Shear bond strength of two composite resin cements to multiphase composite resin after different surface treatments and two glass-ceramics

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Abstract

Aim: To compare the shear bond strength (SBS) after aging of two dual-curing composite resin cements to multiphase composite resin (experiment) and glass-ceramics (control).

Methods: Seventy computer-aided design/computer-aided manufacturing (CAD/CAM) blocks were prepared: 24 multiphase composite resin blocks (Lava Ultimate; experiment), and 12 control blocks (groups 5 and 6: 6 IPS e.max CAD, 6 IPS Empress CAD). Surface treatments of the experiment groups were: 1) Al_2O_3 airborne particle abrasion; 2) bur-roughening; 3) silica-coated aluminum oxide particle abrasion; and 4) hydrofluoric (HF) acid etching. Per study group, Variolink II (a) and RelyX Ultimate (b) were used as cements. Per treatment group, four cement cylinders were adhered to the conditioned blocks (n = 12). After thermocyclic aging (10.000x, 5°C to 55°C), notch-edge shear testing was applied. Modes of failure were examined. A *P* value of 0.05 was considered significant.

Results: Groups 1a (18.68 ± 3.81) and 3a (17.09 ± 3.40) performed equally to 6a (20.61 ± 4.10). Group 5a (14.39 ± 2.80) did not significantly differ from groups 1a, 3a, and 4a (15.21 ± 4.29). Group 2a (11.61 ± 3.39) showed the lowest bond strength. For the RelyX Ultimate specimens, mean bond strengths were: 1b (18.12 ± 2.84) > 4b (15.57 ± 2.31) > 2b (12.34 ± 1.72) = 3b (11.54 ± 2.45) = 6b (12.31 ± 1.87) > 5b (0.78 ± 0.89). Failure mode analysis showed a significant association between bond strength values and modes of failure (chi-square).

Conclusion: The SBS of the composite cements to the multiphase composite resin that was treated by Al_2O_3 or silica-coated aluminum oxide particle abrasion is comparable to the bond of the control groups.

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Introduction

Indirect computer-aided design/computer-aided manufacturing (CAD/CAM)-milled restoration materials used nowadays include feldspathic, leucite-reinforced and lithium disilicate ceramics.[1,2] Recently, hybrid composite materials have been introduced. Benefits are easy intraoral repair with light-cured restoratives and a faster production rate, since firing is not needed.

Prior to cementation, the restoration material has to be conditioned. Different substrates require different surface-conditioning techniques.[1,3] For conventional glass-ceramic restorations, the highest bond strength can be obtained by etching the intaglio surface with a 5% to 9% hydrofluoric (HF) acid, then applying a silane coupling agent.[2,3] For polymeric materials, airborne particle abrasion with silica-coated alumina particles (30 µm), followed by silanization, results in a durable bond that is resistant to aging.[4-7]

Recent composite-based CAD/CAM materials all differ in their composition, but the underlying principle remains the same. They consist of a resin matrix and filler particles but vary in weight percentage, filler content, and size. Lava Ultimate (3M ESPE) is an example of such a CAD/CAM material and is constructed of nanomer and nanoparticles, with a total of nanoceramic material content by weight of approximately 80%, embedded in a resin matrix. There are two types of nanomer particles: silica nanomers of 20-nm diameter, and zirconia nanomers of 4- to 11-nm diameter. Nanoclusters are bound aggregates of these nanomers, with an average particle size of 0.6 to 10 µm. The manufacturer's recommendations for the surface conditioning prior to cementation consist of sandblasting with aluminum oxide particles of ≤ 50 µm at two bars until the bonding surface appears matte. The surface is then cleaned with alcohol and air dried, and Scotchbond Universal Adhesive (3M ESPE) is applied for 20 s.

