©Operative Dentistry, 2022, **47-1**, 107-120

In Vitro Performance of Different Universal Adhesive Systems on Several CAD/CAM Restorative Materials After Thermal Aging

LF Alegría-Acevedo • MF Gutiérrez • J Perdigão • A Núñez
L Méndez-Bauer • A Dávila-Sanchez • A Reis • AD Loguercio

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-08-30 via free access

Clinical Relevance

The performance of universal adhesives varied for different CAD/CAM materials. The results of this study may help clinicians elect the best adhesive system for each specific clinical case.

SUMMARY

Objective: To evaluate the microshear bond strength (mSBS) of 10 universal adhesive systems applied on five different CAD/CAM restorative materials, immediately and after thermal aging.

Methods and Materials: Five CAD/CAM materials

Luisa F Alegria-Acevedo, DDS, MS, PhD, Department of Restorative Dentistry, School of Dentistry, State University of Ponta Grossa, Paraná, Brazil

Mario Felipe Gutiérrez, DDS, MS, PhD, professor, Universidad de los Andes, Facultad de Odontología, Santiago, Chile, and University of Chile, Faculty of Dentistry, Institute for Research in Dental Sciences, Santiago, Chile

*Jorge Perdigão, DMD, MS, PhD, University of Minnesota, Department of Restorative Sciences, Minneapolis, MN, USA, and Universidade Católica Portuguesa, Faculty of Dental Medicine, Centre for Interdisciplinary Research in Health, Viseu, Portugal

Alejandra Núñez, DDS, MS, PhD student, Department of Restorative Dentistry, School of Dentistry, State University of Ponta Grossa, Paraná, Brazil, and Universidad San Francisco de Quito USFQ, Department of Restorative Dentistry and Dental Materials, School of Dentistry, Quito, Ecuador

Luján Méndez-Bauer, DDS, PhD, professor, Department of Restorative Dentistry, School of Dentistry, State University of Ponta Grossa, Paraná, Brazil, and Universidad Francisco Marroquín, Faculty of Dentistry, Department of Research, Cidad de Guatemala, Guatemala

Andrés Dávila-Sanchez, DDS, Ms, PhD, professor, Universidad San Francisco de Quito USFQ, Department of Restorative Dentistry and Dental Materials, School of Dentistry, Quito, Ecuador

Alessandra Reis, DDS, PhD, professor, Department of Restorative Dentistry, School of Dentistry, State University of Ponta Grossa, Paraná, Brazil

Alessandro D Loguercio, DDS, MS, PhD, professor, Department of Restorative Dentistry, School of Dentistry, State University of Ponta Grossa, Paraná, Brazil

*Corresponding author: 515 SE Delaware Street, 8-450 Moos Tower, Minneapolis, MN 55455 USA; e-mail: perdi001@umn.edu

http://doi.org/10.2341/20-203-L

were selected: 1) feldspathic glass ceramic (FeCe); 2) pre-polymerized reinforced resin composite (ReRC); 3) leucite-reinforced glass ceramic (LeGC); 4) lithium disilicate (LiDi); and 5) yttrium-stabilized zirconium dioxide (ZiDi). For each material, 15 blocks were cut into four rectangular sections (6 × 6 × 6 mm; n=60 per group) and processed as recommended by the respective manufacturer. For each indirect material, the following adhesive systems were applied according to the respective manufacturer's instructions: 1) AdheSE Universal [ADU]; 2) All-Bond Universal [ABU]; 3) Ambar Universal [AMB]; 4) Clearfil Universal Bond [CFU]; 5) Futurabond U [FBU]; 6) One Coat 7 Universal [OCU]; 7) Peak Universal Bond [PUB]; 8) Prime&Bond Elect [PBE]; 9) Scotchbond Universal Adhesive [SBU]; 10) Xeno Select [XEN, negative control]. After the application of the adhesive system, cylinder-shaped transparent matrices were filled with a dual-curing resin cement (NX3) and light cured. Specimens were tested in shear mode at 1.0 mm/min (mSBS), after 24 hours and 10,000 thermal cycles (TC). All data were submitted to statistical analysis (α=0.05).

Results: For FeCe, there was no significant decrease in mean mSBS for AMB, FBU, and SBU after TC when compared at 24 hours. For ReRC, AMB and SBU showed higher mean mSBS when compared to CFU and XEN, after 24 hours and TC. For LiDi, FBU and OCU showed higher mean mSBS when compared to CFU and XEN, after 24 hours and TC. For LeGC, AMB and PUB showed higher mean mSBS when compared to XEN, after 24 hours and TC. For ZiDi, OCU and SBU showed higher mean mSBS when compared to XEN, after 24 hours and TC. In addition, PBE and XEN showed the lowest mean mSBS after TC with higher percentage of bond strength reduction.

Conclusions: The mean mSBS among the different universal adhesives varied widely for each CAD/CAM material used. In addition, most universal adhesives underwent a statistically significant bond strength reduction after TC.

INTRODUCTION

Computer-aided design and manufacturing (CAD/CAM) has become an extremely important part of dentistry during the last two decades, as a result of the advances in intra-oral imaging and manufacturing technologies.[1-3] CAD/CAM technology has had a dramatic impact on several disciplines especially in prosthodontics and restorative dentistry, making tooth restoration easier and faster. In addition, it provides immediate chairside restorations without the risk of contamination with temporary cements and the need for provisional restoration.[4,5]

Within the current materials available for CAD/CAM restorations, glass-matrix ceramics and polycrystalline ceramics are the most widely used.[3] Nevertheless, new CAD/CAM ceramics known as resin-matrix hybrid materials have been recently introduced. These include pre-polymerized reinforced resin composite materials and polymer-infiltrated ceramic network (PICN) materials,[6,7] which have some mechanical properties similar to those of glass-matrix ceramics.[7,8]

The cementation procedure of a ceramic restoration is an important step in restorative dentistry for the long-term clinical success. This success depends on two interfaces, dental tissues with resin cement, but also resin cement with the specific restorative material.[9] The intaglio surface of resin-matrix ceramic restorations can be treated with sandblasting or with hydrofluoric acid, followed by a silane coupling agent.[10] On the other hand, sandblasting is not needed for glass-matrix ceramics,[11] as adhesion is achieved through a combination of micromechanical retention upon hydrofluoric acid etching and chemical adhesion provided by the silane coupling agent.[12] In the case of polycrystalline ceramics or glass-free oxide-based ceramics, hydrofluoric acid does not improve bond strengths, since these substrates do not contain a glass matrix.[13] Hydrofluoric acid may be effective on zirconia, but it would require longer application time, higher concentration, and/or a higher temperature, which makes it clinically unfeasible.[14,15]

In this context, several protocols have been advocated to achieve durable adhesion to polycrystalline ceramics. Some of the newest protocols include primers or silanes mixed with functional monomers, such as 10-methacryloyloxydecyl dihydrogenphosphate (MDP), in order to increase the potential for chemical interaction (ie, Monobond Plus [MB+], Ivoclar Vivadent, Shaan, Liechtenstein).[9] In addition, most universal adhesives also contain functional monomers such as MDP. The indications for these adhesive systems has been expanded to glass-matrix ceramics, polycrystalline ceramics, and metal alloys without the need for additional primers. However, a recent study showed that the presence of MDP in universal adhesives did not have a significant influence on the microshear bond strengths of several CAD/CAM materials in the immediate time.[12]

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-08-30 via free access

Another recent development in dental adhesion is the introduction of silane-containing universal adhesives (ie, Clearfil Universal Bond, Kuraray Noritake Dental, Tokyo, Japan) and Scotchbond Universal Adhesive (3M Oral Care, St Paul, MN, USA), in order to bond glass-rich (through silane) and glass-poor ceramics such as zirconia (through MDP). Although a universal adhesive with silane in the same solution is clinically useful, the effectiveness and long-term stability of silane contained in the universal adhesive is controversial.[16,17]

Accordingly, the effectiveness of universal adhesives with or without MDP, as well as universal adhesives containing silane on different CAD/CAM restorative materials, has not been extensively studied. In addition, there are few studies that investigated the effect of thermal aging on the interface between universal adhesives and different CAD/CAM indirect materials, and how substrates and universal adhesives behave after aging.

