

Keywords

Polymerization
Dental Porcelain
Resin Cements
Dentin-Bonding Agents

Authors

Ana Teresa Maluly-Próni *
(DDS, MSc)

Bruna Oliveira-Reis †
(DDS, MSc)

Vitor Ribeiro Jardim §
(BSc, MSc student)

Getulio Vasconcelos §
(BSc, MSc, PhD)

Wirley Gonçalves Assunção ^
(DDS, MSc, PhD)

Prof. Paulo Henrique dos Santos ^
(DDS, MSc, PhD)

Address for Correspondence

Prof. Paulo Henrique dos Santos ^

Email: paulosantos@foa.unesp.br

* PhD student, Department of Dental Materials and Prosthodontics, Araçatuba School of Dentistry, São Paulo State University - UNESP, Araçatuba, São Paulo, Brazil

† PhD student, Department of Restorative Dentistry, Araçatuba School of Dentistry, São Paulo State University - UNESP, Araçatuba, São Paulo, Brazil

§ Aeronautics Institute of Technology, São José dos Campos, São Paulo, Brazil

^ Associate Professor, Department of Dental Materials and Prosthodontics, Araçatuba School of Dentistry, São Paulo State University - UNESP, Araçatuba, São Paulo, Brazil

Received: 08.10.2019
Accepted: 29.01.2020

doi: 10.1922/EJPRD_1975Maluly-Próni10

Influence of the Application and Light-Curing of Bonding Adhesives Inside Lithium Disilicate Ceramic Crowns

ABSTRACT

This in vitro study evaluated the effect of the application and previous photoactivation of bonding adhesives in lithium disilicate ceramics. This was achieved by analyzing the bond strength of the cement-ceramic interface, nanohardness and elastic modulus of bonding adhesives and resin cements. The bonding adhesives used were Scotchbond Multi-Purpose (SBMP); Scotchbond Multi-Purpose light-cured (SBMP+LC); Single Bond 2 (SB2); Single Bond 2 light-cured (SB2+LC); Single Bond Universal (SBU) and Single Bond Universal light-cured (SBU+LC). After luting the ceramic in composite resin blocks, all specimens were subjected to 10,000 thermocycles. Sticks were obtained for the microtensile bonding strength test and the nanohardness and elastic modulus of the bonding adhesive and resin cements. Data were submitted to one-way ANOVA and Fishers tests ($\alpha = 0.05$). The results indicated that the bonding strength and mechanical properties were influenced by the interaction of the bonding adhesive and resin cement. For the mechanical properties analyzed, no statistically significant differences were observed between the groups. Single Bond Universal was the only system that was effective with or without previous light activation. For the other adhesive systems, Scotchbond Multi-Purpose and Single Bond 2, the previous light activation was necessary to optimize the bonding strength of the adhesive interface.

INTRODUCTION

With increasing patient demand for aesthetic and rehabilitation treatments in the field of dentistry, ceramic indirect restorations have become widely used due to their aesthetics, color stability, biocompatibility, longevity, radiopacity, low thermal conductivity, and optimal integration with gum tissues.¹⁻⁴

Until the 1980s, indirect restorations failed rapidly and exhibited a high marginal infiltration rate⁵ due to lack of adequate adhesive procedures and data regarding the surface treatment of feldspathic ceramics.⁶ The development of novel ceramics with higher crystalline content, such as glass ceramics, in which the adhesion to dental tissues is promising, has changed this concept. These materials are composed of glass and crystalline phases, where the concentration of crystals incorporated into the matrix reinforces its structure.⁷ Commonly used glass ceramics are reinforced by lithium disilicate ($\text{Li}_2\text{Si}_2\text{O}_5$) and are composed of 30% amorphous silica and 70% crystalline lithium disilicate.⁸ The flexural strength of these materials is approximately 400 MPa which provides a balance between

mechanical resistance and translucency,⁹ facilitating its use as a monolithic prosthesis in both anterior and posterior teeth.¹⁰

The adhesion of the ceramic restorations to the dental substrate involves two interfaces, the ceramic-cement and cement-substrate,¹¹ whose stability influences the success of the prosthetic rehabilitation with laminates, onlays or crowns.¹² Surface modification of glass ceramics by hydrofluoric acid etching increases the contact surface, improving the interaction between the ceramic and luting agent.¹³ This process produces microporosities due to the removal of the glassy matrix, allowing greater silane penetration which promotes chemical bonding between the inorganic matrix of the ceramic and organic matrix of the resin material.⁹ On the other hand, the application of adhesive materials promotes bonding between glass ceramics and dental structures. The evolution and reliability of adhesive systems¹⁴ and luting agents on the dental structure can improve the clinical success of these restorations,¹⁵⁻¹⁷ resulting in minimal wear of the tooth structure with the use of thin ceramics.^{8,18,19} The micro-retentions and adhesion properties can be improved by cross-linking,²⁰ clinically resulting in high survival rates after long-term follow-up.^{3,21}

The adhesive system formulations and luting agents have been continuously improved to simplify their clinical use, reducing application time and sensitivity after treatment.^{22,23} Adhesive systems can be classified based on their interaction with dental substrates and the number of steps required for their application;²⁴ etch-and-rinse (two- or three-steps, depending on whether the primer and bonding agent are separated or combined in a single bottle) or self-etch adhesives (one- or two-steps, depending on whether the etching/priming agent is separated from the adhesive or combined to allow for a single application procedure).²⁵⁻²⁷ Resin cements can be classified as conventional, requiring prior application of an adhesive system, or self-adhesive types.²⁸

The maintenance of the mechanical strength and marginal integrity of the restoration is essential for the stability of the ceramic/dental substrate bonding interface, which is directly dependent on the degree of conversion of the adhesive systems and resin cements.⁶ To achieve good physico-mechanical properties, these materials must be well polymerized, achieving a high conversion rate of the monomers into polymers, as deficiency in this respect could affect the clinical performance of the resulting resin materials.²⁹ Nanoindentation is a reliable laboratory test that can be used to measure the nanohardness and elastic modulus of specific areas of adhesive interface³⁰ and determine the effects of extended light-curing on the specific adhesive interface components.³¹

Most dental material manufacturers recommend the use of resin cement directly on the silanized inner surface of the glass ceramic. However, the microporosities formed by acid etching could modify the surface wettability,³² damaging cement flow on the silanized surface. Thus, the application of a thin layer of bonding adhesives after silanization could facilitate better interaction between the resin cement and silanized

ceramic. However, the need for prior photoactivation of the dental adhesive is controversial;²⁹ in some situations, it can be performed concomitantly with activation of the resin cement.² The adhesive must be highly polymerized and not interfere in resin cement conversion, resulting in an adhesive interface with high mechanical and clinical strength.

