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Shear Bond Strength between Feldspathic CAD/CAM Ceramic and Human Dentine for Two Adhesive Cements

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Keywords
Feldspathic ceramic; CAD/CAM technology; adhesive systems; luting agents; shear bond strength.

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Abstract

Purpose: The purpose of this study was to evaluate the shear bond strength values between dentin substrate and a feldspathic ceramic material, based on computer-assisted design and manufacture (CAD/CAM) technology, bonded together with two adhesive systems coupled with two dual-polymerized luting agents. In addition, the effect of a silane coupling agent on bond strength was evaluated.
Material and Methods: Forty cylinders (6 mm in diameter, 5 mm thick) obtained from feldspathic ceramic blocks were cemented to the dentin of 40 recently extracted human teeth stored in saline solution at room temperature until testing. The specimens were randomly divided into four groups of ten teeth each. All specimens were airborne-particle abraded and etched with hydrofluoric acid. In the first two groups (A1, A2) 20 ceramic cylinders were cemented using Excite DSC and Variolink II; in the A2 group the bonding surfaces were also treated with a silane coupling agent. In Groups B1 and B2, 20 ceramic cylinders were cemented using Scotchbond MPP and RelyX ARC; in the B2 group the bonding surfaces were also treated with a silane coupling agent as in Group A2. All cemented specimens were submitted to a shear bond strength test to check the strength of adhesion between the two substrates, dentin and ceramic. The data were analyzed with two-way analysis of variance ($p < 0.05$).
Results: The mean values of the shear bond strength were (in MPa): 22 ± 7 for Excite DSC/Variolink II without silanization (Group A1); 29 ± 3 for Excite DSC/VariolinkII with silanization (Group A2); 22 ± 4 for Scotchbond MPP/RelyX ARC without silanization (Group B1); and 26 ± 5 for Scotchbond MPP/RelyX ARC with silanization (Group B2). Two-way ANOVA revealed a significant effect of silanization ($p < 0.01$) and did not reveal any significant effect for either the bonding agents ($p > 0.1$) or the interaction between silanization and bonding agent ($p > 0.05$). Multinomial logit model did not show any statistical effects on the failure mode by the shear bond strength ($p > 0.1$). The hypotheses of independence between failure mode (cohesive vs. adhesive) and both the adhesive system ($p < 0.05$) and silanization ($p < 0.05$) were rejected by Pearson's chi-square test.
Conclusion: Within the assumptions and limitations of this study (including the small number of specimens) both bonding systems used achieved good shear bond strength values. The application of a silane coupling agent on the ceramic surface after etching with hydrofluoric acid increased the adhesion strength with both adhesive materials used.

In recent years, researchers have tried to achieve a more effective and longer-lasting adhesion between restorative materials and dental substrate. The adhesive techniques are based on research on the hybrid layer and on chemical and mechanical adhesion. Some researchers have attempted to shorten the application time and reduce the number of steps,[1] creating new generations of materials and improving their quality. Increasing demand for esthetic restorations has led to greater use of all-ceramic materials because of their improved biocompatibility and optical properties, compared with metal-ceramic restorations.[2]

Advances in computer-aided design (CAD) and computer-aided manufacturing (CAM) systems are providing new options for dentistry, creating an alternative to the conventional impression and casting technique for producing dental restorations.[3] A requirement for the successful function of a ceramic

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restoration is adequate adhesion between ceramic and tooth substance;[4] however, the literature is unclear on which cement, ceramic, conditioning treatment, and dentine bonding agent produce the highest bond strength. Resin composite cements are used to lute conventional metal crowns, fixed partial dentures, ceramic crowns, and veneers and to repair fractured metal-ceramic restorations.[5] Resin cements have been selected for their advantageous mechanical[6] and adhesive properties compared with conventional luting agents.[7] They have shown good marginal integrity and low microleakage.[8,9] The use of resin luting agents also appears to be essential in determining an effective stress distribution, which will prevent crack initiation.[10,11] Bond strength to ceramic material is influenced by the composition of the ceramic substrate as well as by mechanical and chemical interaction between substrate and bonding agent.[12]