Thus, the aim of the present study was to evaluate the microshear bond strength of several universal adhesive systems on five different CAD/CAM indirect materials, after 24 hours of water storage or after 10,000 thermal cycles. The null hypotheses tested were: 1) a given universal adhesive would not result in different bond strengths between immediate time and after 10,000 thermo-cycles, in all five indirect materials; and 2) universal adhesives would not result in different bond strengths for each indirect substrate.

METHODS AND MATERIALS

Sample Size Calculation

To calculate the sample size, we considered the data of Scotchbond Universal (Scotchbond Universal Adhesive, 3M Oral Care), also known as Single Bond Universal in some countries. Means and standard deviations of bond strength of this adhesive system to zirconia as reported in the literature is 29.0 ± 1.1 MPa.[18] According to the free website, www.sealedenvelope.com, the minimum sample size required was six ceramic blocks in each group in order to detect a difference of 7 MPa among the tested groups, using a two-sided test with an α of 0.05 and a power of 80%.

Specimen Preparation

Five CAD/CAM materials were selected: 1) feldspathic glass ceramic (FeCe, Vitablocs RealLife, VITA Zahnfabrik; Bad Säckingen, Germany); 2) pre-polymerized reinforced resin composite (ReRC, Lava Ultimate CAD/CAM Restorative, 3M Oral Care); 3) leucite-reinforced glass ceramic (LeGc, IPS Empress CAD, Ivoclar Vivadent); 4) lithium disilicate (LiDi, IPS e.max CAD, Ivoclar Vivadent) and; 5) yttrium-stabilized zirconium dioxide (ZiDi, Ceramill Zi, Amann Girrbach, Koblach, Austria).

A total of 75 CAD/CAM blocks, 15 for each material, were used. For each material, the blocks (12 × 12 × 6 mm) were cut into four rectangular sections (6 × 6 × 6 mm; n=60 per group), using a diamond disk in slow speed (Isomet, Buehler, Lake Bluff, IL, USA) under water cooling.[12] When applicable, the specimens were fired following the crystallization program recommended by the respective manufacturer. ZiDi specimens were sintered according to the manufacturer's recommended protocol (Table 1).

Experimental Design

The specimens (n=60 for each indirect material) were randomly assigned (http://www.sealedenvelope.com) into 10 groups according to the adhesive system used: 1) Adhese Universal (ADU, Ivoclar Vivadent, also known as Tetric N-Bond Universal, Ivoclar Vivadent, Schaan, Liechtenstein); 2) All-Bond Universal (ABU, Bisco, Schaumburg, IL, USA); 3) Ambar Universal (AMB, FGM Prod Odont, Joinville, SC, Brazil); 4) Clearfil Universal Bond (CFU, Kuraray Noritake Dental); 5) Futurabond U (FBU, VOCO, Cuxhaven, Germany); 6) One Coat 7 Universal (OCU, Coltene, Altstätten, Switzerland); 7) Peak Universal Bond (PUB, Ultradent Products, South Jordan, UT, USA); 8) Prime&Bond Elect (PBE, Dentsply Sirona, Milford, DE, USA); 9) Scotchbond Universal Adhesive (SBU, 3M Oral Care, also known as Single Bond Universal in some countries); and 10) Xeno Select (XEN, Dentsply Sirona, also known as Prime&Bond One Select in some countries, Konstanz, Germany). XEN was used as a negative control, as the respective manufacturer does not recommend XEN for indirect restorations due to its low pH. The composition, application mode, and batch numbers are described in Table 2.

Microshear Bond Strength (μSBS)

The specimens were placed inside polyvinyl chloride (PVC) previously filled with acrylic resin (AutoClear, DentBras, Pirassununga, SP, Brazil), leaving a distance of 3 mm between the free surface of the ceramic and the top of the PVC ring. The description of the materials and their respective surface treatments are displayed in Table 1. Monobond Plus (MB+, silane + MDP solution, Ivoclar Vivadent) was used in all substrates prior to the application of universal adhesives, in order to standardize the experimental procedure. Then, the universal adhesives were applied according to the respective manufacturer's instructions (Table 2). A single operator performed all bonding procedures.

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-08-30 via free access

Table 1: *Materials Used, Composition, and Surface Treatment*

Material	Composition	Surface Treatment
Feldspathic glass ceramic (FeCe, Vitablocs RealLife, Vita)	SiO_2, Al_2O_3, K_2O, Na_2O, CaO, TiO_2	5% Hydrofluoric acid etching applied for 60 s (Vita Ceramics Etch; batch 42530). Rinsed with air-jet drying for 30 s. Ultrasonically clear with distilled water for 180 s. Silane Solution: Monobond Plus (Ivoclar Vivadent) applied with a brush and allowed to react for 60 seconds. Subsequently, the excess was dispersed with a strong stream of air to ensure the solvent evaporation.
Indirect resin composite (ReRC, Lava Ultimate CAD/CAM, 3M Oral Care)	Bis-GMA, UDMA, Bis-EMA, TEGDMA, 80wt% SiO_2 (20 nm) and ZrO_2 (4-11 nm) particles, aggregated ZrO_2/SiO_2 clusters	Sandblast with Al_2O_3, <50 μm (2 bar, until entire bonding surface appears matte. Ultrasonically clear with distilled water for 180 s. Remove sand with alcohol. Dry with oil-free air Silane Solution: Monobond Plus (Ivoclar Vivadent) applied with a brush and allowed to react for 60 s. Subsequently, the excess was dispersed with a strong stream of air to ensure the solvent evaporation.
Leucite-reinforced glass-ceramic (LeGC, IPS Empress CAD, Ivoclar Vivadent)	SiO_2, Al_2O_3, K_2O, Na_2O, other oxides, pigments	5% hydrofluoric acid etching for 60 s. Rinsed with water for 30 s. Ultrasonically cleaned with distilled water for 180 s. Silane Solution: Monobond Plus (Ivoclar Vivadent) applied with a brush and allowed to react for 60 s. Subsequently, the excess was dispersed with a strong stream of air to ensure solvent evaporation.
Lithium disilicate glass-ceramic (LiDi, IPS e.max CAD, Ivoclar Vivadent)	SiO_2, Li_2O, K_2O, P_2O_5, ZrO_2, ZnO, other oxides, coloring oxides	Crystallization in furnace (Programat P300, Ivoclar Vivadent) at 840 - 850ºC for 20 - 31 min. 5% hydrofluoric acid etching for 20 s.[a] Rinsed with water for 30 s. Dried with oil-free air for 30 s. Ultrasonically cleaned with distilled water for 180 s. Silane Solution: Monobond Plus (Ivoclar Vivadent) applied with a brush and allowed to react for 60 s. Subsequently, the excess was dispersed with a strong stream of air to ensure solvent evaporation.
Yttrium-stabilized zirconium dioxide (ZiDi, Ceramill Zi, Amann Girrbach AG)	$ZrO_2 + HfO_2 + Y_2O_3$: >99% Y_2O_3: 4.5 - 5.6 % HfO_2: < 0.5% Al_2O_3: <0.5%	Sintered in a furnace (Ceramill Therm, Amann Girrbach, Curitiba, PR, Brazil) using a universal program (8ºC/min from 200ºC to 1450ºC), 2 h at a fixed temperature of 1450ºC, and the correct cooling time. Sandblasted <50-μm Al_2O_3 particles (2.8 bar, 7s). Ultrasonically cleaned with distilled water for 180 s. The surface was thoroughly rinsed (5 ml) with ethyl alcohol (70%). Dry with oil-free air for 30 s. Silane Solution: Monobond Plus (Ivoclar Vivadent) applied with a brush and allowed to react for 60 s. Subsequently, the excess was dispersed with a strong stream of air to ensure solvent evaporation.

