ORIGINAL RESEARCH

Bond Strength of Two Resin Cements with Leucite-reinforced Ceramic Using Different Bonding Agents

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Abstract

Aim: To compare the bond strength of two resin cements to leucite-reinforced ceramics using three different boding agents and evaluate the compatibility of bonding agents.

Materials and methods: Twenty extracted sound human molars were sectioned horizontally 2–3 mm above the cementoenamel junction (CEJ). CAD/CAM ceramic blocks for inLab were also sectioned to create 4 mm thick and bonded to the dentin. The adhesive groups assigned were divided into four adhesive groups: Group I: Variolink II dual-cure resin cement and Scotchbond Multi-Purpose Plus adhesive, group II: Multilink Automix dual-cure resin cement and Multilink primers, group III: Multilink Automix and Clearfil SE bond 2 (CSE2) adhesive, group IV: Multilink Automix and CSE2 with light curing after adhesive application. Five specimens of each group were sectioned perpendicular to obtain six microsticks of 1 × 1 mm width from each sample. Microtensile bond strength data were expressed in MPa. Fracture modes (FrMs) analyzed for the surfaces were divided into six patterns. Microtensile bond strength data were statistically analyzed with one-way ANOVA and Tukey *post hoc* tests ($\alpha = 0.05$). *T*-test was performed at the 5% significance level to analyze groups III and IV with and without light curing.

Results: Group I showed the highest μTBS average of 13.67 MPa, group IV showed 12.26 MPa, group III showed 12.15 MPa, and group II showed the lowest average of 10.84 MPa. No significant differences were found between the bonding agents. However, the six types of failure modes, although all observed, were characterized by the adhesive system: Type I: adhesive failure of laminated dentin and ceramic; type II: adhesive failure of laminated ceramic; type III: adhesive failure of laminated dentin; type IV: cohesive failure of luting agent; type V: cohesive failure of dentin, and type VI: mixed failure of adhesion and cohesion. As a result, the FrM most commonly observed was the adhesive failure at the luting cement-ceramic block interface.

Conclusion: The combination of resin cements and bonding agents did not significantly affect the bond strength of CAD/CAM ceramic restorations and dentin.

Clinical significance: Several universal bonding agents are currently available for direct and indirect bonding, and using the same bonding agent for direct and indirect restorations could simplify inventory and benefit routine clinical practice.

Keywords: Adhesive bond strength, Bonding agents, CAD/CAM restorations, Leucite-reinforced ceramic, Resin cements.

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Introduction

Currently, restorative dentistry is facing the transition from conventional impression techniques to digital impressions. Chairside milling units for all-ceramic restorations are gaining popularity due to their improvement in trueness and precision in marginal fit.[1] Christensen mentioned that 80.2% of roughly 1 million units of fixed restorations placed in the US were all-ceramic restorations, introducing the data from Glidewell Dental Laboratories. Given that condition, clinicians must be familiar with adhesive cements and their corresponding bonding agents to obtain predictable outcomes.[2] The evidence supports reliable bond strength and superior longevity in two-step-self-etch systems for resin composites.[3] As long as they use conventional methacrylate monomers, the bonding compatibility between the bonding agents and any resin composites is not a problem.[4] Conversely, unlike direct resin composites, many clinicians use bonding agents specially designed for specific resin cements that are part of a kit or a system. Although the adhesion mechanism of indirect and direct restorations is similar, the polymerization process of a resin cement could be prolonged due to the incompatibility with the bonding agent, negatively affecting chemical reactions among the components in the adhesive, activator, and resin cement.[5] On the other hand, some clinicians may use the same bonding agent for indirect and direct restorations. The above-mentioned two-step-self-etch systems can turn into a self-curing mode by adding a dual-cure activator for indirect restorations.[6] Nevertheless, recent manufacturers' recommendations and consensus seem to use optimized bonding agents for properly polymerizing each cement system.[7] This study

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Table 1: Materials used in the study