This *in vitro* study was performed to evaluate the effect of the application and previous photoactivation of various bonding adhesives inside lithium disilicate ceramics. This was achieved by analyzing the bond strength of the cement-ceramic interface and the nanohardness and elastic modulus of different bonding agents and resin cements. The null hypotheses tested were that (1) the application and previous photoactivation of various adhesive systems would not result in significant differences in the bond strength of the luting agents to lithium disilicate ceramics, (2) previous photoactivation of adhesive systems would not affect the mechanical properties of the resin cement and bonding adhesive.

MATERIAL AND METHODS

SAMPLE PREPARATION

A total of 49 sintered blocks of lithium disilicate glass ceramic (IPS e-max CAD, Ivoclar Vivadent), measuring 6×6×2 mm were prepared and 49 composite resin blocks (Z350XT, 3M ESPE) measuring 6×6×4 mm were obtained. The materials, composition, and batch numbers are listed in Table 1. The specimens were sandblasted with 50 µm aluminum oxide at 2 bar pressure for 5 s on a single surface at a working distance of 5 mm to standardize the surface roughness of the ceramic and composite resin blocks after the milling process. Afterwards, the blocks were cleaned in an ultrasonic unit (Cristófoli) for 8 min with distilled water and were subsequently air dried. The ceramic surfaces were then etched with 10% HF (Hydrofluoric acid gel, Dentsply) for 20 s,³³ cleaned with 35% phosphoric acid³⁴ (Ultradent Products Inc.) for 30 s, and the silanizing agent was applied for 60 seconds (RelyX Ceramic Primer, 3M ESPE). The specimens were divided into 7 experimental groups according to bonding adhesive and light-curing process for cementation: control (without adhesive systems); Adper Scotchbond Multi-Purpose Adhesive bonding agent (SBMP); Adper Scotchbond Multi-Purpose + light-curing (SBMP+LC); Adper Single Bond 2 (SB2); Adper Single Bond 2 + light-curing (SB2+LC); Single Bond Universal (SBU); and Single Bond Universal + light-curing (SBU+LC). Thin layer of the adhesive systems was applied followed by application of a mild air stream, and, in the light-curing groups, the bonding adhesives were photoactivated using a LED polywave unit (VALO, Ultradent Products Inc.) for 10 s. The luting agent (RelyX Ultimate, 3M ESPE) was manually manipulated and applied to the pretreated ceramic surfaces and immediately positioned on the composite resin blocks. Before photoactivation, a load of 4.9 N was applied to standardize the thickness of the resin cement. The excess cement was removed with a disposable

Table 1. Materials used in this study and their compositions.

Material	Type of material	Manufacturer/ Batch number	Composition
IPS e.max CAD	Lithium disilicate glass ceramic, D3 LT	Ivoclar Vivadent/R49299	SiO ₂ , Li ₂ O, K ₂ O, MgO, Al ₂ O ₃ , P ₂ O ₅ , ZrO ₂ , ZnO, MgO, and coloring oxides
Filtek Z350 XT	Nanocomposite resin, A6B	3M ESPE/N734382	Bis-GMA, UDMA, TEGDMA, and Bis-EMA
RelyX Ceramic Primer	Ceramic primer (silane)	3M ESPE/N662908	MPS, ethanol, and water
Adper Scotchbond Multipurpose	Total-etch bonding adhesive	3M ESPE/515442	Bis-GMA, HEMA, and triphenylantimony
Single Bond 2	Total-etch bonding adhesive	3M ESPE/639238	Bis-GMA, HEMA, dimethacrylates, ethanol, water, photoinitiators, methacrylate functional copolymer of polyacrylic and polyitaconic acids, and silica nanofiller
Single Bond Universal	Multimode bonding adhesive	3M ESPE/595105	MDP, dimethacrylate resins, HEMA, Vitrebond TM Copolymer, filler, ethanol, water, initiators, and silane
RelyX Ultimate	Resin cement, A1	3M ESPE/596263	Base paste: methacrylate monomers, radiopaque silanated fillers, initiator, stabilizer, and rheological additives Catalyst paste: methacrylate monomers, radiopaque alkaline (basic) fillers, initiator, stabilizer, pigments, rheological additives, fluorescence dye, and dark cure activator for Scotchbond Universal

applicator and then photoactivated using a LED polywave unit for 3 s on the face of the ceramic as part of the pre-polymerization stage, then light-cured for 20 s³⁵ on the ceramic surface. The LED polywave unit power intensity (1280 mW/cm²) was verified using a RD-7 radiometer (Ecel Ind Com).

After the cementation process, the specimens were stored in distilled water at 37 °C for 24 h, and then subjected to thermo-cycling (model 521.4, Ética) with alternating immersion in water baths of 5 and 55 °C. The cycle duration was 1 min with a dwell time in each water bath of 28 s and a transfer time between baths of 2 s for a total of 10,000 cycles, simulating 1 year of aging.³⁶ After the aging, the specimens were sectioned perpendicular to the adhesive interface (IsoMet 5000, Buehler) to obtain sticks with an area of approximately 1 mm². A total of 9 sticks were obtained from each sample.

MICROTENSILE BOND STRENGTH

To test the microtensile bond strength, 8 sticks were obtained from each sample, fixed individually to a metallic device with a cyanoacrylate adhesive (Super Bonder Flex Gel, Loctite) and subjected to tensile force testing using a microtensile test machine (Odeme Microtensile OM 100, Odeme Dental Research) at a crosshead speed of 0.7 mm/min. The bond strength values were obtained in units of MPa as follows: $R_u = (F/A)$ where R_u is the bond strength, F is the maximum force (N), and A is

the area of the bonding interface (mm²). After debonding, the specimen surfaces were analyzed using optical microscopy at a 6.6× magnification (Stemi SV11, Carl Zeiss AG) to characterize the failure modes. The failure types were defined as: a) cohesive failure of the ceramic; b) cohesive failure of the composite resin; c) adhesive failure between the luting agent and ceramic; or d) mixed failure. In addition, SEM images were obtained via backscattered electron diffraction of the resin/cement interface for each experimental group (EVO HD LS-15, Carl Zeiss AG).