Abbreviations: Bis-GMA, bisphenol A diglycidylether methacrylate; UDMA, urethane dimethacrylate; Bis-EMA, ethoxylated bisphenol-A dimethacrylate; TEGDMA, triethylene glycol dimethacrylate.

[a]*Condac Porcelain Etch 5% (FGM Prod Odont Ltda, Joinville, SC, Brazil)*

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-08-30 via free access

Ten polyethylene transparent Tygon tubes (Tygon Medical Tubing Formulations 54-HL, Saint Gobain Performance Plastics, Akron, OH, USA) with an internal diameter of 0.8 mm and a height of 0.5 mm, which were positioned over the substrate. After that, in order to standardize the experimental procedure, an amine-free dual-curing resin cement (NX3, Kerr, Orange, CA, USA) was used to avoid the possible incompatibility of universal adhesives when in contact with dual-cured resin cements. This dual-curing resin cement was carefully packed inside each tube, and a clear Mylar matrix strip was placed over the filled Tygon tube and pressed gently into place. The resin cement was light cured for 20 seconds using an LED light-curing unit set at 1,200 mW/cm^2 (Radii-cal, SDI Limited, Bayswater, Victoria, Australia). A radiometer (Demetron L.E.D. Radiometer, Kerr Sybron Dental Specialties, Middleton, WI, USA) was used to check the light intensity after every five specimens. These procedures were carried out under magnifying loupes.

After storage of the specimens in distilled water for 24 hours at 37°C the Tygon tubes were carefully removed with a blade, exposing the cement cylinders; each specimen was examined under a stereomicroscope at 10× magnification, the bonded cylinder was discarded if there was evidence of porosities or gaps at the interface. Half of the specimens were tested immediately, and the other half tested after 10,000 thermal cycles in distilled water between water baths held at 5 and 55 °C, with a dwell time of 1 minute.[19]

The specimens were attached to a shear-testing jig (Odeme Biotechnology; Joaçaba, SC, Brazil) and tested in a universal testing machine (Kratos IKCL 3-USB, Kratos Equipamentos Industriais; Cotia, São Paulo, Brazil). Each specimen was mounted in the universal testing machine and a thin orthodontic wire (0.2 mm diameter) was looped around the base of each composite cylinder. The orthodontic wire contacted the composite cement cylinder along half of its circumference. The setup was kept aligned (resin/substrate interface, the wire loop, and the center of the load cell) to ensure the correct orientation of the shear forces.[20] The crosshead speed was set at 1 mm/min until failure.

The μSBS values (MPa) were calculated by dividing the load at failure by the surface area (mm^2). After testing, the specimens were examined under an optical microscope (SZH-131, Olympus; Tokyo, Japan) at 100× magnification to define the location of the bond failure. The failure mode was classified as cohesive failure exclusively in resin cement (CR), cohesive failure exclusively in the ceramic or CAD/CAM indirect resin composite (CC), adhesive/mixed (A/M) failure at the cement/ceramic interface, which included cohesive failure of the ceramic and/or indirect resin composite, resin cement, and adhesive material. Also, the premature failures were recorded.

Statistical Analysis

The data were first analyzed using the Kolmogorov-Smirnov test to assess whether the data followed a normal distribution, and Bartlett's test for equality of variances to determine if the assumption of equal variances was valid. After confirming the normality of the data distribution and the equality of the variances, the μSBS (MPa) data were subjected to appropriate statistical analysis. The μSBS of all specimens from the same individual indirect specimens were averaged for statistical purposes. Two-way ANOVA was used to analyze the μSBS data for each indirect material (adhesive vs storage time). After that, the Tukey's post-hoc test was used at $\alpha = 0.05$.

RESULTS

For each indirect material, 30 cylinders were tested at each evaluation time. In the immediate time, the majority of specimens for all indirect materials showed adhesive/mixed failures (Table 3). For some materials there were some cohesive fractures of cement or in the indirect material (Table 3). However, after thermocycling, specimens showed adhesive/mixed failures for all indirect materials, with only a few cohesive fractures of cement or indirect material (Table 3). For ZiDi it is worth mentioning that 16.7% premature failures occurred with CFU, 40% with PBE, and 60% with XEN after thermocycling (Table 3).

For FeCe, the interaction between main factors were statistically significant ($p<0.0001$; Table 4). The application of ADU, ABU, AMB, and PUB resulted in statistically significant higher mean μSBS when compared with those of CFU, OCU, and XEN in the immediate time ($p<0.0001$; Table 4). However, after thermocycling, only AMB, FBU, and SBU showed higher mean μSBS when compared to the remainder of the universal adhesives tested (ADU, ABU, CFU, OCU, PUB, PBE, and XEN; $p<0.0001$; Table 4). XEN showed the lowest mean μSBS after thermocycling; its reduction of more than 40% of bond strength similar only to PBE ($p<0.0001$; Table 4). There was no significant decrease in mean μSBS for AMB, FBU, and SBU after thermocycling when compared to the immediate time ($p>0.05$; Table 4).

