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The Effect of Different Hemostatic Agents on Shear Bond Strength Between Resin Composite and Lithium Disilicate Ceramic

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Abstract

The objective of this study is to evaluate the shear bond strength between resin bonded and lithium disilicate ceramic that was contaminated with hemostatic agents. Forty ceramic discs were prepared by being treated with 5% HF for 20 seconds and applying silane coupling agent (ClearfilTM Ceramic Primer Plus). All specimens were randomly divided into four groups, namely, Group 1: control, Group 2: aluminum chloride, Group 3: aluminum sulfate, and Group 4: ferric sulfate. All groups were cemented with resin cement (Panavia V5), and then the shear bond strength was measured using a universal testing machine at a crosshead speed of 0.5 mm/min. The results were statistically analyzed using one-way ANOVA and Tukey's multiple comparison tests at a 95% confidence level. The shear bond strengths of Groups 2-4 were significantly lower than the control group (P<0.05). There was no significant difference between Group 2 and Group 3, whereas Group 4 had the lowest shear bond strength. From stereomicroscopic images, most specimens represented mixed failure.

Keywords: Contamination, Hemostatic agent, Lithium disilicate

1. Introduction

Nowadays, all-ceramic restorations are commonly used in dentistry because of their excellent properties such as aesthetic, biocompatibility and acceptable mechanical properties (Giordano & McLaren, 2010; Guess et al., 2011). Several types of ceramics have been developed so far.

Lithium disilicate ceramic is a synthetic glass-ceramic which is frequently used in dentistry. It was first launched as Empress 2 (Ivoclar Vivadent) in 1998. After that, IPS e.max[®] Press (Ivoclar Vivadent) was introduced in 2005 to improve its properties. The main compositions of lithium disilicate ceramic include 70% needle-shaped crystals and 30% glass phase (Guess et al., 2011). It can be formed by two techniques: heat pressed and computer-aided design and computer-aided manufacturing (CAD/CAM). Due to its acceptable properties, it can be used in various restorations such as inlay, onlay, crown, veneer, or other fixed partial dentures (Conrad, Seong, & Pesun, 2007).

Before the restoration is cemented, the surface treatment of glass-ceramic should be done. It is treated with hydrofluoric acid (HF) and a silane coupling agent to improve the bond strength (Barghi, 2000; Matinlinna, Lung & Tsoi, 2018; Sriamporn et al., 2019). Many laboratories usually etch the inner surface of a glass-ceramic restoration with HF before sending the restoration to the dental clinic (Alfaro, 2014). Furthermore, applying a silane coupling agent immediately after HF etching before a clinical try-in step is a well-recommended method (Aboush, 1998; Alfaro, 2014; Nubdee, Thamrongananskul, Wattanasrimkit & Mekayarajjananonth, 2017). Many previous studies reveal that contamination from any contaminants, blood, saliva, chemical agents can reduce the bond strength of restoration with the tooth substrate (Eiriksson, Pereira, Swift Jr, Heymann & Sigurdsson, 2004; Harnirattisai, Kuphasuk, Senawongse & Tagami, 2009; Nicholls, 1988; Yoshida et al., 2015). Therefore, the contaminants should be removed or avoid contacting the bonded area. In general, a rubber dam is used to isolate the working field. However, in some situations, for example, filling in a subgingival area or where the margin of restoration is in a subgingival area, one of the most common procedures used for the isolation is retracting with gingival cord soaked with hemostatic agents. This procedure can prevent the entry of gingival fluid and blood in the tooth cavity.

In dentistry, several hemostatic agents are frequently used to control bleeding such as aluminum chloride, aluminum sulfate, or ferric sulfate (Tarighi & Khoroushi, 2014). Some studies indicate that the

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residue from these hemostatic agents can affect the bond results and the quality of restoration (Chaiyabutr & Kois, 2011; Kuphasuk et al., 2007). Sometimes, the residues from these hemostatic agents remain on the restoration surface after the surface cleaning procedure. Therefore, the current study aims to evaluate the effect of hemostatic agents on the shear bond strength between composite resin and lithium disilicate ceramic. The null hypothesis is that there is no significant difference in shear bond strength between hemostatic agent-contaminated ceramic and non-contaminated ceramic.

2. Objective

1. To evaluate the shear bond strength between resin bonded and lithium disilicate ceramic contaminated with hemostatic agents.

2. To evaluate the shear bond strength between resin bonded and lithium disilicate ceramic contaminated with different hemostatic agents.

