

Tensile Bond Strength of So-called Universal Primers and Universal Multimode Adhesives to Zirconia and Lithium Disilicate Ceramics

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Purpose: To test the bond strength and durability after artificial aging of so-called universal primers and universal multimode adhesives to lithium disilicate or zirconia ceramics.

Materials and Methods: A total of 240 ceramic plates, divided into two groups, were produced and conditioned: 120 acid-etched lithium disilicate plates (IPS e.max CAD) and 120 air-abraded zirconia plates (Zenostar T). Each group was divided into five subgroups ($n = 24$), and a universal restorative primer or multimode universal adhesive was used for each subgroup to bond plexiglas tubes filled with a composite resin to the ceramic plate. The specimens were stored in water at 37°C for 3 days without thermal cycling, or for 30 or 150 days with 7500 or 37,500 thermal cycles between 5°C and 55°C, respectively. All specimens then underwent tensile bond strength testing.

Results: Initially, all bonding systems exhibited high TBS, but some showed a significant reduction after 30 and 150 days of storage. After 3, 30, and 150 days, Monobond Plus, which contains silane and phosphate monomer, showed significantly higher bond strengths than the other universal primer and adhesive systems.

Conclusions: The bond strength to lithium disilicate and zirconia ceramic is significantly affected by the bonding system used. Using a separate primer containing silane and phosphate monomer provides more durable bonding than do silanes incorporated in universal multimode adhesives. Only one of five so-called universal primers and adhesives provided durable bonding to lithium disilicate and zirconia ceramic.

Keywords: lithium disilicate, zirconia, universal primers, universal adhesives, resin luting cement.

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All-ceramic materials have become increasingly popular for fabrication of dental restorations due to enhanced mechanical and optical properties. Many clinical studies have supported the use of all-ceramic restorations, showing long-term success when used as single crowns,³⁹ multiunit

fixed dental prostheses (FDPs),³⁶ inlays and onlays,^{16,44} laminate veneers,^{8,27} and resin-bonded FDPs.^{22,40} In general, the use of either zirconia or lithium disilicate ceramics for single crowns or FDPs is increasing and supported by high survival rates.^{36,39}

The cementation process of different types of dental restorations can affect both the longevity of the restorations and the final esthetic result.¹⁴ For zirconia ceramic, due to its outstanding mechanical properties, adhesive bonding may not be essential if a retentive preparation is possible. However, in some situations when retention is compromised or when resin-bonded FDPs are used, a durable bond is required.^{1,5,20} Sasse and Kern⁴⁰ reported very good mid-term clinical survival rates of single-retainer resin-bonded FDPs made from zirconia ceramic, making them a possible minimally invasive treatment option for the anterior region. Such restorations would not be feasible without a strong, durable bond between the zirconia ceramic and the tooth structure.

In contrast, most glass ceramics should be adhesively cemented, regardless of the type of restoration. When glass-ceramic restorations were bonded with a composite,

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they showed higher fracture strengths than when luted with zinc phosphate cement.¹⁷ In general, achieving a strong, durable resin bond to the restorations can increase the fracture strength of the restoration and tooth,¹⁹ increase retention,¹⁵ and inhibit microleakage.⁴² Thus, the decision of which luting agent to use is critical for the overall success of all-ceramic restorations.

The types of bonding systems and their chemical composition influence the success of chemical retention to different substrates and therefore also the bond durability.^{5,14,21} Consequently, the choice of primer or adhesive to provide chemical bonding depends on the restorative material to be bonded, meaning that the adhesives used for bonding silica-based ceramics differ from those used for zirconia ceramic. Silica-based ceramics require a silane coupling agent to bond to their surface;^{3,9,23} the silanol group of the silane bonds with the hydroxyl group of the silicate-ceramic surface.³⁴ While silanes do not promote durable chemical bonding to zirconia, zirconia ceramic requires a phosphate monomer such as methacryloxydecyl-dihydrogenphosphate (MDP).^{3,5,21,33} The strong chemical bond between MDP and zirconia is achieved through the chemical interaction between the phosphate ester group of the MDP and the hydroxyl groups in the zirconia surface.^{20,31,33} When applying a primer containing MDP and bisphenol-A-diglycidyl-methacrylate (bis-GMA), the methacrylate group of the MDP bonds to the resin matrix of the luting resin.²⁰ The use of MDP-containing primer can therefore enhance the bond strength of bis-GMA resin cements to zirconia.^{6,38} Resin cements and adhesives already containing MDP can be used directly without any additional primer to achieve durable bonding.^{21,22,33}