However, since this material also consists of silica and zirconia particles, a more optimal way of conditioning is possible.[3-5] Microtensile and macroshear tests are most frequently used to evaluate adhesion.[8]

The main objective of this study was to investigate the bond of two composite cements to multiphase composite resin after different surface treatments and compare this to two glass-ceramics. Within the CAD/CAM field, lithium disilicate and leucite-reinforced ceramics are materials that share a substantial part of the range of indications with that of multiphase composite resin. In a fatigue resistance study, it was shown that there was no significant difference between lithium disilicate and multiphase composite resin crowns.[9] These materials therefore can be regarded as control groups. The use of dual-polymerizing resin cements is widespread for the cementation of all-ceramic restorations.[10] Variolink II (Ivoclar Vivadent) and RelyX Ultimate (3M ESPE) were the dual-curing resin cements used in this study for the control and experiment blocks, respectively. The null hypothesis tested was that there is no difference in bond strength between composite cements and the different substrates (multiphase composite resin, leucite-reinforced and lithium disilicate ceramic). The cement type was expected not to influence the bond strength.

Materials and methods

Seventy CAD/CAM blocks were prepared: 24 Lava Ultimate A3-HT/14L (3M ESPE), 6 sintered IPS e.max CAD HT A2/C14 (Ivoclar Vivadent), and 6 IPS Empress CAD A2 Multi C14L (Ivoclar Vivadent). To standardize surface texture, all the blocks were ground using coarse and medium grit Sof-Lex Extra-Thin Contouring and Polishing Discs (3M ESPE).

Table 1 Product name, type, manufacturer, composition, and batch number of the materials used in this study

Product name	Type	Manufacturer	Composition	Batch number
Lava Ultimate	Nano resin ceramic	3M ESPE; St. Paul, USA	Zirconia-silica nanoclusters, resin matrix	N4400260
IPS e.max CAD	Lithium disilicate	Ivoclar Vivadent; Schaan, Liechtenstein	Silica, lithium oxide, magnesium oxide, lithium oxide, potassium oxide, aluminum oxide, phosphorus pentoxide, other oxides	R64197 R70382
IPS Empress CAD	Leucite reinforced	Ivoclar Vivadent	Silica, aluminum oxide, potassium oxide, Na2O, CaO, other oxides, pigments	R72464
Variolink II	Dual-curing resin cement	Ivoclar Vivadent	Bis-GMA, urethane dimethacrylate, triethylene glycol dimethacrylate, inorganic fillers, barium glass, ytterbium trifluoride, Ba-Al-fluorosilicate glass, spheroid mixed oxide, catalysts, stabilizers, pigments	Base: P33894 R68680 Catalyst: P24937
RelyX Ultimate	Dual-curing resin cement	3M ESPE	Methylacrylate monomers, radiopaque, silanated fillers, initiator components, stabilizers, rheological additives, radiopaque alkaline fillers, fluorescence dye, dark cure activator, Scotchbond Universal Adhesive	506283 467130
ESPE Sil	Silane coupling agent	3M ESPE	3-methacryloxypropyl-trimethoxy silane, ethanol, ethyl alcohol	437637
Monobond Plus	Silane coupling agent	Ivoclar Vivadent	1% 3-methacryloxypropyl-trimethoxy silane, ethanol	S05679
Heliobond	Light-curing bonding agent	Ivoclar Vivadent	Bis-GMA (60% wt), triethylene glycol dimethacrylate (40% wt)	P06157
Scotchbond Universal Adhesive	Self-etch, bonding primer and silane coupling agent	3M ESPE	MDP phosphate monomer, dimethacrylate resins, HEMA, Vitrebond Copolymer, filler, ethanol, water, initiators, silane	503052
Aluminum oxide	Airsonic	Hager & Werken; Duisburg, Germany	Aluminum oxide particles, particle size: 50 µm	-
CoJet Sand	Sand	3M ESPE	Aluminum trioxide particles coated with silica, particle size: 30 µm	442859
IPS Ceramic Etching Gel	HF acid	Ivoclar Vivadent	5% HF acid	S13497
Liquid Strip – transparent	Glycerine gel	Ivoclar Vivadent	Glycerol	P28325