For ReRC, the interaction between main factors was statistically significant ($p<0.0004$; Table 5). In the immediate time, statistically higher mean μSBS were observed when ABU, AMB, FBU and SBU were compared with those of ADU, CFU, OCU, PUB and

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-08-30 via free access

Table 2: *Adhesive System (Batch Number), Composition, and Application Mode of the Adhesive According the Manufacturer's Instructions*

Adhesive (Batch Number)	Composition	Application Mode[a]
Adhese Universal, ADU, Ivoclar Vivadent (UO2709) pH = 2.5 to 3.0	HEMA, 10-MDP, bis-GMA, MCAP, D3MA, ethanol, water, highly dispersed silicon dioxide and CQ	1. Apply one coat of adhesive for 20 s. 2. Gently air thin for 5 s. 3. Light cure for 10 s at 1,200 mW/cm^2.
All-Bond Universal, ABU, Bisco (1500002859) pH = 3.1 to 3.2	HEMA, 10-MDP, bis-GMA, ethanol, water, initiators	1. Apply one coat of adhesive. 2. Evaporate excess solvent by thoroughly air drying with an air syringe for at least 10 s until no visible movement of the material is observed. The surface should have a uniform, glossy appearance. 3. Light cure for 10 s at 1,200 mW/cm^2.
Ambar Universal, AMB, FGM (210415) pH = 2.6 to 3.0	Methacrylate monomers (UDMA and 10-MDP), photo-initiators, co-initiators, stabilizers, inert silica nanoparticles, ethanol, water	1. Apply two coats vigorously by rubbing the adhesive for 20 s (10 s each). 2. Gently air dry for 10 s to evaporate the solvent. 3. Light cure for 10 s.
Clearfil Universal Bond, CFU, Kuraray Noritake Dental (CR0002) pH = 2.3	Bis-GMA, HEMA, ethanol, 10-MDP, hydrophilic aliphatic dimethacrylate, colloidal silica, CQ, silane coupling agent, accelerators, initiators, water	1. Apply bond and leave it in place for 5 s. 2. Dry by blowing with a mild air stream for 5 s until the mixture does not move. 3. Light cure for 10 s at 1,200 mW/cm^2.
Futurabond U, FBU, Voco (1346518) pH = 2.3	HEMA, bis-GMA, HEDMA, 10-MDP, UDMA, catalyst, silica nanoparticles, ethanol, water	1. Apply the adhesive with microbrush for 20 s. 2. Direct a gentle stream of air over the liquid for about 5 s until it no longer moves and the solvent is evaporated completely. 3. Light cure for 10 s at 1,200 mW/cm^2.
One Coat 7 Universal, OCU, Coltene (F96836) pH = 2.8	HEMA, hydroxypropylmethacrylate, MMA-modified polyacrylyic acid, UDMA, amorphous silica, MDP, ethanol, water	1. Rub with a disposable brush for 20 s. 2. Dry gently with oil-free compressed air for 5 s. 3. Light cure for 10 s at 1,200 mW/cm^2.
Peak Universal Bond, PUB, Ultradent Products (BB7D7) pH = 2.0	Bis-GMA, ethyl alcohol, 0.2% chlorhexidine di(acetate), methacrylic acid, HEMA, 7.5% filler.	1. Apply a puddle coat of Peak Universal Bond with gentle agitation for 10 s. 2. Gently dry with clean air for at least 5 s; surface should have a uniform, glossy appearance. 3. Light cure for 10 s at 1,200 mW/cm^2.
Prime&Bond Elect, PBE, Dentsply Sirona (130811) pH = 2.5	Mono-, di- and trimethacrylate resins, PENTA diketone, organic phosphine oxide, stabilizers, cetylamine hydrofluoride, acetone, water.	1. Apply generous amount of adhesive thoroughly to all surface and leave undisturbed for 20 s. 2. Gently dry with clean air for at least 5 s. Surface should have uniform, glossy appearance. 3. Light cure for 10 s at 1,200 mW/cm^2.

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-08-30 via free access

Table 2: *Adhesive System (Batch Number), Composition, and Application Mode of the Adhesive According the Manufacturer's Instructions (cont.)*

Scotchbond Universal, SBU, 3M Oral Care (523652) pH = 2.7	10-MDP, dimethacrylate resins, HEMA, methacrylate-modified polyalkenoic acid copolymer, nanofiller, ethanol, water, initiators, silane.	1. Apply the adhesive and leave undisturbed for 20 s. 2. Direct a gentle stream of air over the liquid for about 5 s until there is no longer any movement and the solvent is evaporated completely. 3. Light cure for 10 s at 1,200 mW/cm^2.
Xeno Select, XEN, Denstply Sirona (1401001210) pH = 1.6	Bifunctional acrylates, acidic acrylate, functionalized phosphoric acid ester (ethyl 2-[5-dihydrogen phosphoryl-5,2-dioxapentyl]acrylate), water, tert-butyl alcohol, initiator (camphorquinone), co-initiator (DMABN), stabilizer.	1. Apply a generous amount of adhesive to thoroughly wet all surfaces and agitate for 20 s. 2. Gently dry with clean air for a least 5 s. Surface should have a uniform, glossy appearance. 3. Light cure for 10 s at 1,200 mW/cm^2.
NX3 Nexus, Kerr (6108657)	Bis-GMA, UDMA, EBPADMA, TEGDMA, HEMA, activators, stabilizers, ytterbium fluoride, fumed silica, barium aluminoborosilicate.	1. Carefully pack resin cement inside each tube and place a clear Mylar matrix strip over the filled Tygon tube and gently pressed into place. 2. Light cure for 20 s at 1,200 mW/cm^2.
Monobond Plus, MB+ Ivoclar Vivadent (S31153) pH = 3.1	Ethanol, 3-trimethoxysilypropyl methacrylate, methacrylated phosphoric acid ester (10-MDP) and disulfide acrylate.	1. Apply with a brush and allow to react for 60 s. 2. Blow with a strong stream of air to ensure solvent evaporation.

Abbreviations: 10-MDP, methacryloyloxydecyl dihydrogen phosphate; bis-GMA, bisphenol glycidyl methacrylate; MCAP, methacrylated carboxylic acid polymer; CQ, camphorquinone; D3MA, decanediol dimethacrylate; DMABN, 4- (dimethylamino)benzonitrile; HEDMA, hexamethylene dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; PENTA, dipentaerythritol penta acrylate monophosphate; UDMA, urethanedimethacrylate; EBPADM, (ethoxylated bisphenol A-dimethacrylate); TEGDMA, (trieth-ylene glycol dimethacrylate).

[a] *The intensity of light curing was standardized for all materials.*

XEN (p<0.0004; Table 5). After thermocycling, AMB, OCU, and SBU resulted in statistically higher mean μSBS when compared to those of CFU and XEN, which showed 49.4% and 56.1% of bond strength reduction, respectively, after thermocycling (p<0.0003; Table 5).

When the universal adhesives were evaluated on the LiDi substrate, the interaction between main factors was statistically significant (p<0.0003; Table 5). In the immediate time, statistically higher mean μSBS were found for AMB, FBU, OCU, and PUB compared to those of CFU, PBE, and XEN (p<0.0003; Table 5). After thermocycling, FBU, OCU, and SBU showed higher mean μSBS when compared to CFU and XEN (p<0.0003; Table 5). CFU and XEN showed the highest percentage of μSBS reduction with 45.5% and 48.3%, respectively (Table 5).

For LeGC, the interaction between main factors was statistically significant (p<0.0004; Table 6). Statistically higher mean μSBS values in the immediate time were observed only for AMB, PUB, and SBU compared to those of OCU and XEN (p<0.0004; Table 6). After thermocycling, AMB, FBU, and PUB showed higher mean μSBS when compared to XEN, which showed the lowest mean μSBS after thermocycling (p<0.0004; Table 6).