MECHANICAL PROPERTIES

A single stick from each experimental specimen was embedded in acrylic resin (Clássico), manually finished with #320, #600, #800 and #1200 grit silicon carbide paper (Extex Corp.) and polished with diamond pastes (#6, #3, and #1 and #0.25 µm) for 4 min each. The specimens were subsequently cleaned in an ultrasonic unit with distilled water for 8 min between the steps and at the end of the process.

The nanohardness (H) and elastic modulus (Er) values were measured using a nanodurometer (NHT³, Anton Paar) under a load of 1000 µN applied for 5 s, followed by maintenance of the maximum load for an additional 2 s and load removal for 5 s.²⁹ A Berkovich pyramidal tip was used and 3 indentations were made in 2 different regions of the adhesive interface, in

the bonding adhesive and resin cement. H and Er were calculated from the load-displacement curves according to the following relationship:

$$Er = S \sqrt{\pi} / 2 \sqrt{A},$$

where S is the initial unloading stiffness and A is the projected contact area between the indenter tip and the sample at the maximum load:

$$H = P_{\max} / A,$$

where $P_{\max}$ is the maximum load and A is the same projected contact area for the elastic modulus calculation.

STATISTICAL ANALYSIS

The data regarding the bond strength and mechanical properties were subjected to statistical tests of normality Shapiro-Wilk, and one-way ANOVA to examine the differences of bond strength, nanohardness, and elastic modulus of the resin cement. The nanohardness and elastic modulus of the bonding adhesives were analyzed by two-way ANOVA (material and activation mode). Subsequently, Fisher's post-test was used to compare the mean values ($\alpha = .05$).

RESULTS

The results of the ANOVA test for bond strength values showed statistically significant differences among the experimental groups ($P = .043$). Table 2 shows that the highest bond strength values were found for the groups: control (18.97 ± 4.60 MPa), SBMP+LC (18.79 ± 11.05 MPa), SBU (18.54 ± 10.15 MPa), SBU+LC (18.40 ± 7.35 MPa), and SB2+LC (17.24 ± 9.31 MPa), with no significant differences between them ($P > .05$). The lowest bond strength values were observed for the SBMP (9.10 ± 3.48 MPa) and SB2 (9.03 ± 5.26 MPa) groups, which were statistically different from the control ($P = .023$ and $P = .022$, respectively), SBMP+LC ($P = .025$ and $P = .024$, respectively), SBU ($P = .029$ and $P = .028$, respectively), and SBU+LC groups ($P = .031$ and $P = .030$, respectively).

No premature failure was observed in the groups where the adhesives systems were previously light-cured (Table 2). A preponderance of adhesive failure was observed in all experimental groups, followed by mixed type failure (Figure 1). In

the SB2 group, only adhesive failure occurred and no cohesive failures were observed in the ceramic or composite resin for any group (Figure 1).

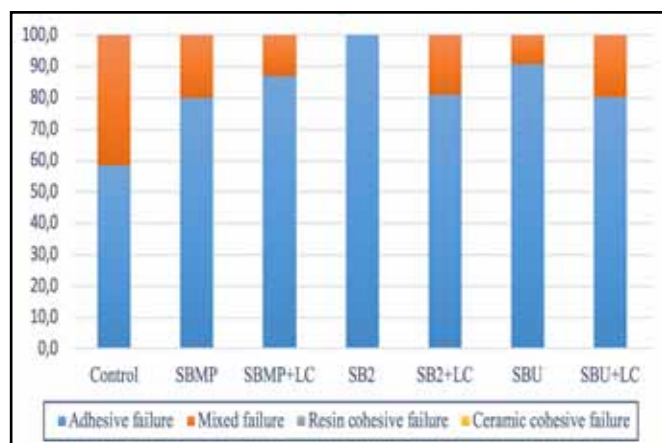


Figure 1: Fracture pattern of the sticks after the bond strength test (%)

Table 3 shows no statistical difference of mechanical properties analyzed in the comparison among all adhesive systems without previous light-curing ($P > .05$). When previous photoactivation was performed, the SBMP group presented significantly higher values of nanohardness (868.20 ± 140.70 MPa) and elastic modulus (14.03 ± 1.23 GPa) compared to the other materials ($P < .05$). No statistically significant difference was observed between the groups previously photoactivated compared to the same groups without prior photoactivation for all studied materials ($P > .05$).

Table 4 shows no statistical difference between the mechanical properties of the resin cement when different bonding adhesive application protocols were used ($P > .05$).

DISCUSSION

The influence of different types of bonding adhesives and previous light-curing of the materials on the bond strength was examined and the first null hypothesis of the study could be rejected. The SBMP and SB2 groups showed the lowest bond strength values of all the groups tested (Table 2). The higher viscosity of the SBMP adhesive resulted in a lower

Table 2. Microtensile bond strength values (MPa, mean $\pm$ standard deviation) and number of premature failures according to the bonding adhesive used..

	Control	SBMP	SBMP+LC	SB2	SB2+ LC	SBU	SBU+LC
Bond Strength	18.97 ± 4.60 A	9.10 ± 3.48 B	18.79 ± 11.05 A	9.03 ± 5.26 B	17.24 ± 9.31 AB	18.54 ± 10.15 A	18.40 ± 7.35 A
Premature failure	2/56	2/56	0/56	2/56	0/56	1/56	0/56

Means followed by different uppercase letters in row are significantly different ($P < .05$).

SBMP: Scotchbond Multipurpose bonding adhesive, SB2: Single Bond 2 bonding adhesive, SBU: Single Bond Universal, LC: light-cured.

Table 3. Nanohardness (H - MPa; mean $\pm$ standard deviation) and elastic modulus values (Er - GPa; mean $\pm$ standard deviation) of the bonding adhesives as a function of previous light-curing.

