

Efficacy of a universal adhesive on the bond strength of a luting cement to leucite-reinforced glass-ceramic

Abstract

Objective

The present study compared the efficacy of a universal adhesive containing silane, bis-GMA and 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) monomer with that of silane applied alone or combined with bis-GMA or 10-MDP, but in separate steps, on the microtensile bond strength of a CAD/CAM leucite-reinforced glass-ceramic to a resin cement.

Materials and methods

Sixty-four blocks from IPS Empress CAD (Ivoclar Vivadent) were etched (5% hydrofluoric acid) and treated with:

- (1) RelyX Ceramic Primer (3M ESPE; control group; group 1);
- (2) RelyX Ceramic Primer + Adper Scotchbond Multi-Purpose Adhesive (group 2);
- (3) Single Bond Universal Adhesive (3M ESPE; group 3);
- (4) CLEARFIL PORCELAIN BOND ACTIVATOR + CLEARFIL SE BOND PRIMER (both Kuraray Noritake Dental; group 4).

The blocks were bonded in pairs with RelyX ARC (3M ESPE) and sectioned into microbars, which were submitted to microtensile testing. Microtensile bond strength data (MPa) were analyzed by 1-way ANOVA and Tukey tests ($\alpha = 0.05$). Failure mode was determined under a stereomicroscope ($\times 20$).

Results

The control group, group 2 and group 4 exhibited microtensile bond strength values not statistically different from each other, but higher than those of group 3. Group 2 presented the lowest percentage of adhesive failures and the highest percentage of cohesive failures within the resin cement.

Conclusion

The universal adhesive showed the worse performance on the microtensile bond strength of a CAD/CAM leucite-reinforced glass-ceramic with a resin cement when compared with that of silane applied alone or combined with bis-GMA or 10-MDP, but in separate steps. Long-term studies investigating how these groups behave when submitted to hydrothermal aging, simulating the oral environment over time, are necessary.

Keywords

Dental bonding; adhesive; dental porcelain.

Francesco Saverio Ludovichetti,^a Samira Branco Martins,^a Flávia Zardo Trindade,^a Filipe de Oliveira Abi Rached,^a Gustavo Moncada,^b Gelson Luis Adabo^a & Renata Garcia Fonseca^a

^a Department of Dental Materials and Prosthodontics, Araraquara Dental School, Universidade Estadual Paulista, Araraquara, São Paulo, Brazil

^b Department of Oral Rehabilitation, University of los Andes, Santiago Chile

Corresponding author:

Dr. Francesco Saverio Ludovichetti

Departamento de Materiais Odontológicos e Prótese
Faculdade de Odontologia de Araraquara
Univ. Estadual Paulista
Rua Humaitá, nº 1680
Araraquara—São Paulo
CEP: 14801-903
Brazil

T +39 340 8573561
f.ludovichetti@gmail.com

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Introduction

Nowadays, the increasing demand for esthetic restorations has stimulated the development of esthetic restorative materials and, concomitantly, new adhesive systems. Although zirconia and lithium disilicate ceramics have been widely used for manufacturing metal-free restorations, in the case of veneers, inlays/onlays and even anterior crowns, leucite-reinforced glass-ceramics could be an interesting option considering their esthetic potential and higher mechanical strength compared with conventional feldspathic porcelains.¹

In order to achieve successful cementation, both micromechanical interlocking and chemical bonding should be present.¹ For silica-based ceramics, the first bonding mechanism is successfully achieved with hydrofluoric acid (HF), which dissolves the glassy matrix surrounding the crystalline phase, creating a microretentive surface and consequently, an increased bonding area.²⁻⁴ The chemical bond between the silica of glass-ceramics (Si–O–Si formation by means of condensation reaction) and the organic groups of resin cements is achieved via silane coupling agents, more commonly methacryloxypropyltrimethoxysilane (MPS).^{1,5-7} Therefore, for bonding glass-ceramics, etching with HF followed by silane is the classical protocol.⁸

More recently, universal adhesives were developed with the aim of simplifying the time-consuming procedure of conditioning both the tooth and the restoration surface with etch-and-rinse adhesives, providing a single product that meets the needs of different substrates. Some of these universal adhesives (Single Bond Universal Adhesive, Scotchbond Universal, CLEARFIL Universal Bond) contain as main components silane, 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) monomer and dimethacrylate (bis-GMA), all together in a single bottle. According to the manufacturer (3M ESPE) of the Single Bond Universal Adhesive, each component was added with a specific purpose, that is, 10-MDP to provide chemical bonding to zirconia, alumina and metals; and silane to chemically bond to glass-ceramic surfaces. The application of a thin layer of resin to the previously HF-etched and silane-treated ceramic surface improves adhesive bonding by providing better wetting of the ceramic surface by the resin cement.^{2,3} The bis-GMA monomer commonly present in universal adhesives can achieve this purpose, in addition to acting as a cross-linker,⁹ without the need for an additional step.

