial transformation ($P < 0.05$) between the following groups: Groups 5 (20.87 ± 3.13 MPa) and 1 (15.67 ± 3.62 MPa) ($P = 0.015$); Groups 5 (20.87 ± 3.13 MPa) and 2 (15.50 ±

2.60 MPa) (P = 0.011); Groups 5 (20.87 ± 3.13 MPa) and 4 (15.15 ± 3.08 MPa) (P = 0.006).

DISCUSSION

The present study was conducted with the aim of evaluating the properties of various surface cleaning regimens on the bond strength of resin cement to zirconia-reinforced lithium silicate ceramic. According to the findings, the mean SBS values varied depending on the surface behavior. Hence, the null hypothesis was rejected.

From the clinical standpoint, there are various contaminants that can interfere with the bonding of ceramic prosthesis. Amongst them all, saliva is considered to be quite relevant and detrimental to the bond strength. [4,5] Apart from the various components found in saliva, the thin salivary film is known to block the hydroxyl sites on the ceramic surface, ultimately decreasing the hydrophilicity of the ceramic. [9] Rinsing with water spray is considered to be insufficient in the removal of the saliva layer from the intaglio surface of the prosthesis after the clinical try-in. [4]

In the current study, Group 1 (positive control group) was not subjected to contamination. Conventional surface treatment using HF acid for etching, surveyed by application of silane coupling agent, in these uncontaminated specimens, showed mean bond strength values greater than those observed in Group 2, where the specimens were contaminated with saliva prior to the same surface treatment. However, the transformation among the groups was not found to be significant. This shows that cleaning contaminated specimens with HF acid, trailed by application of the silanising agent, provided bond strength values comparable to those of the control group.

Group 4, where the pre-treated specimens were contaminated and later cleaned with 37 % phosphoric acid prior to the use silane, showed the lowest mean bond strength values, although not significantly different from that of the control. Phosphoric acid has been proposed as a ceramic cleaning agent as it is an organic solvent and has the potential to remove adsorbed proteins by coagulation. [5,9] However, the lower bond strength values seen in this study could be attributed to the fact that phosphoric acid might alter the ceramic surface energy. [10] It has been reported previously that phosphoric acid has the potential to react with zirconia based ceramics and leave behind a phosphorus residue. [11] This residue is detrimental to the adhesion between 10-MDP based resin cement and zirconia based ceramic. [11] This may have compromised the bond between the self-adhesive resin cement and ZLS ceramic in the current study hence leading to lower bond strength values.

Although the findings of this study show that HF acid conditioning is sufficient in cleaning the contaminated surface, there is a need to find an alternative to HF acid, given the various drawbacks and hazards associated with it. [2] The self-etching ceramic primer has been introduced to serve this purpose. It utilizes ammonium polyfluoride and silane in a single component system, ensuring standardization and providing simultaneous etching and silanization, while eliminating the potential hazards and technique sensitivity associated with HF acid. [12] The product introduced as Monobond Etch & Prime (Ivoclar Vivadent, Schaan, Liechtenstein) contains a methacrylate functionalized silane, which is hydrolyzed methacryloxypropyl trimethoxysilane (MPTMS) as well a bipodal bis-triethoxysilyl ethane (BTSE), as mentioned in the safety data sheet. [13,14] This “bridged” bipodal silane has two silane substitutions and can enhance the crosslinking capacity of the newly formed siloxane network. [13] It is reported that such silanes can retain adhesive properties of MPTMS while improving the hydrolytic stability. [13] In the present study, the greater bond strength values achieved by Group 3, which used the SECP, compared to the bond strength values of Groups 1, 2, 4 may be attributed to these factors. There have been many studies conducted which show that the SECP can provide adequate bond strength and can be considered as an safer substitute to conventional HF acid conditioning of glass ceramics such as lithium disilicate and ZLS. [2,3,13,15,16,17,18,19] However, the effectiveness of the SECP conditioning on saliva contaminated ZLS ceramics has previously not been reported in literature. The results of this study show that the SECP can promote removal of salivary residues on the ceramic surface and deliver bond strength values similar to or higher than that of HF acid and silanization protocols.

In the current study, Group 5, where the alkaline based cleaning solution, Ivoclean (Ivoclar Vivadent, Schaan, Liechtenstein) was used in the decontamination protocol, showed the highest mean bond strength values. The mean SBS value obtained was greater than all other tested groups, and was significantly greater than all groups except that of Group 3. It is a suspension containing zirconium oxide particles which removes salivary phosphates through adsorption due to its strong affinity. [3,5] It may also potentially remove protein residues through dissolution by the sodium hydroxide component. [5] The findings of this study corroborate with the findings of previous research where Ivoclean cleaning paste was found be effective in removal of salivary contaminants from the ceramic surface. [5,7,10,20]

However, there are certain drawbacks to this in-vitro study. Aging methods such as thermocycling and fatigue testing should be included in further studies as a means to determine long term outcomes of various surface conditioning techniques.

CONCLUSION

Overall, the outcomes of this study propose that it may be beneficial to use an alkaline cleaning agent such as Ivoclean, prior to the silanization of pretreated ZLS ceramic, which has been contaminated by saliva during the clinical try - in phase of the prosthesis. The results also show that the self-etching ceramic primer can be an effective substitute to the chairside use of HF acid for

surface cleaning of contaminated specimens, thereby simplifying the clinical technique and reducing the overall chairside time and steps involved.

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