		SBMP	SB2	SBU
H	Without light-curing	676.03 $\pm$ 96.95 Aa	640.60 $\pm$ 81.47 Aa	510.44 $\pm$ 114.52 Aa
	Light-cured	868.20 $\pm$ 140.70 Aa	444.40 $\pm$ 118.29 Ba	444.02 $\pm$ 139.12 Ba
Er	Without light-curing	12.81 $\pm$ 1.54 Aa	12.96 $\pm$ 1.33 Aa	11.71 $\pm$ 2.12 Aa
	Light-cured	14.03 $\pm$ 1.23 Aa	9.59 $\pm$ 2.34 Ba	9.36 $\pm$ 2.33 Ba

Means followed by different letters, uppercase in rows and lowercase in columns are significantly different for each mechanical property analyzed ($P < .05$).
SBMP: Scotchbond Multipurpose bonding adhesive, SB2: Single Bond 2 bonding adhesive, SBU: Single Bond Universal.

Table 4. Nanohardness values (H - MPa; mean $\pm$ standard deviation) and elastic modulus values (Er - GPa, mean $\pm$ standard deviation) of the RelyX Ultimate resin cement, according to the bonding adhesive groups and light-curing procedure.

	CONTROL	SBMP	SBMP+LC	SB2	SB2+LC	SBU	SBU+LC
H	940.21 $\pm$ 28.82 A	854.38 $\pm$ 104.20 A	1471.74 $\pm$ 676.12 A	1644.16 $\pm$ 720.74 A	778.66 $\pm$ 11.24 A	867.75 $\pm$ 101.66 A	988.06 $\pm$ 401.49 A
Er	16.91 $\pm$ 0.94 A	17.48 $\pm$ 1.20 A	19.17 $\pm$ 5.23 A	20.48 $\pm$ 2.66 A	15.22 $\pm$ 0.70 A	17.82 $\pm$ 0.78 A	16.72 $\pm$ 1.77 A

Means followed by different uppercase letters in rows are significantly different for each mechanical property analyzed ($P < .05$).
SBMP: Scotchbond Multipurpose bonding adhesive, SB2: Single Bond 2 bonding adhesive, SBU: Single Bond Universal, LC: light-cured.

flow of the material compared to the other adhesives used,²⁶ creating a thicker layer which prevented interaction with the resin cement, may generating a thick and "pure" layer of adhesive that was less polymerized. This resulted in lower bond strengths when compared to the group where the adhesive was previously light-cured (Table 2). The chemical composition of the adhesive consists of the hydrophobic Bis-GMA and hydrophilic HEMA monomers. Water from the environment could lead to phase separation in the cross-links, creating a resinous mixture.²⁰ The most commonly used photoinitiator, camphorquinone, is hydrophobic which could lead to an inadequate photoinitiation due to the hydrophilic monomers, leading to a decreased polymerization quality.²⁰

In the SB2 groups, the presence of alcohol and water solvents decreased viscosity, allowing for better flow. However, inadequate solvent evaporation could lead to less conversion of the bonding adhesive and the non-evaporated solvent could be replaced by water, which would plasticize the polymer by reducing its mechanical strength.¹⁴ In addition, the large amount of hydrophilic monomers in SB2 could facilitate greater incorporation of water when not correctly photoactivated, resulting in hydrolysis breakage of the polymers by water incorporation.²⁴

In the SBMP and SB2 groups with no previous light-curing, some gaps between the ceramic and luting agents were observed (Figure 2 B and D). This suggests that the molecular mobility of the materials and polymerization contraction¹² led to the formation of defects at the adhesive interface, forming gaps between the ceramic and luting agents and decreasing the area of contact which could result in reduced bond strength values.

The application of SBU without previous photoactivation showed similar results to the other groups with previous photoactivation of the adhesive and those where no bonding adhesive was applied (Table 2). The SBU adhesive is a "multi-mode"⁴ adhesive system containing silane and 10-MDP, which could contribute to the higher bond strength even without previous photoactivation. Silane is a chemical bonding agent which can copolymerize with monomers in the resin cement and react chemically with silicon-containing materials⁴ by connecting the inorganic matrix of the glass ceramic with the organic matrix of resin.⁹ The 10-MDP phosphate acid monomer can chelate metallic cations in polycrystalline ceramics,⁴ increasing the stability of the bond even in an aqueous environment.²³ This bonding characteristic of the adhesive likely influenced the bond strength in the SBU group compared to other groups where no previous activation was applied (Table 2). Moreover, the higher bond strength for the SBU group

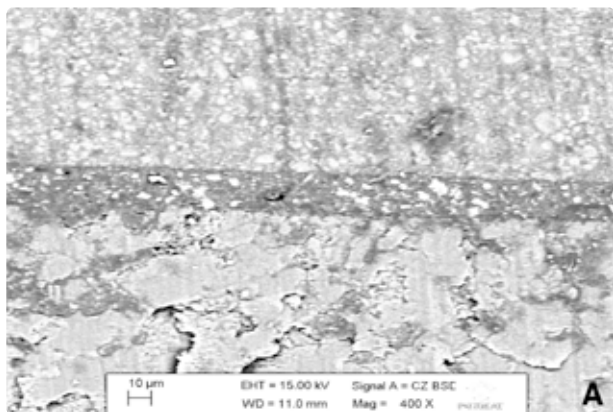


Figure 2A: SEM (backscattered electrons) of the adhesive interfaces of control group

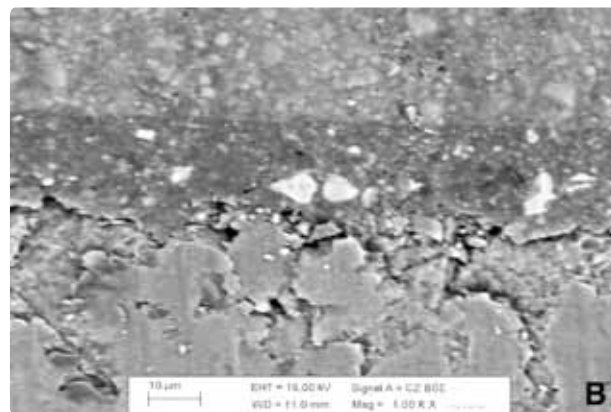


Figure 2B: SEM (backscattered electrons) of the adhesive interfaces of SBMP group.