Material	*Type*	*Manufacturer*
IPS Empress CAD for CEREC and inLab	CAD/CAM ceramic block	Ivoclar Vivadent, Schaan, Liechtenstein
Variolink II dual-cure resin cement	Dual-cure cement	Ivoclar Vivadent, Schaan, Liechtenstein
Multilink Automix dual-cure resin cement	Dual-cure cement	Ivoclar Vivadent, Schaan, Liechtenstein
Multilink primers	Part of Multilink automix system, dual polymerized	Ivoclar Vivadent, Schaan, Liechtenstein
Scotchbond Multi-Purpose Plus Adhesive system	Total-etch adhesive, dual-cure capabilities with 3M Adper Scotchbond multi-purpose plus activator and 3M Adper Scotchbond multi-purpose plus catalyst	3M-ESPE, St Paul, Minnesota, USA
Scotchbond etchant phosphoric acid for Enamel and dentin	Etchant	3M-ESPE, St Paul, Minnesota, USA
Clearfil SE bond 2	Two-step, self-etch bonding system	Kuraray, Tokyo, Japan
Clearfil DC activator	Dual-curing activator, mixed with the bond become dual-cure.	Kuraray, Tokyo, Japan

aims to compare the microtensile bond strength (μTBS) between a resin cement and a leucite-reinforced ceramic using two different bonding agents to evaluate their compatibility. The null hypothesis tested was that any combination of resin cements and bonding agents would provide the same level of bond strength. In addition, the μTBS of the resin cement using a bonding agent with or without photopolymerization before cementation was tested.

Materials and Methods

This *in vitro* study was conducted as an experimental, randomized, and comparative study in the Department of Oral Rehabilitation and Biosciences, Division of Restorative Dentistry, Oregon Health and Science University School of Dentistry (OHSU), USA during the period from November 2017 to May 2018 (7 months). The extracted teeth were obtained from an OHSU Human Tooth Bank for all the experiment. The inclusion criteria for the teeth used in this study were the following: teeth had to be molars, they had to be intact, caries-free and unrestored, without fractures or other defects. All teeth used were anonymized; therefore, an ethical approval was not required.

Twenty extracted sound human teeth were embedded in stone blocks 2 mm below the cementoenamel junction (CEJ). Then, teeth were sectioned 2–3 mm horizontally above the CEJ and occlusal surfaces were polished with #600 grit silicon carbide paper for 1 minute to produce sound flat dentin surfaces before cementation.

Ceramic blocks for CEREC/inLab (IPS Empress CAD, Ivoclar Vivadent, Schaan, Liechtenstein) were sectioned to create 4 mm thickness slices using a slow-speed diamond saw (Accutom-5, Struers, Copenhagen, Denmark). A 9% buffered, gelled hydrofluoric acid (Porcelain Etch, Ultradent, Salt Lake City, UT, USA) was applied to one ceramic slice side for 60 seconds. After rinsing and air-drying, the etched surface was treated with silane (Silane, Ultradent, Salt Lake City, UT, USA) for 60 seconds and air-dried. Two types of resin cements and three types of bonding agents were used in the current study. The information of the materials used in the current study is listed in Table 1.

Steps of Adhesive Cementation

The prepared teeth were randomly assigned to one of the four adhesive groups ($n = 5$ per group). The adhesive groups assigned were the following:

Group I: Variolink II (VII) dual-cure resin cement and Scotchbond Multi-Purpose Plus (SBMP) adhesive, group II: Multilink Automix (MA) dual-cure resin cement and Multilink A and B primers (MP), group III: MA and Clearfil SE bond 2 (CSE2) primer and adhesive, group IV: MA and CSE2 with light curing (LC) after adhesive application.

Following the manufacturer's instructions, ceramic CAD/CAM block slices were bonded to the teeth with each adhesive system for all specimens. In group I, the surfaces of teeth were etched with 37% phosphoric acid for at least 15 seconds for enamel and for no more than 15 seconds for dentin. And then, the surface was rinsed with distilled water, followed by air-drying. Later, the primer and bonding agents were applied. In group II, the surfaces of teeth were treated with the mixture of primers A and B for 30 seconds. In group III, after applying primer to the teeth, the surfaces were dried with mild air. Then the mixture with Clearfil DC activator and Clearfil SE bond 2 was applied to the teeth and air-dried. In group IV, the bonding agent was applied on teeth in the same manner as in group III. Groups I, II, and III were not light-cured after applying adhesives. However, group IV was light-cured using the curing light (SmartLite Max LED curing Light, Dentsply Sirona, Milford, DE, USA) for 20 seconds after applying the bonding agent. For all four groups, ceramic CAD/CAM block slices were bonded to the prepared teeth under constant finger pressure, and photopolymerized using a high-power LED light-curing unit with the irradiance of 500 mW/cm^2 for 20 seconds on each surface of all groups. After photopolymerization, samples were stored in distilled water for 24 hours at 37°C.