However, the combination of all these components in a single bottle might cause some interference in their roles, either by a chemical interaction between them^{10,11} or even by some competition to react with the substrate.¹² In a study in which zirconia was air-abraded with silica-modified Al₂O₃ particles, the authors raised the possibility of having influenced competition between the silane and 10-MDP of Scotchbond Universal adhesive to interact with the ceramic surface, thus preventing each other from acting effectively.¹² The chemical affinity between 10-MDP and zirconia is well established in the literature.^{13,14} However, since the efficacy of 10-MDP on adhesive bonding to glass-ceramic has been insufficiently investigated, it is not known if the silane and 10-MDP would have their roles compromised when applied to the glass-ceramic in a single step.

The aim of the present study was to compare the efficacy of a universal adhesive containing silane, bis-GMA and 10-MDP with that of silane applied alone (control group) or combined with bis-GMA or 10-MDP, but in separate steps, on the microtensile bond strength (MTBS) of a CAD/CAM leucite-reinforced glass-ceramic with a resin cement. The null hypothesis was that the performance of the universal adhesive would be similar to that of the silane applied alone (control group) or combined with bis-GMA or 10-MDP, but in separate steps.

Material and methods

The materials used in the present study are summarized in **Table 1**.

Specimen preparation

Sixty-four ceramic blocks (IPS Empress CAD, Ivoclar Vivadent) were obtained (12 × 10 × 5 mm) using a saw (IsoMet 1000, Buehler) with a water-cooled diamond disk and were polished under wet conditions with 180, 400 and 600 grit silicon carbide abrasive papers.

One surface of each block was etched with 5% HF for 1 min, washed under tap water and dried at room temperature for 24 h. The surfaces received

- (1) RelyX Ceramic Primer (3M ESPE; RX; control group);
- (2) RelyX Ceramic Primer + Adper Scotchbond Multi-Purpose Adhesive (3M ESPE; RXASM);
- (3) Single Bond Universal Adhesive (SBU);

Material	Composition
IPS Empress CAD	Leucite-reinforced ceramic (SiO ₂ , Al ₂ O ₃ , K ₂ O, Na ₂ O, other oxides)
RelyX Ceramic Primer	MPS, ethyl alcohol, water
Adper Scotchbond Multi-Purpose Adhesive	Bis-GMA, HEMA, triphenylantimony
Single Bond Universal Adhesive	Bis-GMA, HEMA, decamethylene DMA, MDP, silane, ethanol, water, silane-treated silica, 2-propenoic acid, 2-methyl-, reaction products with 1,10-decanediol and phosphorous oxide, copolymer of acrylic and itaconic acid, dimethylaminobenzoate(-4), camphorquinone, (dimethylamino)ethyl methacrylate, methyl ethyl ketone
CLEARFIL SE BOND PRIMER	MDP, HEMA, hydrophilic aliphatic dimethacrylate, dl-camphorquinone, water
CLEARFIL PORCELAIN BOND ACTIVATOR	MPS, hydrophobic aromatic dimethacrylate, others
RelyX ARC	Bis-GMA, TEGDMA, silane-treated ceramic, silane-treated silica, reacted polycaprolactone polymer, 2-benzotriazolyl-4-methylphenol, benzoyl peroxide

Table 1

MPS = methacryloxypropyltrimethoxysilane; Bis-GMA = bisphenol-A diglycidyl ether dimethacrylate; HEMA = hydroxyethyl methacrylate; DMA = dimethacrylate; MDP = 10-methacryloyloxydecyl dihydrogen phosphate; TEGDMA = triethylene glycol dimethacrylate.

Table 1
Materials used in the study.

4) CLEARFIL system: CLEARFIL PORCELAIN BOND ACTIVATOR + CLEARFIL SE BOND PRIMER (both Kuraray Noritake Dental; CLESYS).

These were applied according to **Table 2** with a microbrush. In group 3, the silane and 10-MDP were in the same adhesive solution. In group 4, the silane (CLEARFIL PORCELAIN BOND ACTIVATOR) and 10-MDP (CLEARFIL SE BOND PRIMER) were in different bottles.