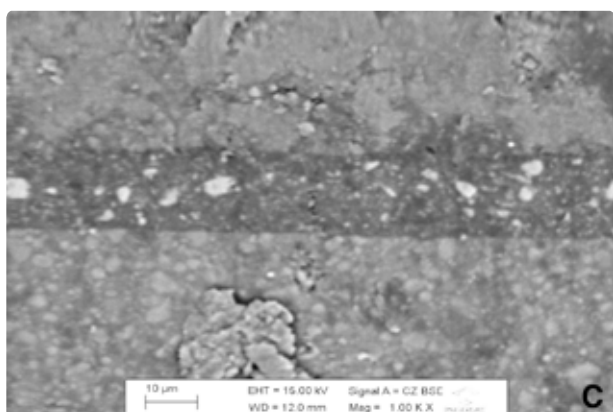


Figure 2C: SEM (backscattered electrons) of the adhesive interfaces of SBMP+LC group

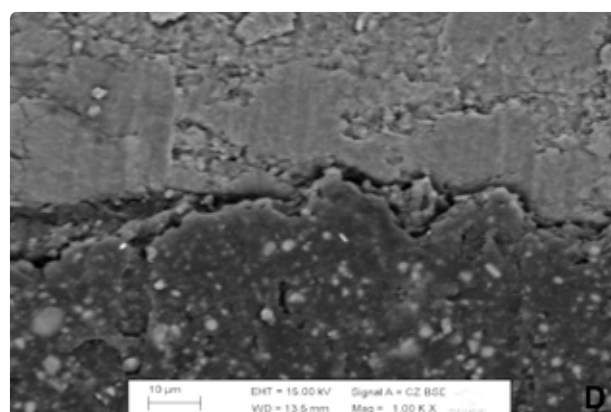


Figure 2D: SEM (backscattered electrons) of the adhesive interfaces of SB2 group

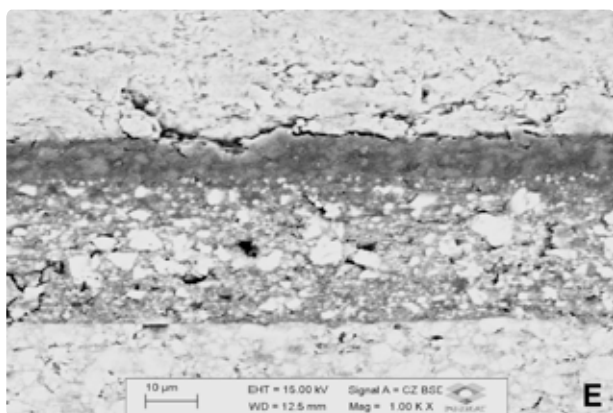


Figure 2E: SEM (backscattered electrons) of the adhesive interfaces of SB2+LC group

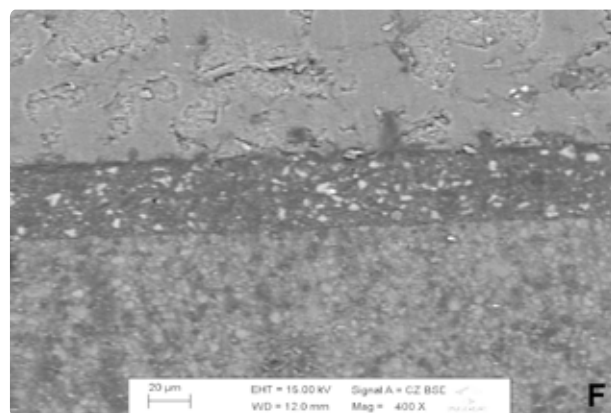


Figure 2F: SEM (backscattered electrons) of the adhesive interfaces of SBU group

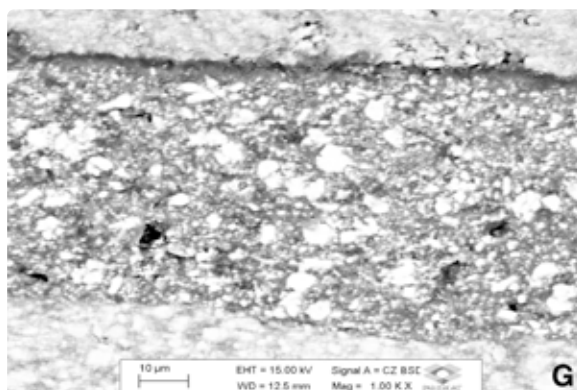


Figure 2G: SEM (backscattered electrons) of the adhesive interfaces of SBU+LC group

compared to those not previously photoactivated is also due to the use of the RelyX Ultimate dual-cured resin cement, which contains a dark cure activator for the SBU adhesive. These materials show better performance when used together, with or without photoactivation of the bonding adhesive.⁴

The ceramic ($\text{Li}_2\text{Si}_2\text{O}_5$) is composed of elongated crystals arranged on top of each other as scaffolds, which would likely prevent light transmission.¹¹ Due to the importance of the previous photoactivation of the SBMP and SB2 bonding adhesives (Table 2), the use of a high-intensity, polywave LED light curing unit is justified, since a substantial amount of light is lost by absorption, dispersion, and transmission by the ceramic.^{2,11} Premature failures in the bonding process, which occurred in the non-photoactivated bonding adhesive groups (Table 2), and the preponderance of adhesive failures (Figure 1) in these groups corroborate these results (Figure 3 D-G).

In the mechanical properties analysis (H and Er) of the bonding adhesives, no significant influence of the previous light-curing was observed (Table 3). In addition, the previous treated had no effect on the mechanical properties of the resin cement (Table 4), so the second null hypothesis of the study could be accepted. Although these differences were not statistically significant, higher values of the mechanical properties were observed for the SB2 and SBU groups that were not light cured compared to the respective photoactivated groups (Table 3). It is likely that the non-light cured bonding adhesives may have been mixed with the resin cement during the luting procedure (Figure 2 B, D and F), which could result in improved mechanical properties due to the presence of inorganic particles in the resin cement, composed of mostly glass or silica particles. This also reduced the solubility and volumetric contraction of the material.¹⁰ In Figure 2 C, E and G an adhesive layer separated from the resin cement when previously light-cured is observed, in contrast to the non-light-cured material (Figure 2 B, D and F).