Microstick Fabrication and Microtensile Bond Strength (μTBS) Test

Teeth were sectioned perpendicular to CAD/CAM block slice-dentin using a slow-speed saw (Buehler Isomet Low-Speed Saw, Buehler, Illinois) with a water-cooled 0.3 mm diamond blade to obtain rectangular microsticks of 1 × 1 mm width. Microsticks were obtained, avoiding the corners of the pulp, and a minimum of six microsticks were taken from each sample. Therefore, six microsticks were obtained from the central part of each tooth. A digital caliper was used to measure the dimensions to calculate the bonded area for each microstick. The microsticks were individually attached to a metal jig with cyanoacrylate glue and placed onto a microtensile bond strength test machine (MTS Static Materials Test System, Eden Prairie, Minnesota, USA). Then they were stressed at a cross-head speed of 0.5 mm/minute until failure. The data of μTBS testing was expressed in MPa, as derived from dividing the load at fracture (N) by the imposed bonding surface area (mm^2).

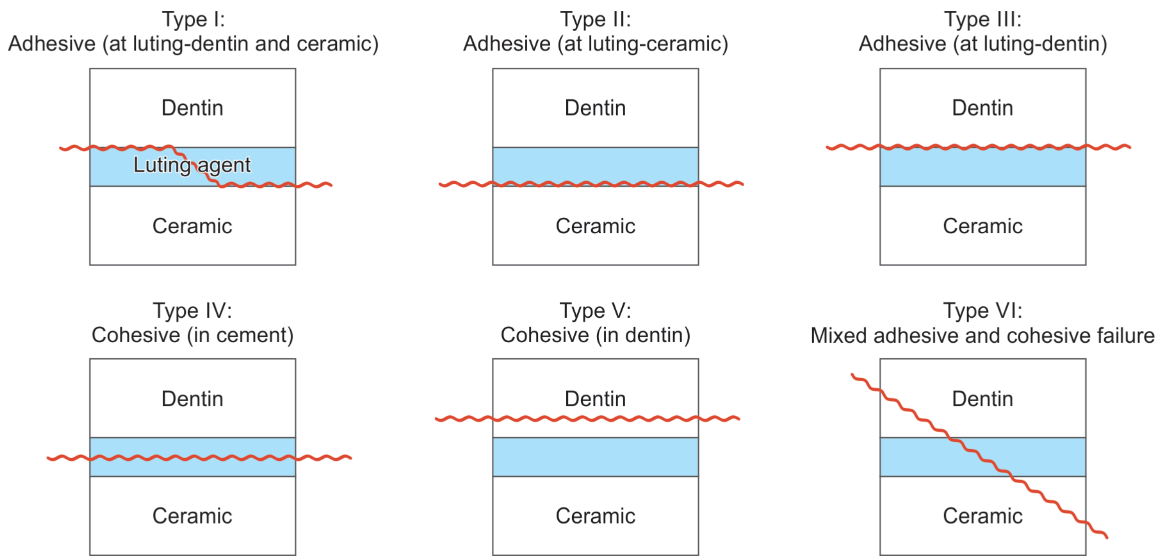

Fig. 1: Fracture modes of the tested adhesives applied to dentin

Fractured Mode (FrM)

To determine the failure mode, fractured surfaces were examined at 40X magnification using a stereomicroscope for each of the six microsticks obtained from all samples.

FrMs were classified into six types. Type I: adhesive failure at luting-dentin and ceramic, type II: adhesive failure at luting-ceramic, type III: adhesive failure at luting-dentin, type IV: cohesive failure in luting agent, type V: cohesive failure in dentin; and type VI: mixed adhesive and cohesive failure (Fig. 1).

Statistical Analysis

The data from µTBS testing (MPa) were statistically analyzed with one-way analysis of variance (ANOVA) and Tukey *post hoc* tests ($\alpha = 0.05$). In addition, a *t*-test was performed at the 5% significance level to analyze between groups III and IV with and without light curing. All statistical analyses of recorded data were performed using the Excel statistical software package (Ekuseru-Toukei 2016; Social Survey Research Information Co., Ltd., Tokyo, Japan).