The interaction between main factors was statistically significant (p<0.0001; Table 6) when the universal adhesives were evaluated in the yttrium-stabilized zirconium dioxide (ZiDi). In the immediate time, the highest mean μSBS values were measured for ADU, ABU, OCU, and SBU, which were statistically higher compared to those of XEN (p<0.00001; Table 6). After thermocycling, AMB, OCU, and SBU showed higher mean μSBS when compared to those of ADU, ABU, CFU, FBU, PUB, PBE, and XEN. However, two universal adhesives (PBE and XEN) showed the

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-08-30 via free access

Table 3: *Number (%) of Specimens According to Fracture Mode*

Immediate															
Adhesive System	**Feldspathic Glass Ceramic (FeCe) -Vita Mark II**			**Indirect Resin Composite (InRC) -Lava Ultimate CAD/CAM**			**Leucite-reinforced Glass-ceramic (LeGC) - IPS Empress CAD**			**Lithium Disilicate Glass-ceramic (LiDi) -IPS e.max CAD**			**Yttrium-stabilized Zirconium Dioxide (ZiDi)- Ceramill Zi**		
	A/M	**CR**	**CC**	**A/M**	**CR**	**CC**	**A/M**	**CR**	**CC**	**A/M**	**CR**	**CC**	**A/M**	**CR**	**CC**
ADU	30 (100)	0 (0)	0 (0)	29 (97)	0 (0)	1 (3)	30 (100)	0 (0)	0 (0)	30 (100)	0 (0)	0 (0)	30 (100)	0 (0)	0 (0)
ABU	16 (53)	2 (7)	12 (40)	21 (70)	0 (0)	9 (30)	20 (67)	0 (0)	10 (33)	28 (93)	2 (7)	0 (0)	28 (93)	2 (7)	0 (0)
AMB	18 (60)	2 (7)	10 (33)	20 (67)	1 (3)	9 (30)	22 (74)	0 (0)	8 (26)	30 (100)	0 (0)	0 (0)	28 (93)	2 (7)	0 (0)
CFU	27 (90)	0 (0)	3 (10)	25 (83)	0 (0)	5 (17)	17 (57)	1 (3)	12 (40)	27 (90)	3 (10)	0 (0)	28 (93)	2 (7)	0 (0)
FBU	18 (60)	3 (10)	9 (30)	20 (67)	0 (0)	10 (33)	20 (67)	0 (0)	10 (33)	30 (100)	0 (0)	0 (0)	30 (100)	0 (0)	0 (0)
OCU	27 (90)	0 (0)	3 (10)	26 (87)	1 (3)	3 (10)	21 (70)	2 (7)	7 (23)	30 (100)	0 (0)	0 (0)	30 (100)	0 (0)	0 (0)
PUB	21 (70)	3 (10)	6 (20)	22 (73)	0 (0)	8 (27)	20 (67)	2 (7)	8 (26)	28 (93)	2 (7)	0 (0)	30 (100)	0 (0)	0 (0)
PBE	20 (67)	0 (0)	10 (33)	26 (87)	0 (0)	4 (13)	21 (70)	0 (0)	9 (30)	26 (87)	4 (13)	0 (0)	29 (97)	1 (3)	0 (0)
SBU	24 (80)	0 (0)	6 (20)	19 (63)	0 (0)	11 (37)	22 (74)	0 (0)	8 (26)	26 (87)	4 (13)	0 (0)	28 (93)	2 (7)	0 (0)
XEN	21 (70)	0 (0)	9 (30)	26 (87)	0 (0)	4 (13)	22 (74)	1 (3)	7 (23)	30 (100)	0 (0)	0 (0)	30 (100)	0 (0)	0 (0)
After Thermocycling															
ADU	29 (97)	0 (0)	1 (3)	30 (100)	0 (0)	0 (0)	30 (100)	0 (0)	0 (0)	30 (100)	0 (0)	0 (0)	30 (100)	0 (0)	0 (0)
ABU	26 (87)	0 (0)	4 (13)	28 (93)	0 (0)	2 (7)	26 (87)	0 (0)	4 (13)	30 (100)	0 (0)	0 (0)	30 (100)	0 (0)	0 (0)
AMB	25 (83)	0 (0)	5 (17)	29 (97)	0 (0)	1 (3)	27 (90)	0 (0)	3 (10)	29 (97)	0 (0)	1 (3)	30 (100)	0 (0)	0 (0)
CFU	28 (93)	0 (0)	2 (7)	30 (100)	0 (0)	0 (0)	25 (83)	0 (0)	5 (17)	30 (100)	0 (0)	0 (0)	25 (83)	0 (0)	0 (0)
FBU	24 (80)	1 (3)	5 (17)	27 (90)	0 (0)	3 (10)	26 (87)	0 (0)	4 (13)	30 (100)	0 (0)	0 (0)	30 (100)	0 (0)	0 (0)
OCU	29 (97)	0 (0)	1 (3)	30 (100)	0 (0)	0 (0)	27 (90)	0 (0)	3 (10)	30 (100)	0 (0)	0 (0)	30 (100)	0 (0)	0 (0)
PUB	24 (80)	0 (0)	6 (20)	28 (93)	0 (0)	2 (7)	29 (97)	0 (0)	1 (3)	30 (100)	0 (0)	0 (0)	30 (100)	0 (0)	0 (0)
PBE	30 (100)	0 (0)	0 (0)	30 (100)	0 (0)	0 (0)	30 (100)	0 (0)	0 (0)	30 (100)	0 (0)	0 (0)	18 (60)	0 (0)	0 (0)
SBU	28 (93)	0 (0)	2 (7)	28 (93)	0 (0)	2 (7)	26 (87)	0 (0)	4 (13)	29 (97)	1 (3)	0 (0)	30 (100)	0 (0)	0 (0)
XEN	30 (100)	0 (0)	0 (0)	30 (100)	0 (0)	0 (0)	30 (100)	0 (0)	0 (0)	30 (100)	0 (0)	0 (0)	12 (40)	0 (0)	0 (0)

Abbreviations: ADU, Adhese Universal; ABU, All-Bond Universal; A/M, adhesive/mixed fracture mode; AMB, Ambar Universal; CC, cohesive in indirect restorative material; CFU, Clearfil Universal Bond; CR, cohesive in resin cement; FBU, Futurabond U; OCU, One Coat 7 Universal; PUB, Peak Universal Bond; PBE, Prime & Bond Elect; SBU, Scotchbond Universal; XEN, Xeno Select.

lowest mean μSBS after thermocycling with higher percentage of bond strength reduction (84.6% and 65.5%, respectively; $p<0.00001$; Table 6).

DISCUSSION

The main objective of the present study was to evaluate whether the mean μSBS of different universal adhesives would decrease after thermocycling when applied on materials used for indirect restorations. Although several universal adhesives are indicated for luting procedures, there are no published studies to our knowledge that have evaluated several universal adhesives applied on a wide number of materials used for indirect restorations.