Several analytical methods have been used to investigate the chemical reaction between MDP and zirconia. These methods, including time-of-flight secondary ion mass spectrometry (TOF-SIMS), x-ray photoelectron spectroscopy (XPS), Fourier-transform infrared spectroscopy (FTIR), and thermodynamic calculations, successfully demonstrated that a true chemical bond of P-O-Zr exists between MDP and zirconia.^{11,28,35,47} Chen et al¹¹ reported that applying zirconia primers made the hydrophilic zirconia surface hydrophobic and significantly improved the bond strength of composite to zirconia. Using TOF-SIMS, they also found that a P-O-Zr chemical group exists.¹¹ Further, it was shown that using MDP-containing primers on yttria-stabilized zirconia (Y-TZP) promoted the chemical adhesion between the zirconia and composite through the formation of carboxylate and phosphate salts on the zirconia.³⁵

The process of choosing the best bonding system based on the restorative material used and the clinical situation seems to be rather complicated for many dentists in general practice. In a recently published survey, dentists in northern Germany were asked three times (2007, 2011, and 2015) about their adhesive cementation procedures for all-ceramic restorations.²⁶ It was found that a high number of dentists still employed inadequate bonding methods, ie, not those supported by the scientific literature.²⁶ Such surveys indicate that it might be beneficial to simplify the bonding process through the use of universal primers or

universal multimode adhesives that can be used on various if not all dental bonding substrates. Universal multimode adhesives can be also used on enamel and dentin in an etch-and-rinse or in a self-etch mode.³⁷

Recently, various dental manufacturers have launched so-called universal adhesives which are claimed to promote bonding to different types of dental substrates and can be used on enamel and dentin either in an etch-and-rinse or in a self-etch mode.^{37,46} However, some of these adhesive systems do not contain silanes or phosphate monomers, which makes their bonding ability to silicate or zirconia ceramics questionable. When adhesion-promoting monomers for different chemical substrates are mixed to obtain a universal primer or adhesive, potential interactions between the components might affect their bonding properties.³⁴ When the tensile bond strength of four universal primers/adhesives to lithium disilicate ceramic was recently investigated, universal adhesives that did not contain a silane did not provide long-term durable bonding.³⁴

The aim of this study was to test the bond strength of universal primers and multimode adhesives on lithium disilicate and zirconia ceramic. In addition, the bonding durability after artificial aging through water storage and thermal cycling was evaluated. The null hypothesis tested was that bond strength to both types of ceramics was not influenced by the so-called universal primers/adhesives used or artificial aging.

MATERIALS AND METHODS

Specimen Preparation

One hundred twenty square plates of lithium disilicate (IPS e.max CAD, Ivoclar Vivadent; Schaan, Liechtenstein) were used for the first bonding group (S). The plates had dimensions of 10 x 10 x 4 mm. Prior to bonding, the plates were ultrasonically cleaned with 99% isopropanol for 3 min and dried with oil-free air. All specimens were etched using 5% hydrofluoric acid (IPS Ceramic Etching Gel, Ivoclar Vivadent) for 20 s, rinsed with water for 20 s, and finally dried with oil-free air for 15 s.

For the second bonding group (Z), 120 zirconia plates (Zenostar T, Wieland; Pforzheim, Germany) having dimensions of 9 x 9 x 4.5 mm were prepared. The plates were polished under water irrigation with rotating silicon carbide papers down to 600 grit. The bonding surfaces were air abraded with 50- μ m Al₂O₃ particles at 0.1 MPa pressure and a distance of 10 mm. Then the plates were cleaned ultrasonically with 99% isopropanol for 3 min to remove any debris/Al₂O₃ particles and dried with oil-free air.

Each group was further divided into five subgroups (n = 24) according to the bonding system used (MPV: Monobond Plus/Variolink Esthetic DC; SRU: Scotchbond Universal/Rely X Ultimate; ONX: OptiBond XTR Adhesive/NX 3; ADL: All Bond Universal/Duo Link Universal; PBC: Calibra Silane, Prime and Bond NT/Calibra Esthetic). Table 1 summarizes the test groups and the method of application of each bonding system. The composition of all the materials used in the study with their batch numbers are listed in Table 2.