A strong resin bond relies on micromechanical interlocking and chemical bonding to the ceramic surface and requires roughening and cleaning for adequate surface activation.[13] Common treatment options are grinding, abrasion with diamond rotary instruments, airborne-particle abrasion with aluminium oxide, acid etching, silanization, or a combination of any of these methods.[2,14,15] Some articles have demonstrated that the preferred surface treatment methods for feldspathic ceramic is acid etching with hydrofluoric acid solutions and subsequent application of a silane coupling agent.[16-18] Etching hydrofluoric acid should selectively dissolve glassy or crystalline components of the ceramic and produce a porous irregular surface;[19] this would increase the surface area and facilitate the penetration of the resin into the microretentions of the etched ceramic surfaces,[4] thus improving wettability.[15] Together with etching, silane treatment has been considered efficacious in the bonding of composites to conventional feldspathic porcelains.[20]

Silane coupling agents are adhesion promoters, capable of forming chemical bonds with organic and inorganic surfaces. Bonding to the resin is affected by an addition polymerization reaction between methacrylate groups of the matrix resin and the silane molecule during curing of the composite. The bond with ceramics occurs by means of a condensation reaction between the silanol group (Si–OH) of the ceramic surface and the silanol group of the hydrolyzed silane molecule, creating a siloxane bond (Si–O–Si) and producing a water molecule byproduct.[21] Moreover, silanization would promote wetting of the ceramic surface, thus enhancing the flow of the low-viscosity resins.[22]

The purpose of this study was to evaluate the shear bond strength values between the dentin substrate and a CAD/CAM ceramic material, bonded together with two adhesive systems coupled with two dual-polymerized luting agents. Moreover, the effect of a silane coupling agent on the bond strength was evaluated.

Materials and methods

Materials used and their descriptions are listed in Table 1. In this study 40 cylinders, (6 mm in diameter, 5 mm thick) were milled from blocks of ceramic Vitablocs Mark II with CAD/CAM technology; they were subsequently cemented to the dentin of recently extracted human teeth stored in saline solution. The bonding surface of each ceramic cylinder was airborne-particle abraded (BASIC Professional IS, Renfert, Hilzingen, Germany) with 110-μm aluminum oxide particles under a pressure of 2.5 atm. Each sample was then air-cleaned to remove any debris. The bonding ceramic substrates were then etched by dabbing a 5% hydrofluoric acid gel (IPS Ceramic Etching Gel) for 60 seconds, thoroughly rinsed for 30 seconds to remove the residual acid after etching and then air-dried. One operator carried out all procedures. According to a list of randomization,[23] the specimens were divided into four groups (A1, A2, B1, B2), each one formed by 10 cylinders.

Group A1: Excite DSC was applied in a thin layer on the bonding surfaces of the ceramic (Fig 1A) after the activation

Table 1 Materials used

Material	Manufacturer	Lot number	Chemical composition*
Vitablocs Mark II	Vita Zahnfabrik, Bad Säckingen, Germany	240	Modified feldspar frits and inorganic pigments molten in feldspar matrix. Aluminum-anodized attachment
IPS Ceramic Etching Gel	Ivoclar Vivadent, Schaan, Liechtenstein	F65924	1.0 g contains: <5 % hydrofluoric acid
Excite DSC	Ivoclar Vivadent, Schaan, Liechtenstein	GO3701	HEMA, dimethacrylates, phosphonic acid acrylate, silicon dioxide, initiators and stabilizers, alcohol
Variolink II	Ivoclar Vivadent, Schaan, Liechtenstein	G03961	Paste of dimethacrylates (BisGMA, UEDMA, TEGDMA), benzoylperoxide, ytterbium trifluoride, inorganic fillers, initiators, stabilizers, pigments
RelyX Ceramic Primer	3M ESPE, St. Paul, MN	3UP	Ethyl alcohol, water
Scotchbond MPP	3M ESPE, St. Paul, MN	Etchant: 4BW	Etchant: water, phosphoric acid, synthetic amorphous silica
		Primer: 4AL	Primer: HEMA, water, copolymer of acrylic and itaconic acids
		Adhesive: 4NR	Adhesive: HEMA, BisGMA
RelyX ARC	3M ESPE, St. Paul, MN	CXEF	Silane-treated ceramic, BisGMA, TEGDMA, silane-treated silica functionalized dimethacrylate polymer

*The chemical composition information was obtained from the manufacturers' Material Safety Data Sheet.