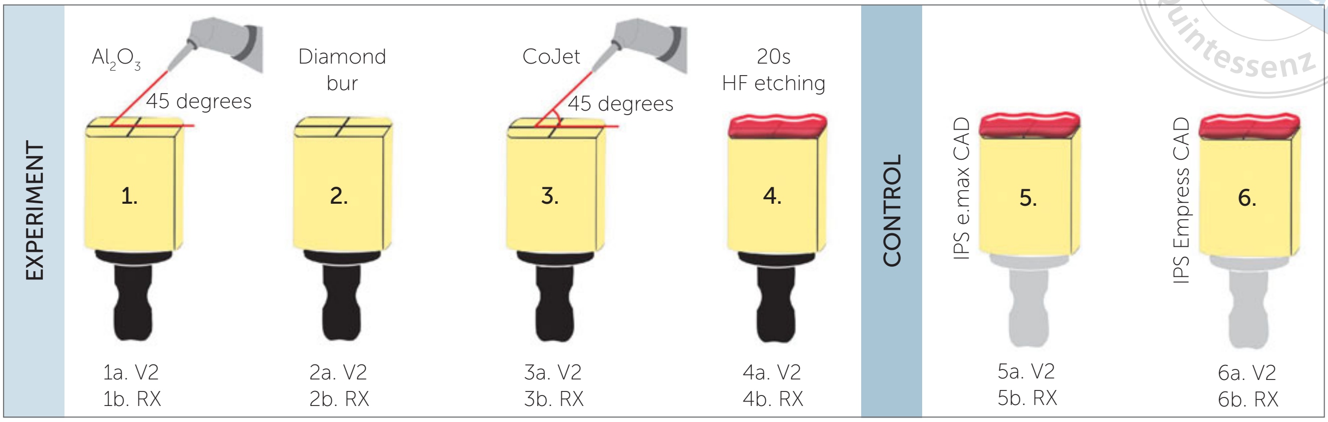

Fig 1 Schematic representation of the study groups (V2 = Variolink II; RX = RelyX Ultimate; n = 12). After exclusion of pretest failures, 10 values per group were used in the statistical analysis.

The blocks were randomly assigned to the treatment groups using the IBM SPSS 20 software package. Table 1 contains an overview of the materials used, and Figure 1 schematically depicts the different groups. Per CAD/CAM block, four cement cylinders were adhered to the conditioned surface (Fig 2).

Experiment groups

All experiment groups (1 to 4) consisted of six Lava Ultimate blocks. Surface conditioning procedures for the experiment groups consisted of:

1. Airborne particle abrasion with 50 µm aluminum oxide particles (Airsonic; Hager & Werken), 2 bar, angle 45 degrees, until surface appeared matte.
2. Roughening the surface with a red ring diamond shoulder bur in a red angle handpiece 8847KR.FG.016 (Komet), water cooled for 10 s.
3. Airborne particle abrasion with 30 µm silica-coated aluminum oxide particles (CoJet Sand; 3M EPSE), 2 bar, angle 45 degrees, until surface appeared matte.
4. Etching with 5% HF acid for 20 s.

Surfaces were cleaned with a combination spray of air and water for 20 s, and dried for 20 s with oil-free air. Group 4 was subsequently cleaned ultrasonically in distilled water for 5 min. For airborne particle abrasion standardization, a special holder was used to obtain the 45-degree angle surface conditioning (Fig 3).

Control groups

Control groups (5 and 6) consisted of six blocks of sintered IPS e.max CAD and IPS Empress CAD, respectively. Both groups were etched with 5% HF acid according to the manufacturer's recommendations (20 s and 60 s, respectively), rinsed with a combination spray of air and water for 20 s, and subsequently cleaned ultrasonically for 5 min in distilled water. After ultrasonic cleansing, the surface was dried with oil-free air for 20 s. Silane was applied as instructed per cement type.

Silanization

Following surface conditioning, silanization was carried out according to the manufacturers' recommendation per cement type. For Variolink II, Monobond Plus (Ivoclar Vivadent) was applied, rubbed for 60 s, and gently air dried using oil-free air for 5 s. For the experiment group 3, ESPE Sil (3M ESPE) was used because of its compatibility with the CoJet system. For RelyX Ultimate, Scotchbond Universal Adhesive was applied and rubbed for 20 s. The adhesive layer was air dried for a maximum of 5 s under an operating microscope at 10x magnification (OPMI pico Dental Microscope; Zeiss) until the adhesive layer stopped moving.