Table 1 Universal primers/adhesives used and their group codes

Group	Subgroup	Ceramic substrate	Bonding system	Manufacturer	Application*
MPV	S-MPV	Lithium disilicate ¹	Monobond Plus / Variolink Esthetic DC	Ivoclar Vivadent	Monobond Plus: A thin coat was applied with a microbrush to the pre-treated surfaces and left to react for 60 s. Then the excess was dispersed with a stream of oil-free air. Variolink Esthetic DC: Applied directly from the Automix syringe on the filled tubes. Disks were placed under a load of 750 g. The excess was removed and glycerin gel (Liquid Strip, Ivoclar Vivadent) was applied on the margins. Specimens were light cured for 20 s from two sides, then placed in light cure unit for 90 s.
	Z-MPV	Zirconia ²			
SRU	S-SRU	Lithium disilicate ¹	Scotchbond Universal / Rely X Ultimate	3M ESPE	Scotchbond Universal: A thin coat was applied to the pre-treated surfaces with a microbrush and left to react for 20 s, then the excess was dispersed with a stream of oil-free air for 5 s. Rely X Ultimate: Applied directly from the Automix syringe on the filled tubes. Disks were placed under a load of 750 g. The excess was removed and glycerin gel (Liquid Strip) was applied on the margins. Specimens were light cured for 20 s from two sides, then placed in the light-curing unit for 90 s.
	Z-SRU	Zirconia ²			
ONX	S-ONX	Lithium disilicate ¹	OptiBond XTR Adhesive / NX 3	Kerr	OptiBond XTR Adhesive: A thin coat was applied with a microbrush to the pre-treated surfaces, then the excess was dispersed with a gentle then a strong stream of oil-free air. NX 3: Applied directly from the Automix syringe on the filled tubes. Disks were placed under a load of 750 g. The excess was removed. Specimens were light cured for 20 s from two sides, then placed in the light-curing unit for 90 s.
	Z-ONX	Zirconia ²			
ADL	S-ADL	Lithium disilicate ¹	All Bond Universal / Duo Link Universal	Bisco	All Bond Universal: A thin coat was applied with a microbrush to the pre-treated surfaces, then the excess was dispersed with a stream of oil-free air. Light curing for 10 s. Duo Link Universal: Applied directly from the Automix syringe on the filled tubes. Disks were placed under a load of 750 g. The excess was removed. Specimens were light cured for 20 s from two sides, then placed in the light-curing unit for 90 s.
	Z-ADL	Zirconia ²			
PBC	S-PBC	Lithium disilicate ¹	Calibra Silane, Prime and Bond NT / Calibra Esthetic	Dentsply DeTrey	S-PBC: Calibra Silane: A thin coat was applied directly from the syringe and left to react for 60 s. Prime and Bond NT: Applied with a microbrush to the pre-treated surfaces and left to react for 20 s, then the excess was dispersed with a stream of oil-free air for 5 s. Light curing for 10 s. Calibra Esthetic: Equal amount of base and catalyst were mixed for 20 s and applied on the filled tubes. Disks were placed under a load of 750 g. The excess was removed. Specimens were light cured for 20 s from two sides, then placed in the light-curing unit for 90 s.
	Z-PBC	Zirconia ²			

*All materials were applied according to the manufacturers' instructions. Surface treatment of ceramic prior to bonding: ¹ Etching with 5% hydrofluoric acid for 20 s, rinsing with water for 20 s and drying with oil-free air for 15 s. ² Air abrasion with 50 µm Al₂O₃ particles at 0.1 MPa pressure, then ultrasonic cleaning with 99% isopropanol for 3 min and drying with oil-free air.

Tensile Bond Strength Testing

The same bonding procedure has been described in detail in other studies^{18,24,25} and was followed here to allow comparable results with previous data.