3. Materials and Methods

3.1 Shear bond strength testing

Forty lithium disilicate ceramic disks were prepared by pressing ceramic ingot shade MO 0 (IPS e.max Press, Ivoclar-Vivadent AG, Japan). These ceramic disks were formed into 5 mm in diameter and 3 mm in depth. Flutings were created under the specimens to retain them with the type III dental gypsum. Polyvinyl chloride (PVC) tube with 22 mm in diameter was cut into 10 mm in height and then filled with dental gypsum. The specimen was placed into the center of the tube where the surface of the specimen was 1 mm higher than the surface of dental gypsum. After dental gypsum had completely set, the surface of specimens was polished with silicon carbide abrasive paper 400-grit and 600-grit respectively by using an automatic polishing machine (Mecatech 234 polishing machine, Presi, France) with water as a lubricant. During the polishing process, silicon carbide abrasive papers were set to rotate counterclockwise at a rate of 100 rotations per minute with a force of 1 kg/cm² while lithium disilicate specimens were set to rotate at the same rate but in the opposite direction. The polishing process was performed for 5 minutes by using 400-grit silicon carbide abrasive paper and then repeated with 600-grit silicon carbide abrasive paper. The new abrasive paper was used for each polishing time. After that, all specimens were cleaned in distilled water by using an ultrasonic cleaner and then air-dried. For each group, 5% HF (IPS® Ceramic Etching Gel Ivoclar Vivadent AG, Liechtenstein) was applied on the ceramic surface and then rinsed with distilled water and air-dried. Next, a layer of silane coupling agent (ClearfilTM Ceramic Primer Plus, Kuraray Noritake Dental Inc., Japan) was used and air-dried with 40-50 lbs/inch² water / oil-free air blow. After that, for Group 2-4, each hemostatic agent was applied and left for five minutes (Figure 1). The specimens were randomly divided into four groups (n = 10):

> Group 1: Control group Group 2: Aluminum chloride (Racestyptine, Septodont, Cedex, France) Group 3: Aluminum sulfate (Alstringent, Alan & co., Verviers, Belgium) Group 4: Ferric sulfate (Viscostat[®] Ultradent Products, Inc)

One-sided tape (Scotch blue Painter's Tape, 3M, Minnesota, USA) with 10*10 mm in size and $80 \mu \text{m}$ in thickness with 2 mm diameter hole at the center of the tape was attached to the treated lithium disilicate ceramic surface. Composite resin blocks were fabricated by pressing composite resin into a silicone mold (Elite HD, Zhermack, Badia Polesine, Italy) with the hole at the center size 2 mm in diameter and 2 mm in depth. Next, resin cement (PanaviaTM V5, Kuraray Noritake Dental Inc., Japan) was applied through a mixing tip on the surface of the composite resin block. Following that, it was placed on the treated ceramic surface to cover the hole of the tape. Afterward, 1 kg weight was placed onto the composite resin block. Next, the excess cement was removed by using a micro brush and light-curing for 20 seconds on each surface. The bonded specimens were immersed in distilled water and kept in an incubator (Contherm 160M, Contherm Scientific Ltd., Korokoro, Lower Hutt, New Zealand) at 37°C for 24 hours. The materials used in this study are summarized in Table 1.

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Figures 1 Schematic diagram representing the experimental procedure and testing groups in this study

Table 1 Products and their compositions used in this study

Products	Company	Lot No.	Compositions
Lithium disilicate ceramic (IPS e.max [®] Press)	Ivoclar Vivadent AG, Liechtenstein	W02583	Lithium disilicate crystals (approx. 70%), Li ₂ Si ₂ O ₅ , embedded in a glassy matrix. Standard compositions: SiO ₂ , Li ₂ O, K ₂ O, P ₂ O ₅ , ZrO ₂ , ZnO, other oxides and ceramic pigments
Resin cement (Panavia TM V5)	Kuraray Noritake Dental Inc., Japan	AN0018	Mixture: bisphenol A diglycidylmethacrylate (Bis-MA), triethylene glycol dimethacrylate (TEGDMA), Silanated barium glass filler, Silanated fluoroalminosilicate glass filler, Colloidal silica, Surface treated aluminum oxide filler, Hydrophobic aromatic dimethacrylate, Hydrophilic aliphatic dimethacrylate, dl-Camphorquinone, Initiators, Accelerators, Pigments
Hydrofluoric acid (IPS® Ceramic Etching Gel)	Ivoclar Vivadent AG, Liechtenstein	W95356	<5 % hydrofluoric acid
Composite resin (Estelite Σ Quick)	Tokuyama Dental Corporation	203E46	(1-Methylethylidene)Bis[4,1-Phenyleneoxy(2-Hydroxy- 3,1-Propanediyl)] Bismethacrylate, 2,2'- Ethylenedioxydiethyl dimethacrylate, Mequinol
Silane coupling agent (Clearfil TM Ceramic Primer Plus)	Kuraray Noritake Dental Inc., Japan	6K0036	Ethanol, 3-trimethoxysilylpropyl methacrylate, 10- Methacryloyloxydecyl dihydrogen phosphate (10-MDP)
Hemostatic agent 1: Aluminum chloride (Racestyptine)	(Septodont, Cedex, France)	BG7KB	Hexahydrated aluminum chloride 25% W/V, oxyquinol, hydroalcoholic excipient
Hemostatic agent 2: Aluminum sulfate (Alstringent)	Alan & co, Belgium	ALST20	25% Aluminum sulfate solution
Hemostatic agent 3: Ferric sulfate (Viscostat [®])	Ultradent Products, Inc.	BGJ62	20% Ferric sulfate solution