Comparing the mechanical properties among the bonding adhesives, for the SBMP and SBMP + LC groups, H and Er were higher than those of the other light-cured bonding adhesives (Table 3). SBMP contains a large amount of Bis-GMA monomers, which have a high cross-linking ability and form rigid polymers,²⁷ besides being hydrophobic in nature with low permeability to water, resulting in a high degree of polymerization.²⁶ The large amounts of solvents in the SB2 and SBU bonding adhesives could cause deterioration of the mechanical properties due to changes in the structure and number of crosslinks, leading to the formation of less resistant polymers.²⁷ The large amount of adhesive failures in these groups agree well with this prediction (Figure 3 D and F).

The results obtained in this study show that the mechanical properties of bonding adhesives and resin cement may not be directly related to the effectiveness of the adhesive interface. During indirect restorations cementation, it is important to achieve greater conversion of the resin materials, and improve the chemical/mechanical interaction among all structures

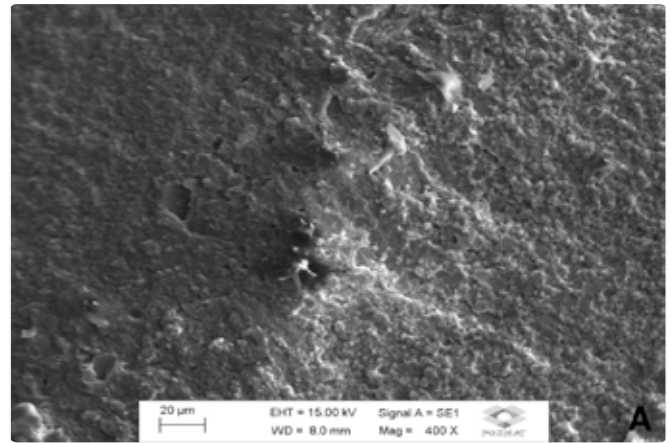


Figure 3A: SEM of representative specimens (original magnification of 400×) of a mixed failure of the ceramic surface without bonding adhesive.

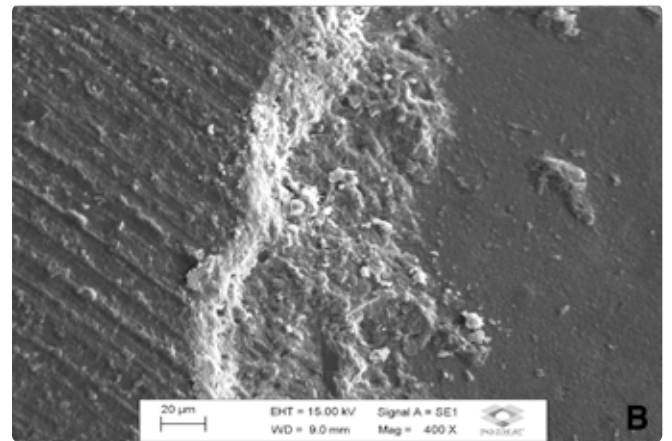


Figure 3B: SEM of representative specimens (original magnification of 400×) of a mixed failure on the ceramic surface with SBMP application.

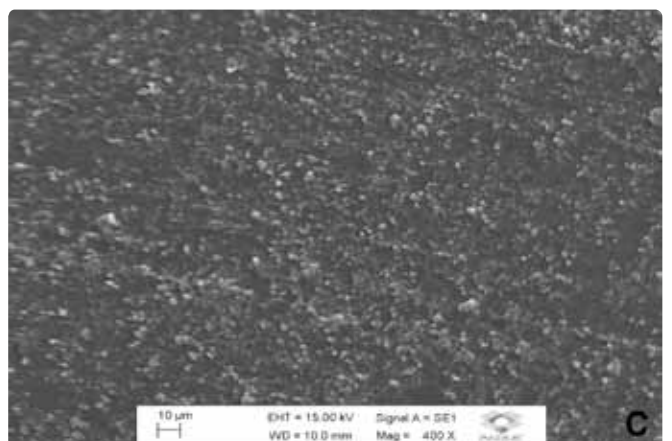


Figure 3C: SEM of representative specimens (original magnification of 400×) of an adhesive failure of the ceramic surface with SBMP+LC application

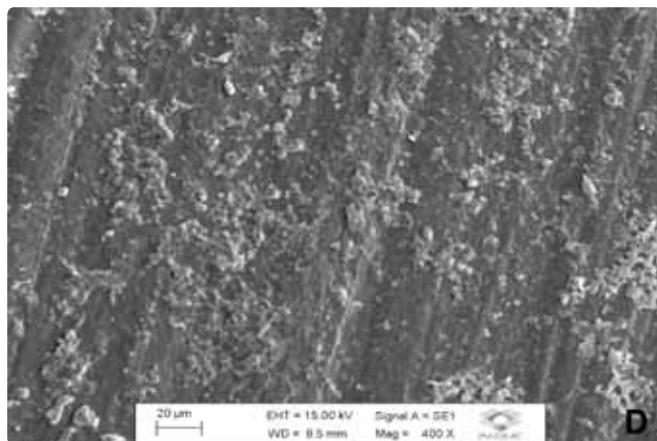


Figure 3D: SEM of representative specimens (original magnification of 400×) of an adhesive failure of the ceramic surface with SB2 application.

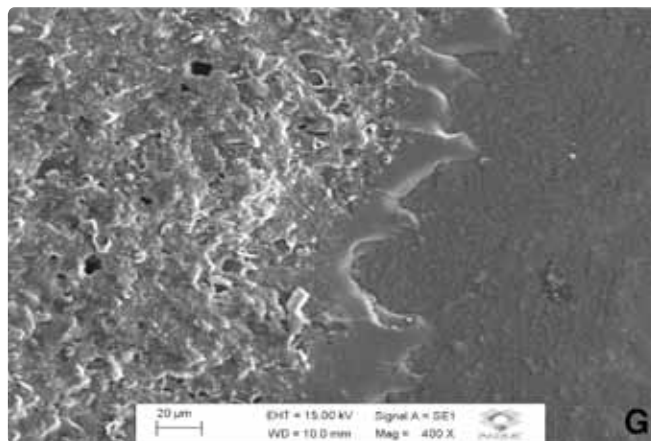


Figure 3G: SEM of representative specimens (original magnification of 400×) of a mixed failure of the ceramic surface with SBU+LC application