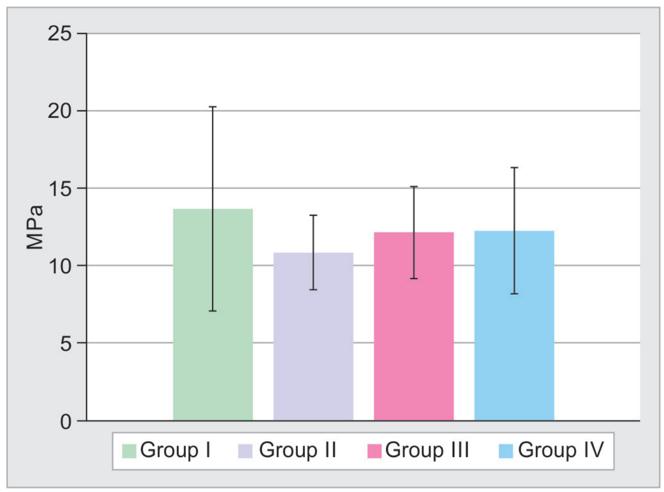

Fig. 2: Microtensile bond strength (µTBS) values in MPa

Results

Microtensile Bond Strength (µTBS)

Data from µTBS testing is presented in Figure 2. Group I showed the highest µTBS mean value (13.67 MPa) and group II presented the lowest mean value (10.84 MPa). Groups III and IV used MA and CSE2 and had mean values of 12.15 and 12.26 MPa, respectively. There were no significant differences among the adhesive groups shown in Figure 2 ($\alpha = 0.05$). Furthermore, a *t*-test was conducted, $t(58) = 0.09$, $p = 0.92$, indicating no significant difference between the groups III and IV.

Fracture Mode (FrM)

Fracture mode was observed using a microscope on the surface of each specimen after the µTBS test. The FrM of specimens after the µTBS test was done are noted as shown in Figure 3. The fracture modes of surfaces demonstrated a more significant proportion of type II FrM for all groups, except for group II. In group II, the FrM presented in a higher percentage was type I.

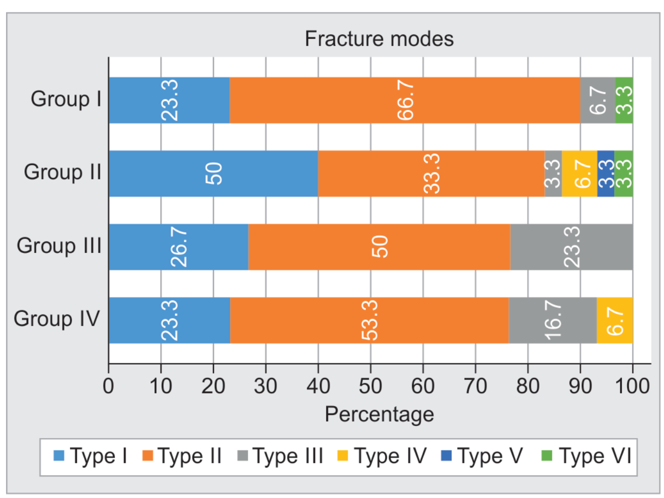

Fig. 3: Fracture modes of specimens after µTBS test (%)

Groups III and IV presented type I FrM at similar percentages (26.7% in group III and 23.3% in group IV). Groups III and IV also had higher percentages of type III than groups I and II. Groups II and IV showed some type IV, which was not observed in other groups.

Type VI, an adhesive failure involving fracture of dentin and ceramic, was also observed in a small number of samples from groups I and II. Type V was not observed in most groups, except for a small number of specimens in group II.

The inferences of current study indicates that there were no significant differences in bond strength depending on the bonding system. However, FrM was characterized by each group. Group I showed a considerably higher percentage of FrM at the bond interface of the laminated ceramic than at the interface between dentin and ceramic, indicating better immediate bond strength to dentin than groups III and IV. Group IV showed stronger bonding to dentin than group III, most of which had weak bonding to dentin and fractured. The results showed that most of them were fractured with weak adhesion to dentin. In group II, we also observed type V, which fractured within the dentin, and type VI, which fractured in both dentin and ceramic. It was suggested that the adhesive strength was stronger than that of the dentin and ceramic blocks or that the prepared specimens may have been defective.

Discussion

The current study evaluated the bond strength of CAD/CAM leucite-reinforced feldspathic blocks to dentin using resin cement and performed µTBS tests using different bonding strategies.