Briefly, plexiglas tubes of an inner diameter of 3.2 mm were filled with a dual-curing composite (Multicore Flow, Ivoclar Vivadent). Seven minutes after filling the tubes, they were bonded to the conditioned lithium disilicate or zirconia plates using one of the five bonding systems listed in Table 1. To ensure uniform bonding for all specimens, an alignment apparatus was used which provided a constant load of 750 g and ensured perpendicular alignment of the tube axis to the bonding surface.^{18,24,25}

After removing excess cement, an air-blocking gel (Liquid strip, Ivoclar Vivadent) was applied. Then, all specimens were light polymerized at a light intensity of 650 mW/cm² (Elipar 2500, 3M ESPE; Seefeld, Germany) from two opposing sides for 20 s each and finally cured in a light-curing unit (Dentacolor XS, Heraeus Kulzer; Hanau, Germany) for another 90 s to ensure optimal light polymerization. Afterwards, all the bonded specimens of all groups were left for 10 min at room temperature, and then stored in 37°C water. For each bonding system, a subgroup of 24 specimens was bonded. Each subgroup (n = 24) was then divided to three further groups of 8 specimens each, where the specimens (n = 8) were stored either for 3 days

Table 2 Primers, adhesives, and luting resins used, with their composition and batch numbers

Material	Batch No.	Composition	Manufacturer
Monobond Plus	T07775	Ethanol, methacrylated phosphoric acid ester, silane, sulfide methacrylate	Ivoclar Vivadent
Variolink Esthetic DC	S49410	Ytterbiumtrifluoride, urethandimethacrylate, 1,10-decandioldimethacrylate, α,α -dimethylbenzylhydroperoxide	
Scotchbond Universal	557776	MDP, dimethacrylate, HEMA, Vitrebond copolymer, fillers, ethanol, water, initiators, silane	
Rely X Ultimate	559158	Dimethacrylate, 1-benzyl-5-phenyl-barbic acid, calcium salt, 1,12-dodecandioldimethacrylate, sodiumtoluol-4-sulfinate, silica, calcium dihydroxide, 1-methylethylidene)bis(4,1-phenyleneoxy-3,1-propanediyl) bismethacrylate, titanium dioxide	3M ESPE
OptiBond XTR Adhesive	5203273	Hydroxyethylmethacrylate, ethanol, disodium-hexafluorosilicate, 4-methoxyphenol, barium glass	Kerr
NX 3	5110167	Hydroxyethylmethacrylate (HEMA), 4-methoxyphenol (MEHQ), 2-pyridylthiourea	
All Bond Universal	1400002817	Bis-GMA, ethanol, MDP, 2-hydroxyethyl methacrylate (HEMA)	Bisco
Duo Link Universal	1400003403	Bis-GMA, triethyleneglycol dimethacrylate, urethane dimethacrylate, glass filler	
Calibra Silane	140205	Silane, ethanol, water	
Prime and Bond NT	1406000338	Di- and trimethacrylate, amorphous silica oxide, PENTA (dipentaerythritolpentacrylate phosphoric acid monomer), cetylaminhydrofluoride, acetone, initiators	Dentsply DeTrey
Calibra Esthetic	140325	Bis-GMA, ethoxylated bisphenol a dimethacrylate, 2,2'-ethylenedioxydiethylidimethacrylate	

(initial time group) without thermal cycling (TC), 30 days with 7500 thermal cycles, or 150 days with 37,500 thermal cycles, ie, 7500 thermal cycles every 30 days. Thermocycling was performed between 5°C and 55°C with a dwell time of 3 s.

Following the different storage times, tensile bond strength (TBS) was tested at a crosshead speed of 2 mm/min in a universal testing machine (Zwick Z010, Zwick; Ulm, Germany) using a special test configuration. An alignment jig was attached to the load cell and crosshead by upper and lower chains.^{18,24,25} This resulted in self-alignment of the whole system, providing a moment-free axial force application.

Morphological Examination

All specimens were inspected using a light microscope (Wild Makroskop M420; Heerbrugg, Switzerland) at 30X magnification to calculate the fractured interfaces of the debonded ceramic and to determine failure modes (adhesive, cohesive, or mixed). In addition, randomly selected representative samples were sputter coated with a gold alloy and examined using a scanning electron microscope (SEM, XL 30 CP, Eindhoven; The Netherlands) operating with an acceleration voltage of 15 kV.

Statistical Analysis

The test results were statistically analyzed with the Kruskal-Wallis test at $p < 0.05$, followed by multiple paired comparisons of the means with the Wilcoxon rank sum test. Significance levels were adjusted with the Bonferroni-Holm correction for multiple testing.

RESULTS

TBS medians as well as means and standard deviations (in MPa) following different surface treatments and storage conditions of all test groups for lithium disilicate and zirconia ceramic are shown in Tables 3 and 4, respectively.