RelyX ARC resin cement (3M ESPE) was mixed for 20 s and the blocks were bonded in pairs, resulting in 4 groups (8 blocks/group). Therefore, the block was considered the experimental unit. From each block, 10 microbars were obtained. The MTBS values from 10 microbars were averaged to provide a single mean value per block. The mean MTBS values of the 8 blocks were averaged to provide the mean value of each group. The blocks were submitted to a mass of 1,000 g for 5 min, and during this time, the cement excess was removed. All 4 sides of the blocks were photopolymerized (Radii-cal light-curing unit, SDI) for 40 s at an intensity of 800 mW/cm².

Microtensile bond strength test

The blocks were stored in distilled water for 48 h and were then sectioned under constant irrigation into 1.0 × 1.0 mm microbars using a saw machine and a 0.3 mm thick diamond-coated cutting disk. Each specimen consisted of 10 microbars, which were fixed to a stainless-steel attachment unit. The MTBS was tested by applying tensile load to the bonded interface in a mechanical testing machine (EMIC

DL2000, EMIC Equipment and Systems Testing, São José dos Pinhais, PR, Brazil) using a 1 kN load cell at a crosshead speed of 0.5 mm/min.

Statistics

MTBS data (MPa) were analyzed by 1-way ANOVA followed by the Tukey honestly significant difference (HSD) post hoc test ($\alpha = 0.05$) to determine differences between the different bonding protocols. Statistical analysis was performed using the IBM SPSS Statistics software (Version 20).

Failure mode analysis

After the MTBS test, the fractured surfaces were examined under a stereomicroscope (M80, Leica Microsystems) at ×20 magnification by a single trained observer, and the failure mode was classified as adhesive (the complete ceramic surface was visible), mixed (both the ceramic surface and a cement layer were visible), cohesive within the resin cement (almost all of the surface was covered with resin cement) and cohesive within the ceramic (**Fig. 1**).

Results

Microtensile bond strength

The 1-way ANOVA ($F = 10.90, P < 0.001$) revealed significant differences in MTBS values between the groups. **Table 3** presents the mean MTBS values, standard deviations and statistical results verified by the Tukey HSD test. The RX,

Table 2

Material	Application method
RelyX Ceramic Primer	Applied for 5 s and air-dried
Adper Scotchbond Multi-Purpose Adhesive	Applied, air-dried and photopolymerized for 20 s
Single Bond Universal Adhesive	Applied for 20 s, air-dried and photopolymerized for 10 s
CLEARFIL SE BOND PRIMER/CLEARFIL PORCELAIN BOND ACTIVATOR	1 drop of each bottle was dispensed into a well of the mixing dish and the mixture was applied for 5 s and air-dried

Table 2

Application of the bonding agents according to the manufacturers' instructions.

Fig. 1

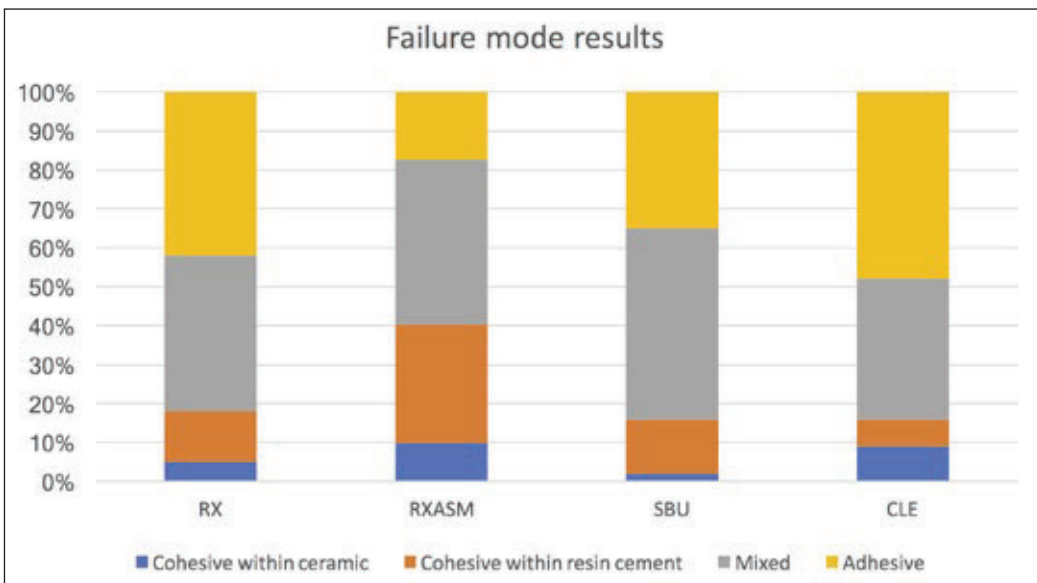


Fig. 1

Representation of the failure mode percentage after the MTBS test: cohesive within the ceramic; cohesive within the resin cement (almost all of the surface was covered with resin cement); mixed (both ceramic surface and a cement layer were visible); adhesive (complete ceramic surface was visible).