In the present study, no significant differences were found between each group. Although there were differences in FrMs, the bond strength was considered to be maintained. This result suggests that the bonding agents used for direct bonding restorations can be used for bonding ceramics since the same level of bond strength could be obtained with any resin cement and adhesive system used and combined.

Dong et al. reported that adhesion to dentin depends on the bonding agent formulation. They compared the bond strength of two different resin cements in combination with four different bonding agents (Scotchbond Multipurpose, Prime & Bond NT, Integra Bond, and Single Bond). Bonding agents and resin cements from different manufacturers can result in lower bond strength due to resin composition incompatibilities.[8] For composite resins and adhesive resin cements, the initiator and its initiation rate are essential for bond strength. For light-curing types, polymerization initiators often combine camphorquinone and tertiary amines. In addition, because of the limited depth of light irradiation, many resin cements are dual-cure. In such cases, benzoyl peroxide (BPO) is often included as an initiator and tertiary amines as catalysts since they are also responsible for chemical polymerization.[9] Tay et al. compared the results in simple bonding systems such as two-step and one-step. They reported that acidic monomers promote the consumption of tertiary amines, the initiators of resin cements, resulting in incomplete polymerization and reduced bond strength.[10–12]

The µTBS test has recently been considered a versatile and standard bond strength test.[13] The µTBS test can obtain exclusively adhesive bond failures if the bonded surface area is about 1 mm^2. Therefore, in the current study, µTBS was used to validate the adhesion strength using a 1×1 mm^2 sample.[14]

Regarding FrMs, the most common FrM was type II that is between adhesive at luting resin cement and ceramics. Hikita et al. reported more fractures between dentin and cement, although it depends on the material used.[15]

The finding from group I suggests that the FrM indicates stronger adhesion at the dentin–ceramic interface than in the other groups. Kwong et al. suggested that removing the hardened dentin surface layer or conditioning it with acid may be effective in obtaining adhesion to the hardened dentin.[16]

From the result of group IV, bonding polymerization prior to cement filling may cause cohesive failure due to stronger adhesion to the resin cement. In addition, although adhesion to dentin and ceramic was strong, cohesive failure was also observed in group II. Therefore, it is necessary to consider the strength of the resin cement itself as well as the interfacial fracture.

The current study showed that most FrMs belong to types I and II, including fracture at the ceramic interface. In other words, adhesion between resin cement and ceramic was inferior to that between resin cement and dentin. The possibility of post-etching debris affecting bonding has not been investigated. Thus, it is necessary to continue to investigate ceramic surface preparation methods. Souza et al. reported that bond strength varied with self-etching primer composition in Multilink, and Variolink with etching and rinse was the strongest.[17] Similar results were observed in the current study, although the difference was not significant.

The limitation of the present study is the exclusive investigation of immediate bond strength without evaluating long-term bond strength or fatigue testing. Besides, even if the polymerization rate of Clearfil SE bond 2 samples with camphorquinone and tertiary amines as initiators was slower, the polymerization had progressed after 24 hours when the µTBS test was performed. Therefore, the µTBS values may not have differed significantly from those of Multilink or Scotchbond. Clinically, this compromised polymerization speed due to the lack of "tailored" initiators could be a problem in using a different manufacturer's bonding agent for cementing. Based on the results of the FrMs in the present study, further investigation of surface treatment prior to adhesive manipulation will be needed.

Generally, the manufacturer recommends using bonding agents from each cementation kit for the proper polymerization or a bonding agent recommended by the same manufacturer. With this in mind, the results of the current study could serve for daily clinical practice since no significant difference in bond strength was observed between the combinations of adhesive systems. According to the results the current study obtained, the null hypothesis tested was accepted. If the same bonding agent is used for resin cements as for direct restoration resin composites, it is important to understand which initiator is being used to initiate the polymerization and to select the right one. Even after light curing, a layer of residual acidic monomer may inhibit adhesion, and it is recommended that a combination of monomer acidity and polymerization speed should be taken into consideration when using adhesives with tertiary amines.

Conclusion

Within the limitations of the current study, the combination of resin cements, adhesive, and bonding cure mode did not significantly affect the bond strength of CAD/CAM restorations and dentin. Several universal bonding agents are currently available for direct and indirect bonding, and using the same bonding agent for direct and indirect restorations could simplify inventory and benefit routine clinical practice.

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