Tensile Bond Strength to Lithium Disilicate Ceramic

Groups S-MPV and S-PBC showed the highest initial TBS with medians of 45.0 and 40.5 MPa, respectively. Their initial TBS was significantly higher than those obtained for the other groups.

All groups exhibited a decrease in TBS during artificial aging, which was significant for all groups except group S-PBC. However, group S-ADL showed the most severe reduction in bond strength, with all specimens debonding spontaneously during aging.

Table 3 Tensile bond strength to lithium disilicate ceramic after different storage times

Group	Storage time					
	3 days / 0 TC		30 days / 7500 TC		150 days / 37,500 TC	
	Median	Mean \pm SD	Median	Mean \pm SD	Median	Mean \pm SD
S-MPV	45.0 ^{aA}	43.1 \pm 6.1	33.8 ^{bA}	35.2 \pm 7.2	25.4 ^{bA}	27.7 \pm 6.5
S-SRU	15.9 ^{aB}	15.6 \pm 1.5	12.6 ^{bB}	12.0 \pm 2.5	5.0 ^{cB}	5.4 \pm 1.0
S-ONX	12.4 ^{aB}	13.6 \pm 2.7	3.3 ^{bC}	4.0 \pm 2.4	2.1 ^{cC}	1.96 \pm 1.0
S-ADL	19.6 ^{aC}	20.1 \pm 2.2	0.0 ^{bD}	0.0 \pm 0.0	0.0 ^{bD}	0 \pm 0
S-PBC	40.5 ^{aA}	40.8 \pm 10.7	36.8 ^{aA}	33.2 \pm 6.5	36.7 ^{aA}	32.0 \pm 11.7

Medians, means and standard deviations in MPa (n = 8). TC: thermal cycles; SD: standard deviations. Within the same column, medians with the same upper case superscript letter are not statistically different ($p > 0.05$). Within the same row, medians with the same lower case superscript letter are not statistically different ($p > 0.05$). Global Kruskal-Wallis test followed by pairwise comparison using the Wilcoxon test modified by Bonferroni-Holm.

Table 4 Tensile bond strength to zirconia ceramic after different storage times

Group	Storage time					
	3 days / 0 TC		30 days / 7500 TC		150 days / 37,500 TC	
	Median	Mean \pm SD	Median	Mean \pm SD	Median	Mean \pm SD
Z-MPV	46.0 ^{aA}	42.7 \pm 10.4	45.3 ^{aA}	47.1 \pm 8.0	29.6 ^{bA}	31.4 \pm 8.7
Z-SRU	45.0 ^{aA}	44.7 \pm 7.2	38.3 ^{abA}	40.6 \pm 11.1	33.9 ^{bA}	32.5 \pm 3.6
Z-ONX	30.0 ^{aB}	28.8 \pm 6.4	16.5 ^{bB}	17.2 \pm 6.2	3.6 ^{cB}	4.1 \pm 2.7
Z-ADL	45.1 ^{aA}	44.8 \pm 6.6	35.4 ^{abA}	35.0 \pm 12.6	31.9 ^{bA}	31.7 \pm 10.4
Z-PBC	17.7 ^{aC}	19.9 \pm 6.9	13.6 ^{bB}	12.5 \pm 2.4	4.1 ^{cB}	4.0 \pm 0.7

Medians, means and standard deviations in MPa (n = 8). TC: thermal cycles; SD: standard deviations. Within the same column, medians with the same upper case superscript letter are not statistically different ($p > 0.05$). Within the same row, medians with the same lower case superscript letter are not statistically different ($p > 0.05$). Global Kruskal-Wallis test followed by pairwise comparison using the Wilcoxon test modified by Bonferroni-Holm.

Tensile Bond Strength to Zirconia Ceramic

Groups Z-ADL, Z-SRU, and Z-MPV showed the highest initial TBS with medians exceeding 45 MPa, which were significantly higher than those obtained for the other groups. However, the TBS of all groups decreased significantly during 150-day aging.

Results of Failure Mode Analysis

Figure 1 illustrates the type of failure modes in all groups. Groups S-MPV, S-PBC, Z-MPV, Z-SRU, and Z-ADL showed predominantly cohesive failure after 3, 30, and 150 days. Randomly selected images taken by SEM are presented in Fig 2.