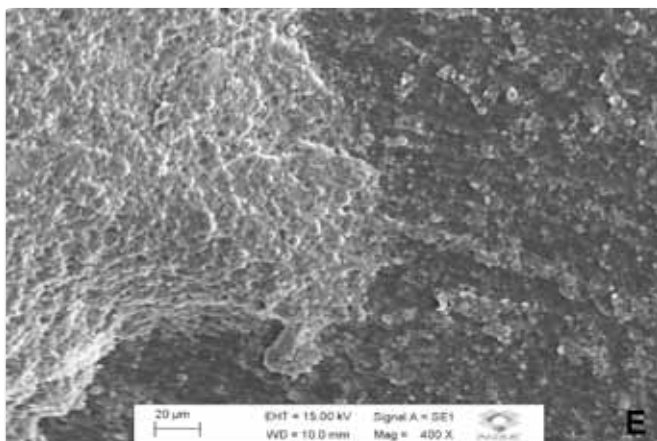


Figure 3E: SEM of representative specimens (original magnification of 400×) of a mixed failure of the ceramic surface with SB2+LC application.

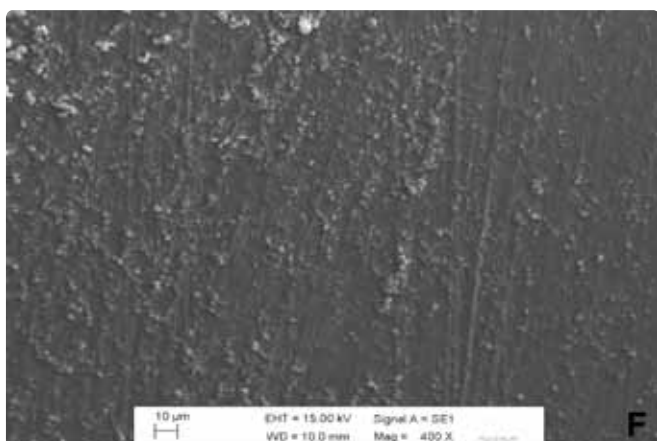


Figure 3F: SEM of representative specimens (original magnification of 400×) of an adhesive failure of the ceramic surface with SBU application

involved in the bonding interface. In general, the results show the importance of the previous light-curing of bonding adhesives inside ceramics crowns, in particular for SBMP and SB2 adhesives. The application of bonding adhesives without previous light-curing can influence the polymerization of the resin cement and inadequate polymerization could prevent the formation of the necessary covalent bonds.³¹ The SBU adhesive was the only bonding adhesive where previous photoactivation did not result in improvement of the adhesive interface compared to the previous light-curing group.

Some limiting factors of this study include the lack of immediate evaluation of the bond strength and mechanical properties of the materials as well as limited aging procedures. Future studies should involve materials that promote effective ceramic crown cementation in the search for the best clinical protocol, especially for long-term analysis. Thus, the materials involved in the resin cementation process are essential for the longevity and clinical success of indirect restorations.

CONCLUSION

According to the obtained results, it was concluded that the application of bonding adhesives on the silanized ceramic surface was unnecessary. Among the bonding adhesives studied, SBU was the only material where previous light curing was not necessary. For the other bonding adhesives, SBMP and SB2, previous light-curing was necessary to optimize the bond strength of the adhesive interface.

MANUFACTURER'S DETAILS

- IPS e-max CAD, Ivoclar Vivadent, Schaan, Liechtenstein
- Z350 XT, 3M ESPE, St. Paul, MN, USA
- Ultrasonic Unit, Cristófoli, Campo Mourão, PR, Brazil
- Hydrofluoric acid gel, Dentsply, Petrópolis, RJ, Brazil

- Phosphoric acid, Ultradent Products Inc., South Jordan, UT, USA
- RelyX Ceramic Primer, 3M ESPE, St. Paul, MN, USA
- Adper Scotchbond Multi-Purpose; 3M ESPE, St. Paul, MN, USA
- Adper Single Bond 2; 3M ESPE, St. Paul, MN, USA
- Single Bond Universal; 3M ESPE, St. Paul, MN, USA
- VALO, Ultradent Products Inc., South Jordan, UT, USA
- RelyX Ultimate, 3M ESPE, St. Paul, MN, USA
- RD-7, Ecel Ind. Com., Ribeirão Preto, SP, Brazil
- Model 521.4, Ética, São Paulo, SP, Brazil
- IsoMet 5000, Buehler Ltd., Lake Bluff, IL, USA
- Super Bonder Flex Gel, Loctite, Itapevi, SP, Brazil
- Odeme Microtensile OM 100, Odeme Dental Research, Luzerna, SC, Brasil
- Stemi SV11, Carl Zeiss AG, Orberkochen, Germany
- EVO HD LS-15, Carl Zeiss AG, Orberkochen, Germany
- Clássico, São Paulo, SP, Brazil
- Extec Corp., Enfield, CT, USA
- NHT3, Anton Paar, Graz, Áustria

DISCLOSURE

The authors declare that they have no conflict of interest.