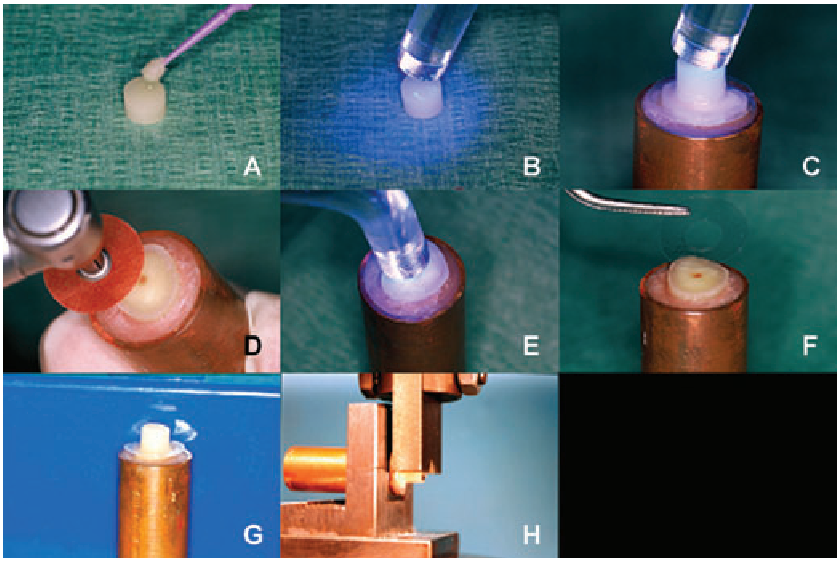

Figure 1 (A) Bonding agent applied on the ceramic substrate. (B) Bonding agent light-polymerized on the ceramic surface. (C) Polymerization of resin luting agent. (D) Dentin surface finished off with paper discs. (E) Bonding agent light polymerized on the dental surface. (F) Vinyl ring applied on the dentin surface to standardize the adhesion area. (G) Completed specimen. (H) Specimen fixed in a jig for the shear test.

of the single-dose unit and was polymerized for 20 seconds (Fig 1B). Then the ceramic specimens were cemented on a dentin surface, which had been treated with the same adhesive, with the dual-polymerization resin luting agent Variolink II.

Group A2: The same adhesive and luting agents were used as in Group A1. The bonding surfaces of the ceramic were also treated with a silane (RelyX Ceramic Primer) coupling agent for 60 seconds. As confirmed by its expiration date, the silane agent was not more than 2 months old at the time of the experiment. Shen et al, Barghi et al, and Roulet et al have demonstrated that the heat treatment of the silanized surface at 100°C for 60 seconds causes a significant improvement in the shear bond strength.[16,18,19] This is attributed to the elimination of water and other contaminates such as alcohol or acetic acid from the silane-treated surface during heating. Removal of water should drive the silane/silica surface condensation reaction toward completion and promotes covalent silane/silica bond formation. Evaporation of compounds such as alcohol and acetic acid, which otherwise hydrogen-bond to the silica surface, increases the number of bond sites available for reaction with silane.[16,18,19] This study was limited to the investigation of the effect of silanization without heat treatment. The surfaces were then air-dried.

Group B1: Scotchbond MPP was applied on the bonding surfaces of the ceramic and was polymerized for 20 seconds. Then the cylinders were cemented on a dentin surface, which had been treated with the same adhesive, with the dual-polymerization resin luting agent RelyX ARC.

Group B2: The same adhesive and luting agents were used as in Group B1. The bonding surfaces of the ceramic were also treated with a silane (RelyX Ceramic Primer) coupling agent for 60 seconds. The surfaces were then air-dried.

During cementation, the catalyst and the base of both cements were dispensed onto a glass mixing pad and mixed for 10 seconds with a stainless steel spatula. A thin layer of cement was applied and distributed to the bonding surface of the ceramic specimens by means of a Hideman spatula. On each specimen, five surfaces were identified: mesial, lingual, distal, buccal, and occlusal. Every surface was light-polymerized for 1 minute (Fig 1C) with a Mini-LED lamp (Satelec, Mérignac-Cedex, France). Light-intensity was 800 mW/cm^2. The power output (light intensity) of the Mini-LED was measured with a Cure Rite radiometer (Caulk-Dentsply mod. 644726, Konstanz, Germany).