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All specimens were presented for the shear bond strength testing by a universal testing machine (EZ-S 500N, Shimadzu Corporation, Kyoto, Japan). After the specimen had been fixed in the machine, the shearing blade was placed parallel to the junction between composite resin and lithium disilicate ceramic at a distance of 1 mm A shear load was applied until failure occurred at a crosshead speed of 0.5 mm/minute. The shear bond strength (MPa) was calculated by dividing the maximum ultimate load to failure by the bonded cross-sectional area. The means and standard deviations were recorded. After the shear bond strength testing, the fractured surface areas at lithium disilicate ceramic/resin cement interfaces were examined by using a stereomicroscope (SZ 61, Olympus Co., Japan) at x40 magnification to acquire the mode of failure. The modes of failure were classified into three categories: (1) Adhesive failure, (2) Cohesive failure, (3) Mixed failure. The data were subjected to one-way analysis of variance (ANOVA), followed by Tukey's HSD to determine the significant differences among the testing groups at a confidence level of 95%.

4. Results and Discussion

The means and standard deviations of shear bond strength are presented in Table 2. One-way analysis of variance revealed a significant difference among the groups. Hemostatic agents contaminated groups (Group 2-4) revealed significantly lower shear bond strength than the control group (P < 0.05). No statistically significant difference was found between Group 2 (surface contaminated with aluminum chloride) and Group 3 (surface contaminated with aluminum sulfate). The lowest mean bond strength was found in Group 4 (surface contaminated with ferric sulfate) with significant differences among the other groups at P < 0.05.

The failure modes were evaluated by using a stereomicroscope. The distribution of the failure modes is shown in Table 3. Also, Figures 2 and 3 show Stereomicroscopic images at 40x magnification of the samples from experimental groups. The modes of failure were classified into three categories: (1) Adhesive failure at the ceramic/resin cement interface, of which the ceramic surface revealed more than 75% throughout the surface, (2) Cohesive failure within resin cement, of which >75% of resin cement was found on the ceramic surface, and (3) Mixed failure, partially adhesive and cohesive failure, of which neither unveiled more than 75% of ceramic nor resin cement surface. Calculated the area by using the ImageJ program.

Groups	Shear bond strength in MPa)SD(
Group 1: Control	22.41 ± 2.92^{a}	
Group 2: $HF + silane + AlCl_3$	8.41 ± 2.83^{b}	
Group 3: $HF + silane + Al_2(SO_4)_3$	8.04 ± 3.39^{b}	
Group 4: $HF + silane + Fe_2(SO_4)_3$	$3.34\pm2.83^{\circ}$	

Table 2 The means and standard deviations of shear bond strengt
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*Superscript letters following mean values: the same letter indicates not significantly different at p-value 0.05

Table 3 Mode of failure for different lithium disilicate ceramic surface treatment	
Mode	of failure

		widde of failure				
Cohesive (resin cement)	Cohesive (ceramic)	Mixed				
-	-	100				
-	-	100				
-	-	100				
-	-	90				
	Cohesive (resin cement) - - - - -	CohesiveCohesive(resin cement)(ceramic)				

 $AlCl_3 =$ aluminum chloride, $Al_2(SO_4)_3 =$ aluminum sulfate, $Fe_2(SO_4)_3 =$ ferric sulphate

From this study, the effect of the shear bond strength between resin cement and lithium disilicate ceramic contaminated with hemostatic agents was investigated. The shear bond strength test was carried out because it is a simple procedure. The specimens are created uncomplicatedly, and the failure which commonly occurs within the mouth is usually caused by shear force (Braga, Meira, Boaro & Xavier, 2010; Lee, Han, Chang & Son, 2017). The study demonstrates the bonding procedure. Proper isolation of the working field during cementation is required. Using the hemostatic agents is one of the most common procedures to control