DISCUSSION

The dimensions of the lithium disilicate plates were slightly larger than those of the zirconia as a result of the produc-

tion process and the sintering shrinkage. However, as the inner diameter of the Plexiglas tubes was uniform (3.2 mm), the bonding area was identical in all specimens of both groups.

Since adhesion and composites are affected intraorally by mechanical, chemical, and thermal factors, artificial aging should be performed in laboratory bond strength tests.^{25,30,32,45} Many studies^{4,33,45} revealed that bond strengths often decrease significantly after aging. In order to test the hydrolytic durability of bonding systems, adequate artificial aging should provide full water saturation of the bonded specimens, which may take several months of immersion in water.^{2,7,13,29,41}

When bonding to lithium disilicate, group S-MPV (Monobond Plus/Variolink Esthetic) showed the highest initial TBS (45.0 MPa) of all groups; although the bond strength decreased during aging, it was still acceptable after 150 days (25.4 MPa). Monobond Plus contains silane, which can ex-

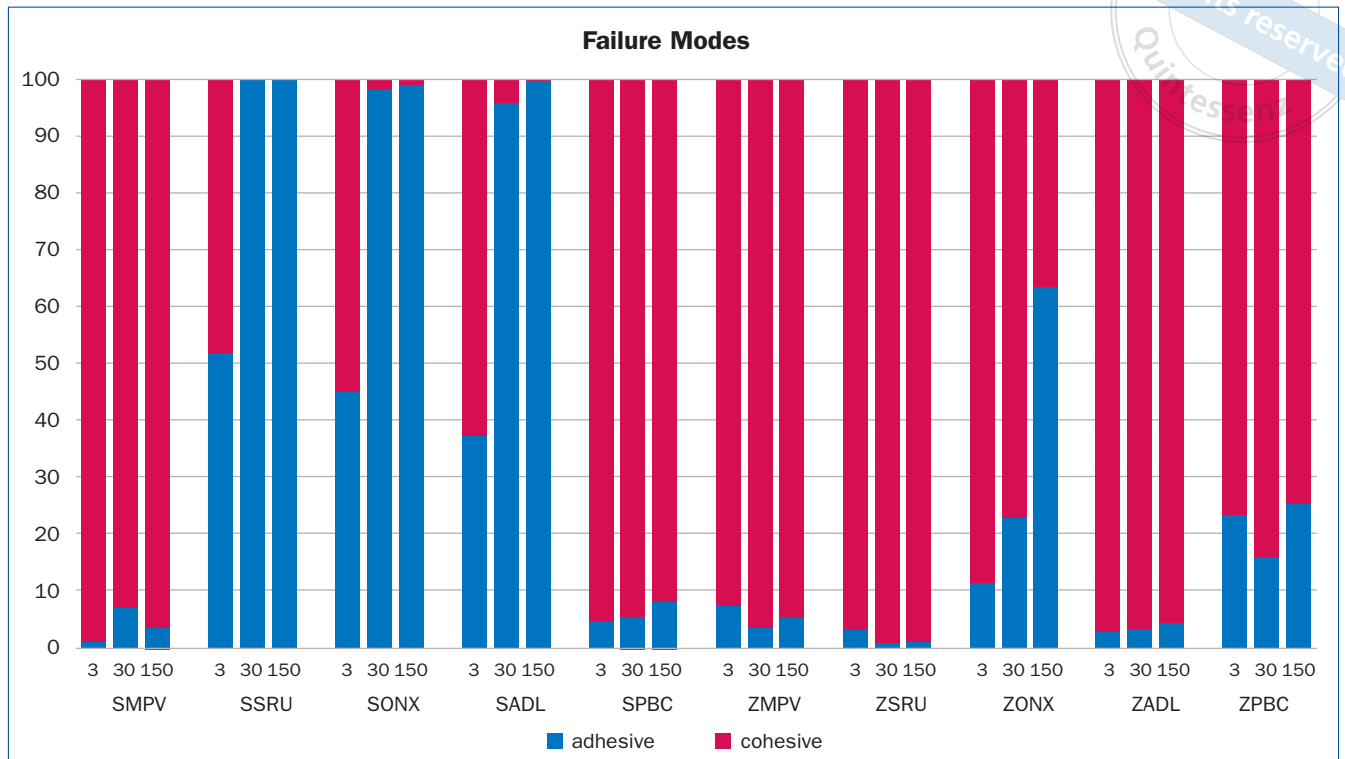


Fig 1 Failure modes after 3, 30, and 150 days (y-axis in %).