Cementation procedures

The cements were prepared according to the manufacturers' instructions. For Variolink II, Heliobond (Ivoclar Vivadent) was applied after silanization to promote the adaptation of the cement. Cement was injected into cylindrical transparent polyethylene molds with an internal diameter of 3 mm and a height of 5 mm. The cylinder was placed onto the substrate, and cement was packed against the surface with a composite modeling instrument. Light curing (Elipar 3M; 3M ESPE) was carried out for 20 s (> 1,000 mW/cm^2). Glycerine gel (Liquid Strip, transparent; Ivoclar Vivadent) was applied around the margins of all four cylinders, and the cement was light cured for another 20 s from three sites for each cylinder. Twelve cylinders were fabricated per study group.

Aging, testing procedure, and failure analysis

All specimens were thermocycled (Willytec) for 10,000 cycles, from 5°C to 55°C, with a dwell time of 30 s and a transfer time of 5 s.

The aged specimens were mounted in a universal jig (Zwick ROELL Z2.5 MA 18-1-3/7). Shear force was applied until failure occurred (crosshead speed: 1 mm/min; Fig 4). After debonding, the cylinders were checked for air bubbles.

Failure types were assessed at 25x magnification via a dental operation microscope (OPMI pico Dental Microscope) using a three-point scale: 1) cohesive failure in the substrate or mixed failure involving the substrate; 2) cohesive failure in the cement; and 3) failure at the adhesive interface. The size of the failure was also estimated to be smaller or larger than one third of the bonding area.

Fig 2 Completed specimen.

Fig 3 Standardization of airborne particle abrasion via a special apparatus.

Fig 4 Shear bond testing.

Table 2 Mean shear bond strength (SBS) in MPa (± SD)

Variolink II groups[1]	Mean ± SD	RelyX Ultimate groups[2]	Mean ± SD
1a. Lava Ultimate – Al_2O_3	18.68 ± 3.81^{b,c}	1b. Lava Ultimate – Al_2O_3	18.12 ±2.84^{d}
2a. Lava Ultimate – Bur	11.61 ± 3.39^{a}	2b. Lava Ultimate – Bur	12.34 ± 1.72^{b}
3a. Lava Ultimate – CoJet	17.09 ± 3.40^{b,c}	3b. Lava Ultimate – CoJet	11.54 ± 2.45^{b}
4a. Lava Ultimate – HF acid	15.21 ± 4. 29^{a,b}	4b. Lava Ultimate – HF acid	15.57 ± 2.31^{c}
5a. IPS e.max CAD	14.39 ± 2.80^{a,b}	5b. IPS e.max CAD	0.78 ± 0.89^{a}
6a. IPS Empress CAD	20.61 ± 4.10^{c}	6b. IPS Empress CAD	12.31 ± 1.87^{b}

The same superscript letters in the same column per cement type indicate no significant differences (α= 0.05).

[1] One-way ANOVA; [2] Kruskal-Wallis test

Statistical analysis

Specimens that contained air bubbles at the adhesive interface were excluded from the statistical analysis. Results were analyzed using IBM SPSS 20 statistical software. After checking the assumptions for normality and homogeneity of variance, a one-way analysis of variance (ANOVA) was conducted for the Variolink II group, with the bond strength as the dependent and the substrate as the independent variable. To test the hypotheses, the data were submitted to post hoc tests (Tukey's HSD). Data for the RelyX Ultimate group did not meet the assumptions of normality and a Kruskal-Wallis test was performed, followed by a stepwise step-down procedure. Failure analysis was performed using a chi-square test per cement type. A *P* value of < 0.05 was considered significant in the aforementioned tests. In addition, a Mann-Whitney U test was performed per substrate group to evaluate the effect of the cement (*P* value < 0.01 was considered significant).