RXASM and CLESYS groups exhibited MTBS values statistically similar to each other and higher than those of the SBU group.

Failure mode analysis

Table 4 presents the percentage distribution of failure mode between the groups. The RXASM group exhibited the lowest percentage of adhesive failures (2.0, 2.4 and 2.7 times lower than those presented by the SBU, RX and CLESYS groups, respectively) and the highest percentage of cohesive failures within the resin cement (2.2, 2.3 and 4.3 times higher than those presented by the SBU, RX and CLESYS groups, respectively).

Discussion

The research hypothesis, which assumed that the performance of the universal adhesive would be similar to that of the silane applied alone (control group) or combined with bis-GMA or 10-MDP, but in separate steps, was rejected, since SBU exhibited the lowest MTBS values when compared with the other groups. In the present study, the RX, RXASM and CLESYS

groups exhibited MTBS values similar to each other and higher than those of the SBU group. In the RX group, only the silane RX was applied after etching the glass-ceramic surface with HF. According to Makishi et al., the 3 silanol groups resulting from the hydrolysis of the silane RX (which contains 3-methacryloxypropyltrimethoxysilane) form covalent bridges with the hydroxyl groups of the glass-ceramic phase.⁹ Afterward, the organofunctional groups of the silane react with the resin monomers of the resin cement.⁴ The application of only a silane coupling agent (RX group) over the etched surface has been indicated for a long time as a chemical treatment to improve the bonding at the glass-ceramic/resin cement interface,^{15, 16} and therefore, this group was considered the control treatment in the present study.

It was expected that the use of the hydrophobic unfilled RXASM after application of the silane (RXASM group) would increase the bond strength in comparison to the RX group, keeping in mind that the resin monomers of the adhesive might improve the wetting of the glass-ceramic by the resin cement;^{2, 17} however, no difference in MTBS values was found between these

Table 3
MTBS mean values and standard deviations (MPa).

RX	RXASM	SBU	CLESYS
31.0 ± 5.4 (A)	30.8 ± 1.6 (A)	19.1 ± 5.9 (B)	29.1 ± 5.3 (A)

Table 3

Different letters indicate significant differences ($P < 0.05$).

Table 4
Percentage of each failure mode in the testing groups.

	RX	RXASM	SBU	CLESYS
Cohesive within ceramic	5%	10%	2%	9%
Cohesive within resin cement	13%	31%	14%	7%
Mixed	40%	42%	49%	36%
Adhesive	42%	17%	35%	48%

Table 4

RX = RelyX Ceramic Primer (control group); RXASM = RelyX Ceramic Primer + Adper Scotchbond Multi-Purpose Adhesive; SBU = Single Bond Universal Adhesive; CLESYS = CLEARFIL PORCELAIN BOND ACTIVATOR + CLEARFIL SE BOND PRIMER.

groups. The similarity between the RX and RXASM groups might have been due to the already low viscosity of the RelyX ARC resin cement.¹⁸ If a high-viscosity resin cement had been used, the adhesive may have played a role in improving the wettability. In the study by Fabião et al. in which 2 glass-ceramics were HF-etched and silane-treated with RX, when the RXASM was used before the RelyX U100 resin cement, a significantly higher bond strength and a slight decrease in adhesive failures and increase in cohesive failures within the ceramic were observed in comparison to when this cement was used without that adhesive.¹⁷ These authors related this improvement to the increase in the wetting ability of the cement provided by the adhesive.¹⁷ Hooshmand et al. observed similar behavior regarding bond strength and failure mode after having treated the silanized leucite-reinforced ceramic surface with a thin layer of unfilled resin.⁷ The authors attributed the increase in cohesive failures within the resin cement to a reduction in the number and size of the flaws at the adhesive interface. According to them, this situation represents the best bonding condition. In the present study, despite the statistical similarity in MTBS values between the RX and RXASM groups, the RXASM group exhibited, in comparison with the RX group, a decrease in the percentage of adhesive failures (from 42.0% to 17.0%) and an increase in the percentage of cohesive failures within the resin cement (from 13.0% to 31.0%) and the ceramic (from 5.0% to 10.0%). Therefore, although no significant difference in the mean MTBS values was observed, the change in the failure mode indicates that the application of a thin layer of adhesive after the silane may provide better wetting of the glass-ceramic. The adhesive strengthened the bond of the resin cement to the ceramic surface. Besides the probable mechanical role