Forty recently extracted human molars were scaled using a hand instrument and were then stored in saline solution at room temperature until testing. The roots of each tooth were embedded in a copper cylinder with an external diameter of 16 mm and a height of 40 mm, filled with an autopolymerized acrylic resin (Pro Base, Ivoclar Vivadent, Schaan, Liechtenstein); the crowns were sectioned 90° to the long axis of the teeth with a diamond rotary cutting instrument of medium grit (Intensiv FG 859 D3, Lot B-0147, Intensiv SA, Grancia, Switzerland) to expose the dentin surface. The dentin surface was finished off with Sof-Lex Pop On XT paper discs (3M ESPE, St. Paul, MN) (Fig 1D) with a diameter of 12.7 mm and a grit of 50–90 μm; this procedure created a very smooth surface and reduced any micromechanical interlocking that could affect the real bonding influence of the tested adhesive cements. The dentin surface was treated for 1 minute with a cotton pellet impregnated with Tubulicid Blue (Dental Therapeutics AB, Saltsjö-Boo, Sweden) without fluorine, a cleaner that removes the smear layer without opening the dentinal tubules.[24] The surface was then rinsed. The dentin substrates were acid-etched with 35% phosphoric acid (3M ESPE) for 15 seconds. The etched substrates were rinsed with water and gently air dried to remove excess water. After the activation of the single-dose unit, the single-component adhesive Excite DSC was applied in a thin layer on 20 teeth (Groups A1 and A2). Scotchbond MPP was applied on the other 20 teeth (Groups B1 and B2). First, the priming agent was applied by using a micro-brush; then, after the priming agent had been slightly air-dried, the bonding agent was applied. Excessive resin was removed with air. Both adhesives were light-polymerized (Fig 1E) for 20 seconds with the Mini-LED lamp (Satelec). Light-intensity was 800 mW/cm^2. Finally, a vinyl ring (Ciac srl, San Pietro in Casale, Italy) with an internal diameter of 5.5 mm was applied under the dentin surface to standardize the adhesion area (Fig 1F).

The specimens (Fig 1G) were stored in water at room temperature for 24 hours after cementation. Then they were all submitted to a shear bond strength test to check the strength of adhesion between the two substrates, dentin and ceramic. This test is defined as a test in which two materials are connected by an adhesive agent and loaded in shear until separation occurs.[25] Specimens were mounted on the jig (Fig 1H) of a universal testing machine (Erichsen mod. 476, Hemer, Germany), and shear force was applied to the adhesive interface until fracture occurred. The specimens were loaded at a crosshead speed of 1.0 mm/min. The calculated shear bond strength was determined by dividing the strength at which bond failure occurred by the bonding area.[25]

Fractured surfaces of each specimen were inspected with an optical microscope (Leica MZ 7.5, Leica Microsystem Ltd, CH-9435 Heerbrugg, Switzerland).

To maximize standardization, the same operator prepared the specimens and conducted the tests.

The data were evaluated by two-way analysis of variance to assess quantitative differences for the shear bond strength between groups of specimens treated with a different bonding agent and a different cement, also considering the different treatments of the ceramic surface. Means and standard deviations were calculated for each group.

Multinomial logit model was used to assess quantitative differences for the failure mode by the shear bond strength.

The hypotheses of independence between failure mode (cohesive vs. adhesive) and both adhesive system and silanization were evaluated by Pearson's chi-square test. In this analysis, the cohesive failure in dentin was merged with the cohesive failure in ceramic, because the cells with zero count (Table 2) prevent the correct use of the Pearson's chi-square test.

Differences were considered to be significant at $p < 0.05$.

Results

Number of specimens per group, mean shear bond strength values, and standard deviations of the groups are listed in Table 2.

Two-way ANOVA revealed the significant effect of silanization ($p < 0.01$) and did not show significant differences for either the bonding agents or the interaction between silanization and bonding agents ($p > 0.05$).

In this study, because of the limitation of the optical evaluation, three failure modes were considered: adhesive, cohesive in dentin, and cohesive in ceramic.[26]

Multinomial logit model did not show any statistical effects on the failure mode by the shear bond strength ($p > 0.1$).

The occurrences of the three failure modes considered for each bonding agent are listed in Table 2. The hypothesis of independence between failure mode (cohesive vs. adhesive) and both the adhesive systems ($p < 0.05$) and the silanization ($p < 0.05$) was rejected by Pearson's chi-square test. This means that there is a significant association between the bonding system Excite DSC/Variolink II and cohesive failures, and between the bonding system Scotchbond MPP/RelyX ARC and adhesive failures. Moreover, there is a significant association between silanization and cohesive failures, and between no silanization and adhesive system.