A recent meta-analysis of universal adhesives used for indirect procedures showed that the majority of the studies evaluated up to four universal adhesives (ABU, ADU, CFU, and SBU) applied to one or two indirect substrates.[21] However, the majority of studies evaluated universal adhesives against lithium disilicate glass ceramics[17,22,23] or yttrium-stabilized zirconium dioxide.[17,18,24] Only a few studies have evaluated the bond strength to glass ceramics, leucite-reinforced[25] or indirect composite.[26] Therefore, it is relevant to test the longevity of several different universal adhesives *in vitro* applied on the recent CAD/CAM materials for indirect restorations.

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-08-30 via free access

Table 4: *Mean ± Standard Deviation of Microshear Bond Strength (μSBS) of Universal Adhesives Bonded to Feldspathic Glass Ceramic*[a]

Adhesive System	Feldspathic Glass Ceramic (FeCe)		
	Immediate	After Thermocycling	Bond Strength Reduction (%)
ADU	32.2 ± 1.5 A	21.1 ± 3.3 D	34.4
ABU	29.3 ± 1.6 A	21.9 ± 2.6 D	25.3
AMB	28.5 ± 1.3 AB	25.8 ± 4.1 B	9.5
CFU	23.2 ± 1.9 C	16.7 ± 2.8 D	28.0
FBU	27.1 ± 1.8 B	24.2 ± 2.6 BC	10.7
OCU	24.2 ± 1.3 C	20.8 ± 3.2 D	14.1
PUB	28.2 ± 1.1 AB	21.4 ± 2.9 D	24.1
PBE	25.2 ± 1.6 BC	17.5 ± 1.0 DE	30.6
SBU	26.3 ± 1.3 BC	22.3 ± 3.1 C	15.2
XEN	22.5 ± 1.6 C	13.4 ± 0.8 E	40.4

Abbreviations: ADU, Adhese Universal; ABU, All-Bond Universal; AMB, Ambar Universal; CFU, Clearfil Universal Bond; FBU, Futurabond U; OCU, One Coat 7 Universal; PUB, Peak Universal Bond; PBE, Prime & Bond Elect; SBU, Scotchbond Universal; XEN, Xeno Select.
[a]*Different letters indicate statistically significant differences (two-way ANOVA, Tukey's test,* $p<0.05$*).*

Table 5: *Mean ± Standard Deviation of Microshear Bond Strength (μSBS) of Universal Adhesives Bonded to Pre-polymerized Reinforced Resin Composite and Lithium Disilicate Glass-ceramic*[a]

Adhesive System	Pre-polymerized Reinforced Resin Composite (ReRC)			Lithium Disilicate Glass-ceramic (LiDi)		
	Immediate	After Thermocycling	Bond Strength Reduction (%)	Immediate	After Thermocycling	Bond Strength Reduction (%)
ADU	25.3 ± 1.5 B	16.8 ± 1.8 DE	33.6	25.5 ± 1.4 b	16.8 ± 2.9 d	34.1
ABU	30.4 ± 1.7 A	17.2 ± 2.8 DE	43.3	25.9 ± 1.5 b	16.2 ± 1.5 d	37.5
AMB	28.1 ± 1.9 A	18.2 ± 2.8 D	35.2	28.2 ± 1.2 ab	16.4 ± 1.8 d	41.8
CFU	24.3 ± 1.1 B	12.3 ± 1.3 E	49.4	23.1 ± 1.8 c	12.6 ± 2.2 e	45.5
FBU	27.0 ± 1.7 A	16.6 ± 2.0 DE	38.5	27.2 ± 1.2 ab	19.6 ± 2.7 c	28.0
OCU	24.2 ± 1.1 B	18.9 ± 2.2 D	21.9	29.0 ± 1.4 a	18.2 ± 2.6 cd	37.3
PUB	24.1 ± 1.2 B	17.4 ± 2.7 DE	27.8	29.0 ± 1.2 a	17.7 ± 1.7 d	39.0
PBE	26.2 ± 1.6 AB	15.2 ± 2.3 DE	42.0	22.2 ± 1.7 c	15.2 ± 2.0 de	40.2
SBU	30.1 ± 1.9 A	19.0 ± 2.3 D	36.8	25.4 ± 1.4 b	19.7 ± 2.6 c	32.5
XEN	24.6 ± 1.2 B	10.8 ± 1.4 F	56.1	17.2 ± 1.3 cd	8.9 ± 2.5 f	48.3

[a]*Different uppercase (ReRC) and lowercase (LiDi) letters indicate statistically significant differences for each restorative material (two-way ANOVA, Tukey's test,* $p<0.05$*).*

Despite some exceptions (AMB, FBU, and SBU bonding to FeCe), all of the universal adhesives showed a reduction in mean μSBS for all indirect restorative materials when submitted to thermocycling. The reduction in mean μSBS after thermal fatigue may be associated with the small molecular size and high molar concentration of water, which allows penetration of the nano-size spaces between polymer chains or clusters around functional groups that are capable of hydrogen bonding.[27] This phenomenon could result in a decrease in thermal stability and polymer plasticization.[28] In addition, the temperature changes to which the specimens are subjected increase the coefficient of thermal expansion at the adhesive/ceramic interface leading to a premature failure and/or lower μSBS values, due to the loss of chemical retention.[28] This

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-08-30 via free access

Table 6: *Mean ± Standard Deviation of Microshear Bond Strength (μSBS) of Universal Adhesives Bonded to Leucite-reinforced Glass-ceramic and Ytrium-stabilized Zirconium Dioxide*[a]

Adhesive System	Leucite-reinforced Glass-ceramic (LeGC)			Yttrium-stabilized Zirconium Dioxide (ZiDi)		
	Immediate	After Thermocycling	Bond Strength Reduction (%)	Immediate	After Thermocycling	Bond Strength Reduction (%)
ADU	27.2 ± 1.7 B	14.5 ± 2.7 D	46.7	32.1 ± 1.5 a	15.4 ± 1.5 de	52.1
ABU	28.7 ± 1.7 AB	14.2 ± 2.2 D	50.5	33.4 ± 1.2 a	17.9 ± 1.9 d	46.5
AMB	28.9 ± 2.0 A	18.4 ± 3.1 C	39.1	30.6 ± 2.0 ab	20.0 ± 2.1 c	34.2
CFU	26.2 ± 1.9 B	12.0 ± 1.8 DE	54.2	30.7 ± 1.7 ab	13.2 ± 1.5 e	57.0
FBU	27.6 ± 1.5 B	16.2 ± 3.2 CD	41.3	29.2 ± 1.3 b	17.7 ± 1.1 d	39.4
OCU	20.3 ± 1.3 C	13.2 ± 2.8 DE	35.0	33.1 ± 1.4 a	20.0 ± 2.8 c	39.6
PUB	25.2 ± 1.6 A	15.0 ± 3.4 CD	40.5	30.2 ± 1.6 ab	15.8 ± 1.9 de	47.7
PBE	26.4 ± 1.4 B	12.7 ± 2.6 DE	51.9	31.5 ± 1.5 ab	4.7 ± 0.7 f	84.6
SBU	30.2 ± 1.9 A	14.7 ± 2.2 D	51.3	32.6 ± 1.1 a	20.7 ± 3.2 c	36.5
XEN	18.1 ± 2.0 C	10.0 ± 2.2 E	44.8	22.3 ± 0.9 c	7.7 ± 4.1 f	65.5

[a]*Different uppercase (ReRC) and lowercase (LiDi) letters indicate statistically significant differences for each restorative material (two-way ANOVA, Tukey's test,* $p<0.05$*).*

could explain the number of adhesive/mixed failures for all indirect materials after thermocycling, with only a few cohesive fractures of cement or indirect material. For ZiDi it is worth mentioning that 16.7% of premature failures occurred with CFU, 40% with PBE, and 60% with XEN after thermocycling (Table 3).