Results

After thermocycling, 11 pretest failures occurred (seven in group 5b; and one each in groups 3a, 3b, 2a, and 2b). These were set to 0 in the statistical analysis. After exclusion of the specimens with air bubbles (one each in groups 3a, 3b, and 6) on the interface, 10 remaining specimens were subjected to shear testing for groups 1 to 4 and 6. For group 5, five specimens were tested (n = 115). To compare all groups, the missing values in group 5 were set to 0 (n = 5). This seemed justified due to the high rate of pretest failures in this group.

Bond strength

Mean bond strength values per cement group and significant differences are presented in Table 2 and Figure 3.

For the Variolink II specimens, there was a significant effect of the conditioning on the bond strength at the $P < 0.05$ level for the six groups, F (5.54) = 7.67, $P = 0.000017$, ω = 0.60.

Post hoc comparisons indicated that the mean bond strength of group 5a was not significantly different from the experiment groups (all *P* values ≥ 0.05). The mean bond strength of group 6a, however, was significantly higher than groups 2a ($P = 0.000$) and 4a ($P = 0.021$). There was no significant difference in mean bond strength between

Table 3 Failure modes

	Substrate/mixed		Cement		Adhesive	
	a	b	a	b	a	b
1. Lava Ultimate – Al_2O_3	6^6	10^{10}	4	0	0	0
2. Lava Ultimate – Bur	0	3^3	5	7^2	5	0
3. Lava Ultimate – CoJet	8^7	1^1	2	8^1	0	1
4. Lava Ultimate – HF acid	0	4^3	5^1	5^3	5	1
5. IPS e.max CAD	3	0	3	0	4	5
6. IPS Empress CAD	10^{10}	5^2	0	5^1	0	0

Superscript numbers represent the number of failures involving a surface area of more than one third of the bonding area.
a = Variolink II; b = RelyX Ultimate

group 6a and groups 1a and 3a (P = 0.847 and P = 0.282, respectively).

For the RelyX Ultimate specimens, the bond strength was significantly affected by the substrate, H (5) = 43.66, P = 0.000. To test the null hypothesis, a stepwise step-down analysis was performed. Compared to group 5b, the effect on bond strength was significantly greater for all experiment groups ($P < 0.05$). The effect on bond strength of group 6b was not significantly different (P = 0.79) from that of groups 2b and 3b. Groups 1b and 4b, however, did have a significantly greater effect on the mean bond strength than group 6b.

Bond strengths for groups 4a and 4b (U = 10.00, z = -3.02, P = 0.002, z = -0.68), groups 5a and 5b (U = 0.00, z = -3.808, P = 0.00, r = -0.85), and groups 6a and 6b (U = 1.00, z = -3.704, P = 0.00, r = -0.83) differed significantly, and the type of cement had a large effect on the bond strength.

Failure mode

Table 3 summarizes the frequency of the failure types. There was a significant association between the substrate and the mode of failure for the Variolink II ($\chi 2$ (10) = 39.68, P = 0.000) and the RelyX Ultimate ($\chi 2$ (10) = 69.51, P = 0.002) groups. No adhesive failures occurred in groups 1a, 1b, 6a, and 6b, with groups 1b and 6a presenting only mixed and substrate failures. In contrast, the specimens in group 5b failed exclusively at the adhesive interface (n = 10). All mixed failures showed a cohesive substrate failure larger than one third of the bonding area.

Discussion

The main objective of this study was to investigate the bond strength of two composite cements to multiphase composite resin after different surface treatments and compare this to two glass-ceramics. Within the limitations of this study, the null hypothesis cannot be rejected. Since cement type influenced the bond strength values, the second hypothesis can be rejected.