plain the high TBS. The second highest TBSs for lithium disilicate were obtained with S-PBC (Calibra Silane, Prime and Bond NT/Calibra Esthetic). The initial TBS for S-PBC (40.5 MPa) was lower than that of S-MPV, but after 150 days it was the most stable value (36.7 MPa). Although Scotchbond Universal contains silane, the TBS of group S-SRU (Scotchbond Universal/Rely X Ultimate) was low initially (15.9 MPa) and decreased to a very low level after 150 days (5.0 MPa). In contrast, the adhesives that do not contain silane showed very low TBS especially after artificial aging. In group S-ONX (OptiBond XTR/NX 3), the TBS was 12.4 MPa and 2.1 MPa after 3 and 150 days, respectively. Group S-ADL (All Bond Universal/Duo Link Universal) achieved an initial TBS of 19.6 MPa. However, this adhesive was not able to maintain bonding over long-term water storage with thermocycling and all specimens debonded spontaneously during storage, resulting in a TBS of 0 MPa.

The mode of failure for the groups SMPV and SPBC was mainly cohesive, which can be explained by the strong bond achieved in both groups. As an example, Fig 2a presents an SEM image of a group-SMPV specimen after 3 days, showing purely cohesive failure. For the other three groups, SSRU, SONX, and SADL, the mode of failure was divided between adhesive and cohesive after 3-day storage, whereas they showed predominantly adhesive failure after 30 and 150 days (Fig 2b), which is a result of the weak bond obtained by these three adhesives.

This reveals the importance of using silane coupling agent in bonding to silica-based ceramics. These results

correspond to a recent study which also showed that the presence of silane in the adhesive was critical to achieving a strong bond to lithium disilicate.³⁴ Despite containing a silane coupling agent, Scotchbond Universal showed low, unstable TBS. This might be due to 1. the presence of MDP and water, which create an acidic medium; and/or 2. incorporated resins which might affect silane bonding to lithium disilicate ceramic. The incorporated resins might hinder the evaporation of water, which is a byproduct of the condensation reaction between silane and glass-ceramic, as shown in a recent study.¹⁰ The stability of the TBS in group S-PBC also shows that using a separate silane primer produced more durable bonding than using a universal adhesive.

When bonding to zirconia, the universal primers/adhesives showed higher TBS than to lithium disilicate. Groups Z-MPV (Monobond Plus/Variolink Esthetic), Z-SRU (Scotchbond Universal/Rely X Ultimate), and Z-ADL (All Bond Universal/Duo Link Universal) showed high initial TBS that was also durable after 150 days of water storage with 37,500 thermal cycles. Moreover, these groups presented over 90% cohesive failures, which emphasizes the strong bond of the bonding systems in the three groups to zirconia. In contrast, group Z-PBC (Calibra Silane, Prime and Bond NT/Calibra Esthetic) showed significantly lower TBS followed by Z-ONX (OptiBond XTR/NX 3), with both groups showing an increased percentage of adhesive failures.

The differences in the TBS achieved with the five primers/adhesives might be related to the variations in the composition of the bonding systems and the phosphate-based func-