Discussion

Two adhesive systems were used in this study to cement the ceramic to the dentin substrate: the adhesive Excite DSC coupled with the cement Variolink II and the adhesive Scotchbond MPP coupled with the cement RelyX ARC. Both bonding systems used for cementation of CAD/CAM technology-based feldspathic ceramic to the dentin substrate achieved good shear bond strength values. The specimens were stored in water at room temperature for 24 hours after cementation. Then they were all submitted to a shear bond strength test to check the strength of adhesion between the two substrates, dentin and ceramic. Because of the limitations of this study due to the small number of specimens, the tests were accomplished only 24 hours after cementation for all specimens. Running the tests at 7 days instead of 24 hours may result in more clinically relevant final outcomes.

Many articles related to adhesive procedures used for the cementation of ceramic to tooth structure have shown that the presence of a hybrid layer between adhesive resin and dentin seems to adequately seal the dentinal tubules and allows a cellular reorganization of the pulpal tissues.[27–35] Both tested

Table 2 Shear bond strength means and standard deviations (MPa) and failure mode data of groups with different adhesive systems and different ceramic surface treatments

	A1 Excite DSC VariolinkII No silane	A2 Excite DSC VariolinkII Silane	B1 Scotchbond MP RelyX ARC No silane	B2 Scotchbond MP RelyX ARC Silane
n	10	10	10	10
Mean ± SD	22 ± 7a	29 ± 3b	22 ± 4a	26 ± 5b
Adhesive failure in cement	6	6	8	6
Cohesive failure in dentine	4	0	2	4
Cohesive failure in ceramic	0	4	0	0

Different lowercase letter in a row indicates significant difference.

cements are based on these adhesive procedures, which determine the formation of the hybrid layer and lead to the creation of a stronger link between dental structure and composite cement.

Another variable considered in this study was the different treatments of the ceramic surface. Sandblasting and etching with hydrofluoric acid have often been used to increase bonding strength to ceramic.[17,20-22] Some studies have demonstrated that these treatments, followed by the application of silane coupling agents, have been able to increase adhesion strength to feldspathic ceramic.[17,20-22] When a silane agent is used to improve the bond between composite and ceramic surfaces, the silanol groups of the silane agent condense with the silanol groups on the ceramic surface to form siloxane bonds that bind silane to the ceramic surface.[16] When the adhesive resin contacts the silane and both are activated by light, the methacrylate groups within the resin copolymerize with the silane, resulting in a bond between composite and ceramic through the adhesion promotion of silane.[16] In accordance with values reported by the quoted studies,[16,18,19] the present research shows a statistical difference between silanized and nonsilanized specimens in both groups in which the two cementation systems were used. This study was limited to the investigation of the effect of silanization without heat treatment. Further studies are needed to elucidate the influence of this post-silanization treatment; however the adhesion values obtained in this study are in accordance with values reported by other authors,[3,20,36] despite a difficult comparison of results because of variables such as specimen preparation technique, crosshead speed, cross-sectional surface area, and the type of test.[37] The values of shear bond strength obtained for both adhesive cementation systems tested in this study are able to guarantee an effective link between the dentin and the ceramic restoration in the specimens in which the ceramic substrate was only etched.

The results of this study suggest that accurate and meticulous procedures during the cementation phase may play an essential clinical role in achieving a valuable connection between the dentin and the ceramic restoration. Sandblasting and etching with hydrofluoric acid increased the bond strength to ceramic. These treatments, followed by the application of silane coupling agents, have been able to increase the adhesion strength to the feldspathic ceramic in accordance with previous studies.[17,20-22]

In this study the number of adhesive failures was higher in the specimens in which the adhesive Scotchbond MPP was used than in the specimens in which the adhesive Excite DSC was tested, where the number of cohesive failures is prevalent. The high number of cohesive failures, especially in the specimens in which the bonding system Excite DSC/Variolink II was tested, shows the efficacy of the link between the interfaces of the different substrates, dentin/resin and resin/ceramic. Further studies should be conducted to test the correlation of bond strength for both tested materials to 48 hours, 1 week, 1 month, and 1 year to evaluate the time factor related to the effective quality of bonding between ceramic material and dentin. Additional research should also be conducted to test the correlation between porosity, poor wetting, high viscosity, and failures.

Conclusions

Within the assumptions and limitations of this study (including the small number of specimens) the following conclusions may be drawn:

1. Both bonding systems used for cementation of the CAD/CAM technology-based feldspathic ceramic to the dentin substrate achieved good shear bond strength values. Two-way ANOVA did not reveal significant differences for either of the bonding agents.
2. The application of a silane coupling agent to the ceramic surface after etching with hydrofluoric acid increased the adhesion strength with both adhesive materials used. Two-way ANOVA revealed a significant effect of silanization ($p < 0.01$).

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