In this study, 21 of the 115 observations (18.26%) concerned adhesive failures. This high rate of cohesive failures is in accordance with previous studies.[8,11,12] However, the experiment groups cannot be compared on bond strength values alone. Cohesive failures can be regarded as an indication of the internal strength of the substrate or cement. The high rate of cohesive failures can be explained in two ways: 1) the bending moment that is created during shear testing leads to a higher rate of cohesive failures;[8,11,12] and 2) in relative terms: a high rate of cohesive failures might indicate that the bond of the adhesive interface exceeds the internal strength of the substrate or cement.[6,13] Although it is reasonable to assume that the bending moment explains part of the cohesive failures in the shear testing of restoration–cement specimens, this part may be smaller than in tooth substrate testing. After pooling shear bond results of 37 studies, Braga et al[14] report an incidence of 45% cohesive failures in the tooth substrate, whereas Brendeke and Ozcan[15] found 96% cohesive failures in a composite repair study. Hu et al[16] also showed a high incidence of cohesive failures for a CAD/CAM composite (70%) and feldspathic ceramic (68%). Other authors affirm these findings.[17] It is reasonable to assume that a cohesive failure in the substrate indicates a higher adhesive bond than a cohesive failure in the cement, since the internal strength of the CAD/CAM material is higher than that of a dual-curing cement. Since there was a high rate of cohesive failures, it was decided to include them in the bond strength analysis, as opposed to the advice of Scherrer et al[18] for tooth-substrate testing. It was, however, not possible in the current study design to determine exactly which failures were the result of a strong bond, the bending moment or a combination of both.

The majority of the failures in groups 1a, 1b, 3a, and 6a included the substrate and consisted of more than one third of the bonding area, suggesting that the adhesive bond was stronger than the internal strength of the substrate. In contrast, all failures in group 5b occurred at the adhesive interface. A possible explanation could be that no additional silanization step was performed. A silane is included in Scotchbond Universal Adhesive and the application time is 20 s. This timeframe may be too short for the silane to take maximum effect. Successful silanization significantly improves the bonding of cements to glass-ceramics and composite materials,[3,19] and also helps for a durable bond after aging.[20] In a shear bond strength (SBS) study of composite adhered to IPS e.max CAD,[21] the separate application of a silane (20 s) before Scotchbond Universal significantly improved bond strength (19.08 ± 3.0 MPa versus 40.47 ± 4.0 MPa). The higher value of 19.08 ± 3.0 MPa versus 0.78 ± 0.89 MPa might be explained by a more comprehensive surface standardization protocol, compared with the use of Sof-Lex discs in the present study.

These results suggest that the bond of both cements to Lava Ultimate is at least comparable to the glass-ceramics, and that airborne particle abrasion with aluminum oxide and silica-coated aluminum oxide shows promising results. This is in line with a recent surface conditioning protocol for nanocomposite indirect restorations.[22] In a microtensile study[23] of a self-adhesive resin to Lava Ultimate, no difference was found between the bond strength of 110 µm aluminum oxide particle abrasion compared with HF acid etching after aging (13.88 ± 3.47 versus 14.35 ± 2.56 MPa). In contrast, another microtensile test[24] found a significantly higher bond strength for aluminum oxide particle abrasion compared with HF acid etching for a cement to Lava Ultimate (16.0 ± 4.0 versus 10.0 ± 3.5 MPa). Kassotakis et al[25] compared different particle abrasion protocols and showed that both 50 µm aluminum oxide and silica-coated aluminum oxide resulted in a sufficient bond for Lava Ultimate (104.45 ± 18.76 versus 105.55 ± 11.88 MPa). However, they did not use a silane for the silica-coated group. It is possible that doing so would further improve the bond strength.

The effect of thermocycling on the bond strength of ceramic/composite cement specimens is contradictory. Adhesion of Variolink II to lithium disilicate was not significantly reduced.[3,26] In contrast, the bond of Variolink II to a leucite-reinforced ceramic was significantly affected after 6,000 cycles (5°C to 55°C; dwell time 30 s).[26] This was also true for the bond of composite cements to three different hybrid composites.[13]

Conclusions

Within the limitations of this study, the following conclusions can be drawn:

- The SBS of the composite cements to the multiphase composite resin that was treated by Al_2O_3 or silica-coated aluminum oxide particle abrasion is comparable to glass-ceramics.
- For the glass-ceramic groups, Variolink II seemed to perform with higher bond strength than RelyX Ultimate.

Clinical relevance

The SBS of the composite cements to the multiphase composite resin that was treated by Al_2O_3 or silica-coated aluminum oxide particle abrasion is comparable to glass-ceramics.

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