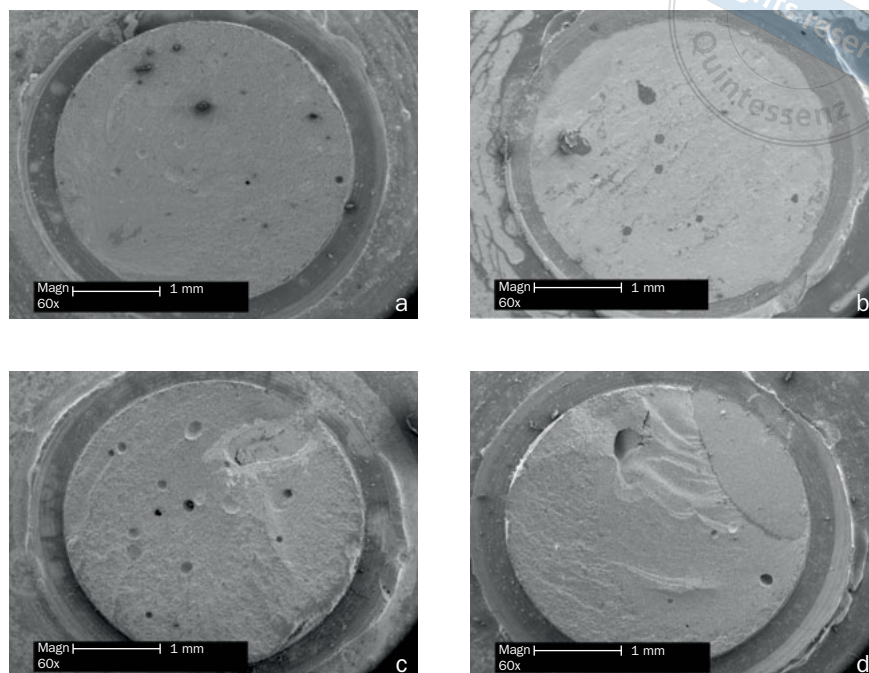


Fig 2 SEM images of representative samples. a) Group SMPV, 3-day series (purely cohesive failure). b) Group SONX, 150-day series. c) Group ZMPV 3-day series. d) Group ZONX, 30-day series.

tional monomers. Phosphate monomers (phosphate ester monomers) have been intensively studied and integrated in zirconia primers, as they have proven to provide high, durable bond strength between the zirconia and resin.^{11,46} The most well-documented and commonly used form of phosphate monomers is MDP. Another phosphate monomer is dipentacrythritol pentacrylate phosphate (PENTA), but it is not as well documented in the literature. In this study, the TBS of group Z-PBC was significantly lower than that obtained for the other groups. The low TBS of this group could be explained by the fact that in the Prime and Bond NT primer, the phosphate-containing reactive group is PENTA and not MDP. When compared with MDP-containing primers, Calibra Esthetics used with Prime and Bond NT showed reduced TBS to zirconia.⁴³ This might be related to the increased viscosity of the PENTA-containing primer as a result of the presence of five vinyl groups, which may hinder the ability of the primer to establish a strong chemical bond to zirconia.^{12,43} It could also be related to the fact that MDP contains a longer C-chain than PENTA, which could result in a more stable bond between the resin and the zirconia. However, when primers containing PENTA with different concentrations were tested, it was found that increasing the concentration of PENTA improves its binding affinity to zirconia.¹²

A recent study evaluated the use of three different one-bottle universal adhesives containing MDP, with and without the prior use of separate zirconia primer, on the bond strength of the resin cement to Y-TZP.⁴⁶ It was found that the use of the universal adhesives after alumina air abrasion showed comparable bond strength to tribochemical silica coating with silanization. Moreover, it was shown that pre-treatment of the zirconia with a separate zirconia primer

before the use of universal adhesives is not necessary.⁴⁶ These findings are in agreement with the results of the current study, as the use of specific universal adhesives without additional zirconia priming provided a strong, durable bond of the luting resin to zirconia.

In summary, Monobond Plus with Variolink Esthetic showed promising bond strengths to both lithium disilicate and zirconia. Calibra Silane, Prime and Bond NT/Calibra system achieved durable bonding to lithium disilicate but low bond strength to zirconia after aging. Conversely, Scotchbond Universal/Rely X Ultimate and All Bond Universal/Duo Link Universal mediated low bond strengths to lithium disilicate, but showed durable bonding to zirconia. Finally, OptiBond XTR/NX 3 did not provide durable bonding to either lithium disilicate or zirconia.

CONCLUSIONS

- The tensile bond strength to zirconia ceramic and lithium disilicate ceramic is significantly influenced by the primer/adhesive used.
- The presence of silane coupling agent is important for bonding to lithium disilicate ceramics.
- The effect of silane incorporated in a universal multi-mode adhesive might be limited.
- In general, so-called universal primers/adhesives achieve more durable bonding to zirconia than to lithium disilicate.
- Only the universal primer Monobond Plus in combination with Variolink Esthetic yielded adequate bond strength to both lithium disilicate and zirconia, which remained relatively stable after aging.

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Clinical relevance: So-called universal primers and multimode adhesives provide more durable bonding to zirconia than to lithium disilicate. The presence of silane coupling agent is important for bonding to lithium disilicate ceramic.