REFERENCES

- Pieger S, Salman A, Bidra AS. Clinical outcomes of lithium disilicate single crowns and partial fixed dental prostheses: A systematic review. *J Prosthet Dent* 2014;**112**:22-30.
- Lührs AK, De Munck J, Geurtsen W, Van Meerbeek B. Composite cements benefit from light-curing. *Dent Mater* 2014;**30**:292-301.
- Toman M, Toksavul S. Clinical evaluation of 121 lithium disilicate all-ceramic crowns up to 9 years. *Quintessence Int* 2015;**46**:189-97.
- Murillo-Gómez F, Rueggeberg FA, De Goes MF. Short-and-long term bond strength between resin cement and glass-ceramic using a silane-containing universal adhesive. *Oper Dent* 2017;**42**:514-25.
- Walton JN, Gardner FM, Agar JR. A survey of crown and fixed partial denture failures: length of service and reasons for replacement. *J Prosthet Dent* 1986;**56**:416-21.
- Rathke A, Hokenmaier G, Muche R, Haller B. Effectiveness of the bond established between ceramic inlays and dentin using different luting protocols. *J Adhes Dent* 2012;**14**:147-54.
- Taskanak B, Sertgöz A. Two-year clinical evaluation of lithia-disilicate-based all-ceramic crowns and fixed partial dentures. *Dent Mater* 2006;**22**:1008-13.
- Kelly JR, Benetti P. Ceramic materials in dentistry: historical evolution and current practice. *Aust Dent J* 2011;**56**:84-96.
- Kalavacharla VK, Lawson NC, Ramp LC, Burgess JO. Influence of etching protocol and silane treatment with a universal adhesive on lithium disilicate bond strength. *Oper Dent* 2015;**40**:372-8.
- Lawson NC, Burgess JO. Dental ceramics: a current review. *Compend Contin Educ Dent* 2014;**35**:161-8.
- Ilie N, Hickel Reinhard. Correlation between ceramics translucency and polymerization efficiency through ceramics. *Dent Mater* 2008;**24**:908-14.
- Diaz-Arnold AM, Vargas MA, Haselton DR. Current status of luting agents for fixed prosthodontics. *J Prosthet Dent* 1999;**81**:135-41.
- Sundfeld Neto D, Naves LZ, Costa AR, Correr AB, Consani S, Borges GA et al. The effect of hydrofluoric acid concentration on the bond strength and morphology of the surface and interface of glass ceramics to a resin cement. *Oper Dent* 2015;**40**:470-9.
- Pashley DH, Tay FR, Breschi L, Tjäderhane L, Carvalho RM, Carrilho M et al. State of the art etch-and-rinse adhesives. *Dent Mater* 2011;**27**:1-16.
- Tian T, Tsoi JK, Matinlinna JP, Burrow MF. Aspects of bonding between resin luting cements and glass ceramic materials. *Dent Mater* 2014;**30**:147-62.
- Van Dijken JW, Hasselrot L. A prospective 15-year evaluation of extensive dentin-enamel-bonded pressed ceramic coverages. *Dent Mater* 2010;**26**:929-39.
- Radovic I, Monticelli F, Goracci C, Vulicevic ZR, Ferrari M. Self-adhesive resin cements: a literature review. *J Adhes Dent* 2008;**10**:251-8.
- Oztürk E, Hickel R, Bolay S, Ilie N. Micromechanical properties of veneer luting resins after curing through ceramics. *Clin Oral Investig* 2012;**16**:139-46.
- Benetti P, Della Bona A, Kelly JR. Evaluation of thermal compatibility between core and veneer dental ceramics using shear bond strength test and contact angle measurement. *Dent Mater* 2010;**26**:743-50.
- Ye Q, Spencer P, Wang Y. Nanoscale patterning in crosslinked methacrylate copolymer networks: an atomic force microscopy study. *J Appl Polym Sci Symp* 2007;**106**:3843-51.
- Beier US, Kapferer I, Burtscher D, Dumfahrt H. Clinical performance of porcelain laminate veneers for up to 20 years. *Int J Prosthodont* 2012;**25**:79-85.
- Peutzfeldt A, Sahafi A, Flury S. Bonding of restorative materials to dentin with various luting agents. *Oper Dent* 2011;**36**:266-73.
- Van Meerbeek B, Yoshihara K, Yoshida Y, Mine A, De Munck J, Van Landuyt KL. State of the art of self-etch adhesives. *Dent Mater* 2011;**27**:17-28.
- Breschi L, Mazzoni A, Ruggeri A, Cadenaro M, Di Lenarda R, Dorigo, ES. Dental adhesion review: aging and stability of the bonded interface. *Dent Mater* 2008;**24**:90-101.
- Van Meerbeek B, De Munck J, Yoshida Y, Inoue S, Vargas M, Vijay P et al. Buonocore memorial lecture. Adhesion to enamel and dentin: current status and future challenges. *Oper Dent* 2003;**28**:215-35.
- Silva e Souza MH, Carneiro KG, Lobato MF, Silva e Souza P de A, De Góes MF. Adhesive systems: important aspects related to their composition and clinical use. *J Appl Oral Sci* 2010;**18**:207-14.
- Gaglianone LA, Lima AF, Gonçalves LS, Cavalcanti NA, Aguiar FH, Marchi GM. Mechanical properties and degree of conversion of etch-and-rinse adhesive systems cured by a quartz tungsten halogen lamp and a light-emitting diode. *J Mech Behav Biomed Mater* 2012;**12**:139-43.

28. Hitz T, Stawarczyk B, Fischer J, Hämmerle CH, Sailer I. Are self-adhesive resin cements a valid alternative to conventional resin cements? A laboratory study of the long-term bond strength. *Dent Mater* 2012; **28**:1183-90.
29. Strazzi-Sayon HB, Chimanski A, Toshimura HN, Dos Santos PH. Effect of previous photoactivation of the adhesive system on the color stability and mechanical properties of resin components in ceramic laminate veneer luting. *J Prosthet Dent* 2018; **120**:631.e1-631.e6.
30. Higashi C, Michel MD, Reis A, Loguercio AD, Gomes OM, Gomes JC. Impact of adhesive application and moisture on the mechanical properties of the adhesive interface determined by the nano-indentation technique. *Oper Dent* 2009; **34**:51-7.
31. Catelan A, Pollard T, Bedran-Russo AK, Santos PD, Ambrosano G, Aguiar F. Light-curing time and aging effects on the nanomechanical properties of methacrylate- and silorane-based restorations. *Oper Dent* 2014; **39**:389-97.
32. Della Bona A, Shen C, Anusavice KJ. Work of adhesion of resin on treated lithia disilicate-based ceramic. *Dent Mater* 2004; **20**:338-44.
33. Borges GA, Spohr AM, de Góes MF, Sobrinho, LC, Chan DC. Effect of etching and airborne particle abrasion on the microstructure of different dental ceramics. *J Prosthet Dent* 2003; **89**:479-88.
34. Prata RA, de Oliveira VP, de Menezes FC, Borges GA, de Andrade OS, Gonçalves LS. Effect of "try-in" paste removal method on bond strength to lithium disilicate ceramic. *J Dent* 2011; **39**:863-70.
35. Ilie N, Hickel Reinhard. Correlation between ceramics translucency and polymerization efficiency through ceramics. *Dent Mater* 2008; **24**:908-14.
36. Armstrong S, Breschi L, Özcan M, Pfefferkom F, Ferrari M, Van Meerbeek. Academy of Dental Materials guidance on in vitro testing of dental composite bonding effectiveness to dentin/enamel using micro-tensile bond strength (μ TBS) approach. *Dent Mater* 2017; **33**:133-43.