However, the decrease in mean μSBS was not similar for all universal adhesives when evaluated in different indirect materials. Universal adhesives were developed based on the idea of several manufacturers to include acidic functional monomers, such as MDP, in the composition of these adhesives. The addition of acidic functional monomers provides them with a versatility to adhere to dental substrates of different characteristics, such as enamel and dentin, in addition to other substrates including glass-matrix ceramics, oxide-based ceramics, and metal alloys without the need for additional primers.[25,29]

However, some recent studies showed that the presence of MDP in universal adhesives did not have a significant influence on bond strengths when evaluated in the immediate time.[12,30] In fact, several other acidic functional monomers, such as PENTA, MCAP and D3MA (Table 1), could be added to improve the bonding to direct and indirect materials.[31]

In a previous study, PUB, a MDP-free universal adhesive, was the only adhesive for which the mean μSBS reached the highest ranking of statistical significance among all adhesives for all five CAD/CAM indirect materials after 24 hours of storage in distilled water.[12] At that time, the authors attributed these results to the higher viscosity of PUB compared to that of other universal adhesives, which might be responsible for better physical properties. However, in the current study, PUB showed an intermediary behavior in terms of μSBS after thermocycling, with a reduction of 35.8% when all indirect materials are evaluated together. This can be explained because it is not possible to find the presence of any functional monomer within the composition of PUB. Actually, methacrylic acid is a common component of several adhesives without any specific functionality in terms of bonding.[32] According to the respective manufacturer, PUB contains diacetate of chlorhexidine in its composition, mainly because the addition of this compound helps prevent dentin bonding degradation in the adhesive interface *in vitro*.[33] However, there is no evidence that the use of chlorhexidine could improve the bonding to indirect materials. Future studies need to be done to evaluate this hypothesis.

Two other MDP-free universal adhesives were evaluated in the present study: PBE and XEN. Both showed one of the highest reductions in mean μSBS (49.8% and 47.4%, respectively). Both materials contain PENTA or a phosphoric acid ester group in the functionalized monomer very similar to PENTA.[34] Chen and others[35] observed that primers containing 15 and 20 wt% PENTA increased the binding affinity with ZiDi, via the formation of Zr-O-P bond, when compared to a primer containing MDP. Unfortunately, the exact concentration of PENTA or derivates in PBE and XEN are a property of the respective

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-08-30 via free access

manufacturer. However, when commercial universal adhesives containing PENTA were compared to commercial universal adhesives containing MDP, lower bond strength to Lidi and ZiDi were observed for the former.[18,22,24] According to Elsayed and others,[24] this could 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, mainly to ZiDi.[35,36] This mechanism may explain the worst behavior of PBE and XEN in terms of bond strength reduction (84.6% and 65.5%, respectively) when evaluated on ZiDi. In general, intermediary results were obtained for MDP-free universal adhesives when compared to MDP-containing adhesives.

It is worth mentioning that PBE is the only acetone-based universal adhesive. Ethanol, water, or a mix of them are commonly used as solvents of universal adhesive. Acetone has a higher vapor pressure than ethanol and water, which may reduce the time required for evaporation compared to ethanol.[32] A recent study showed that a longer evaporation time than that recommended by the respective manufacturer was required for PBE.[37] This may occur because of the high acetone content in PBE, which may hinder an adequate solvent evaporation after application. This can leave residual solvent in the adhesive resin, which results in porosities in the cured adhesive layer,[38] which has been corroborated by several authors when acetone-based universal adhesives were applied under indirect substrates.[18,22,24]

Also, the pH of universal adhesives seems to play an important role in the bond strengths to different substrates. XEN has the lowest pH among universal adhesives used in this study (pH=1.6)[39] while PBE has a pH around 2.5.[37] These factors might be responsible for the lower adhesion capability of PBE and XEN. However, it is worth mentioning that the respective manufacturer does not recommend XEN for indirect restorations.

We should also point out that an MDP-containing silane was applied on the indirect materials surface prior to the universal adhesives tested. Several studies have shown that the use of an MDP - containing silane improves the chemical interaction when associated with MDP-containing universal adhesives applied under glass ceramics, leucite-reinforced,[25] lithium disilicate[22,23] and yttrium-stabilized zirconium dioxide surfaces.[18,40] For glass ceramics, methacrylate groups within the adhesive can copolymerize with silane molecules[41] and silanol groups produced by the corresponding methoxy groups can react with the glass ceramic surface.[42,43] In the case of yttrium-stabilized zirconium dioxide, MDP may also react with zirconia through hydroxyl groups present both on the MDP molecule and the zirconia surface.[44-46] On the other hand, the application of a second adhesive coating may protect the surface of the MDP-containing silane, maintaining the bonding of silane to lithium disilicate[22] and yttrium-stabilized zirconium dioxide.[18]

Despite the presence of MDP in the majority of universal adhesives evaluated, several differences were observed between MDP-containing adhesives evaluated. These differences could be explained based on the composition of the different adhesives. For example, considering the concept of versatility to different substrates, at least two silane-containing universal adhesives (ie, CFU and SBU) were launched in the market. According to the respective manufacturers, the silane helps improve the bonding to glass-matrix ceramics, such as feldspathic, leucite-reinforced, and lithium disilicate.

However, some concerns have been raised in the literature regarding this issue. It is generally understood that acidic pre-hydrolyzed silane coupling agents have a relatively short shelf life.[47] This occurs due to the hydrolysis and self-condensation of silane being affected by the pH value of a solution. Generally, the silane used in dentistry has a pH between 4 and 5.[48] On the other side, the pH of CFU is 2.3 and the pH of SBU is 2.7.[12] Therefore, it is unlikely that the incorporation of silane increases the bond strengths for these universal adhesives.[17] In fact, this reinforces the idea that a previous application of a silane-based primer is crucial for current silane-containing universal adhesives.[18,22,43] According to Cuevas-Sanchez and others,[21] there are currently three universal adhesives for which the respective manufacturers indicate that the use of a separate primer for adhesion to silicate ceramics is not necessary (CFU, FBU, and SBU).

However, a closer view of the results in our study showed a lower performance of CFU when compared with SBU in four of the five substrates after thermocycling, which is supported by several studies.[42,49] This may be explained by the presence of silane and a high concentration of Bis-GMA in the composition of CFU (15%-35%) in comparison with the concentration of Bis-GMA in SBU (15%-25%). The two components coexisting in one bottle may have a negative influence on the efficiency of a universal adhesive. Chen and others[50] reported that the incorporation of Bis-GMA monomer significantly inhibited the action of silane-containing porcelain primers and inhibited the chemical reaction between silane primer and glass ceramic.