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bleeding (Chaiyabutr & Kois, 2011). Aluminum chloride, aluminum sulfate, and ferric sulfate were used as hemostatic agents in this study since they are frequently used due to their effectiveness to control bleeding (Kuphasuk, Harnirattisai, Senawongse & Tagami, 2007; Nowakowska, Saczko, Kulbacka, & Choromanska, 2010; Tarighi & Khoroushi, 2014). These hemostatic solutions were applied for five minutes according to the company instruction and in clinical operation (Chaiyabutr & Kois, 2011; Saad et al., 2019). The null hypothesis, which is that there is no difference among groups, was rejected. In the current study, the control group had the highest bond strength because it was not contaminated. The contaminated hemostatic groups had a significantly lower bond strength. Many previous studies indicate that the contamination during the bonding procedure by agents, such as saliva, blood, zinc oxide eugenol, or a hemostatic agent, could impair the bond strength (Eiriksson et al., 2004; Harnirattisai et al., 2009; Nicholls, 1988). Groups 2, 3, and 4 were contaminated with aluminum chloride, aluminum sulfate, and ferric sulfate, respectively. These three groups had a significantly lower bond strength than the control group because of their contamination. It has been reported that any remnants from a hemostatic agent can disturb the bond strength between tooth and restoration (Chaiyabutr & Kois, 2011; Kuphasuk et al., 2007; Nowakowska et al., 2010; O'Keefe, Pinzon, Rivera & Powers, 2005; Tarighi & Khoroushi, 2014). Also, many studies demonstrated that when aluminum in hemostatic agent remained on the substrate surface, a decrease in bond strength might have occurred (Chaiyabutr & Kois, 2011; Harnirattisai et al., 2009; Kuphasuk et al., 2007). According to these studies, energy dispersive spectrometer (EDS) analysis was performed. It was found that there is more aluminum content in the groups of hemostatic agent contaminated dentin than non-contaminated groups. There was no statistically significant difference in shear bond strength between Group 2 and Group 3, but these groups had significantly higher bond strength than Group 4. In this study, it was found that the ceramic surface contaminated with ferric sulfate resulted in the lowest bond strength. The previous study showed that contamination with ferric sulfate yielded the lowest bond strength after being cleaned with different methods (Pucci et al., 2016). The reason was that the gel consistency of ferric sulfate-based hemostatic solution had a thickener in its composition. The previous study reveals that specimens contaminated with ferric sulfate dropped in bond strength when compared with the control group (Pucci et al., 2016). It is previously reported that the maximum microleakage was found on specimens contaminated with a ferric sulfate-based hemostatic agent (Kumar, Shenoy & Joshi, 2012). In the current study, resin cement and composite resin block were used because a 1 kg weight must be placed to control the force we applied and to control film thickness of the resin cement. So, a 1 kg weight must be placed on a hard object which is composite resin block. Besides, using composite resin block instead of a bulk of resin cement can reduce the voids that might be occurred. Besides, the quality of the bond adhesion can estimate by the mode of failure after the specimen fracture. In this study, there was any fracture that occurred within ceramic or resin cement. So, cohesive failure in ceramic or cohesive failure in resin cement cannot be found in this study. The mode of failure for all the groups in this test was predominantly the mixed failure type when a remnant of resin cement was detected in at least one area of the ceramic specimen. The lowest shear bond strength was shown by Group 4 as confirmed by microscopic images showing the adhesive failure of this group.



Figures 2 Stereomicroscopic images A and B demonstrate an adhesive failure of a cross-sectional ceramic at 40x magnification. (C = Ceramic)8

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Figures 3 Stereomicroscopic images A and B demonstrate a mixed failure of a cross-sectional ceramic at 40x magnification. The red area in the right represents the resin cement remnants. (C = Ceramic, R = Resin cement)

However, the limitation of this study was that the researcher investigated only one substrate, a ceramic substrate. Clinically, the restoration must be attached to the tooth substrate. Different results might be obtained if the substrate had been changed. This topic should be further investigated. Also, the substrates contaminated with the hemostatic agents should be cleaned before the bonding procedure. Consequently, there should also have a further investigation into the cleansing agents or methods to remove hemostatic agents prior to the bonding procedure.

5. Conclusion

It is found that the hemostatic agents affected the shear bond strength between resin cement and lithium disilicate ceramic. The shear bond strength of the groups that were contaminated with hemostatic agents was significantly lower than the control group. Among all, the group that was contaminated with ferric sulfate had the lowest shear bond strength.

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