(wetting ability) of the RXASM at this interface, its monomers co-polymerize with the methacrylate end of the silane and with the monomers of the resin cement.⁹

The CLESYS group, which had the silane MPS (as the RX group) and the 10-MDP phosphate monomer, in separate bottles, exhibited no significant difference in MTBS values and a similar failure mode pattern with equal distribution between adhesive and mixed failures when compared with the RX group. This similarity was probably due to the chemical reaction that occurred between the silane and the hydroxyl groups of the glass-ceramic in both groups.^{4, 9} Having the 10-MDP applied afterward (CLESYS group) did not contribute to improving the MTBS value. In order to compare the results of the present study with those in the literature, no studies were found that evaluated in glass-ceramics the efficacy of a silane coupling agent applied separately followed or not by an application of 10-MDP, as in the RX and CLESYS groups of the present study. In general, the silane and the 10-MDP were in the same bottle mixed with other ingredients, creating another situation.

Unlike the RX, RXASM and CLESYS groups, in which the MPS-based silane was applied in a separate step, alone (RX group) or combined with the bis-GMA monomer (RXASM group) or with 10-MDP (CLESYS group), the universal adhesive used in the SBU group had all these components in a single bottle. In order to justify the poorer performance of the SBU group in comparison with the RX, RXASM and CLESYS groups, 2 possible explanations were found in the literature:

- (1) In SBU, the bis-GMA monomer, by being in the same bottle as the silane, may have inhibited the chemical reaction that occurred between the silane and the hydroxyl groups of the ceramic.^{10, 11}

(2) The low pH (2.7) of the tested universal adhesive may have promoted premature silane hydrolysis followed by dehydration condensation, resulting in the formation of oligomers that cannot bond to glass.⁸ Yoshihara et al. did not detect silanol in the Scotchbond Universal adhesive (same composition as SBU), although the manufacturer states the presence of silane in its composition.⁸ Some authors have recommended using a separate silane primer to achieve enough silane-coupling effect on glass-ceramics.^{8, 9, 19} Both explanations cited here justify the insufficient effectiveness of the silane incorporated in SBU.

In the RXASM group, silane and adhesive containing bis-GMA were applied separately, allowing the silane to react with the glass-ceramic. When the failure mode of the RXASM and SBU groups was compared, the percentage of adhesive failures of the SBU group was twice that of the RXASM group (35% against 17%). Some studies have also demonstrated superiority of the combination of RX and RXASM over SBU^{11, 20} and Scotchbond Universal adhesive,⁹ exhibit similar formulations. When the SBU and CLESYS groups were compared, although both adhesives had silane and 10-MDP adhesive monomers, CLESYS does not contain bis-GMA and, unlike SBU, comes in 2 bottles, 1 (CLEARFIL PORCELAIN BOND ACTIVATOR) containing the silane MPS (applied in a separate step) and the other (CLEARFIL SE BOND PRIMER) the 10-MDP adhesive monomer.

Therefore, in the SBU group, the mean MTBS value probably resulted from the mechanical interlocking provided by the HF etching^{3, 19} added or not to an eventual chemical reaction between the MDP monomer and the glass-ceramic components that, if it occurred, was presumably weaker than that between the silane and the glassy matrix. During the development of the present study, a group HF-etched and treated only with CLEARFIL SE BOND PRIMER (which contains only 10-MDP) was also tested under the same conditions as those of the other 4 groups (although it was not included in the study), achieving a mean MTBS value of 21.5 MPa, which is very close to that found for the SBU group (19.1 MPa). Kim et al., evaluating the performance of universal adhesives on the microshear bond strength to leucite-reinforced ceramic, found no significance difference between SBU and All-Bond Universal, which contains basically the MDP adhesive monomer,

similar to CLEARFIL SE BOND PRIMER.¹¹ Studies that elucidate the chemical interaction between the 10-MDP monomer and glass-ceramic are crucial to prove or disprove this assumption.

Conclusion

The present study indicated that SBU, which contains silane, bis-GMA and 10-MDP in a single bottle, showed the worst performance when compared with that of silane applied alone or combined with bis-GMA or with 10-MDP, but in separate steps, on the MTBS of a CAD/CAM leucite-reinforced glass-ceramic with a resin cement. However, the long-term durability of the groups was not evaluated and is crucial to determine their effectiveness. Studies that investigate how these groups behave when submitted to hydrothermal aging, simulating the oral environment over time, are necessary.

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Competing interests

The authors declare that they have no competing interests.

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