In a recent study,[17] nuclear magnetic resonance analyses showed that the spectra peaks of 9.90 ppm (Si-O-Si- group) indicates the formation of silane

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-08-30 via free access

oligomers over time, which potentially impair the bonding performance. In this context, another study[51] showed that low Si-O-Si peaks were registered in SBU, probably due to increased propensity for intermediate reactions between silane and the variety of -OH sources in SBU (2-HEMA, MDP, VP-copolymer, water, etc). Thus, even if the silane in SBU does not help improve the adhesive properties, the mixture of all other components is likely to maintain a good chemical interaction of SBU with indirect substrates. This might be the reason why SBU showed better results than CFU in four of the five indirect substrates in our study, with the exception of LeGC.

Although MDP-containing universal adhesives showed higher mean μSBS after thermocycling when compared to MDP-free universal adhesives, there is no consensus on which is the better universal adhesive to be used in all substrates, as per the results of our study. Unfortunately, the exact amount of MDP is a manufacturer's trade secret. For instance, AMB, as well as SBU, showed higher mean μSBS even after thermocycling for four of the five indirect substrates, with the exception to LiDi. According to the manufacturer of AMB,[52] MDP has a higher reactivity, which results from redistributing the concentrations between solvents, water, and acidic monomers. Nevertheless, this concept has not been proven. In addition, the ideal amount of MDP to enhance its interaction with indirect materials is not consensual.

In fact, there are some studies that evaluated the effect of different concentrations of MDP on bonding to zirconia. While Yoshida and others[53] and Nagaoka and others[46] reported increasing bond strengths to zirconia for higher concentrations of MDP up to 1 wt%, Chen and others[54] showed that concentrations of MDP up to 10% showed higher bond strengths. However, the chemical affinity of MDP for zirconia is optimally achieved using 10 wt% MDP. On the other hand, Llerena-Icochea and others[55] evaluated experimental adhesives containing 3 to 15 wt% MDP when applied to zirconia. The results showed that there was no significant correlation between the concentration of MDP in the experimental adhesives and mean bond strengths to zirconia. Future studies need to be carried out to evaluate the exact concentration of MDP needed in the primers and universal adhesives to improve the bonding interaction to indirect resin composite, glass-matrix ceramics, and yttrium-stabilized zirconium dioxide.

Thus, the first null hypothesis was rejected, because all universal adhesives underwent a statistically significant bond strength reduction between the immediate time and after 10,000 thermal cycles, except AMB, FBU, and SBU for FeCe substrate. The second null hypothesis was rejected, as the mean microshear bond strengths among the different universal adhesives varied widely for each CAD/CAM material used.

CONCLUSION

Factors such as pH, type of solvent, and the presence of silane and/or MDP in the composition of each adhesive seem to be important in choosing a universal adhesive system for each particular substrate.

Acknowledgments

The authors would like to thank NipponLab (Ponta Grossa, PR, Brazil) for donation of all indirect materials used in the present study and Kerr Brazil for the donation of NX3. This study was partially supported by the National Council for Scientific and Technological Development (CNPq) under grants 303332/2017-4 and 308286/2019-7 and Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) - Finance Code 001.

Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 20 March 2021)

REFERENCES

1. van Noort R (2012) The future of dental devices is digital *Dental Materials* **28(1)** 3-12. doi: 10.1016/j.dental.2011.10.014
2. Alghazzawi TF (2016) Advancements in CAD/CAM technology: Options for practical implementation *Journal of Prosthodontic Research* **60(2)** 72-84. doi: 10.1016/j.jpor.2016.01.003
3. Ruse ND & Sadoun MJ (2014) Resin-composite blocks for dental CAD/CAM applications *Journal of Dental Research* **93(12)** 1232-1234. doi: 10.1177/0022034514553976
4. Frankenberger R, Hartmann VE, Krech M, Krämer N, Reich S, Braun A, & Roggendorf M (2015) Adhesive luting of new CAD/CAM materials *International Journal of Computerized Dentistry* **18(1)** 9-20.
5. Davidowitz G & Kotick PG (2011) The use of CAD/CAM in dentistry *Dental Clinics of North America* **55(3)** 559-570. ix. doi: 10.1016/j.cden.2011.02.011
6. Gracis S, Thompson VP, Ferencz JL, Silva NRF, & Bonfante EA (2015) A new classification system for all-ceramic and ceramic-like restorative materials *International Journal of Prosthodontics* **28(3)** 227-235. doi: 10.11607/ijp.4244
7. Wendler M, Belli R, Petschelt A, Mevec D, Harrer W, Lube T, Danzer R, & Lohbauer U (2017) Chairside CAD/CAM materials. Part 2: Flexural strength testing *Dental Materials* **33(1)** 99-109. doi: 10.1016/j.dental.2016.10.008

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-08-30 via free access

8. Spitznagel FA, Boldt J, & Gierthmuehlen PC (2018) CAD/CAM ceramic restorative materials for natural teeth *Journal of Dental Research* **97(10)** 1082-1091. doi: 10.1177/0022034518779759

9. Ozcan M & Bernasconi M (2015) Adhesion to zirconia used for dental restorations: A systematic review and meta-analysis *Journal of Adhesive Dentistry* **17(1)** 7-26. doi: 10.3290/j.jad.a33525

10. Flury S, Schmidt SZ, Peutzfeldt A, & Lussi A (2016) Dentin bond strength of two resin-ceramic computer-aided design/computer-aided manufacturing (CAD/CAM) materials and five cements after six months storage *Dental Materials Journal* **35(5)** 728-735. doi: 10.4012/dmj.2016-095

11. Maruo Y, Nishigawa G, Irie M, Yoshihara K, Matsumoto T, & Minagi S (2017) Does acid etching morphologically and chemically affect lithium disilicate glass ceramic surfaces? *Journal of Applied Biomaterials & Functional Materials* **15(1)** e93-e100 doi: 10.5301/jabfm.5000303

12. Siqueira F, Cardenas AM, Gutierrez MF, Malaquias P, Hass V, Reis A, Loguercio AD, & Perdigão J (2016) Laboratory performance of universal adhesive systems for luting CAD/CAM Restorative Materials *Journal of Adhesive Dentistry* **18(4)** 331-340. doi: 10.3290/j.jad.a36519

13. Krämer N & Frankenberger R (2005) Clinical performance of bonded leucite-reinforced glass ceramic inlays and onlays after eight years *Dental Materials* **21(3)** 262-271. doi: 10.1016/j.dental.2004.03.009

14. Lee MH, Son JS, Kim K-H & Kwon T-Y (2015) Improved Resin-zirconia bonding by room temperature hydrofluoric acid etching *Materials (Basel)* **8(3)** 850-866. doi: 10.3390/ma8030850

15. Liu D, Tsoi JK-H, Matinlinna JP, & Wong HM (2015) Effects of some chemical surface modifications on resin zirconia adhesion *Journal of the Mechanical Behavior of Biomedical Materials* **46** 23-30. doi: 10.1016/j.jmbbm.2015.02.015

16. Yoshihara K, Nagaoka N, Sonoda A, Maruo Y, Makita Y, Okihara T, Irie M, Yoshida Y, & Van Meerbeek B (2016) Effectiveness and stability of silane coupling agent incorporated in 'universal' adhesives *Dental Materials* **32(10)** 1218-1225. doi: 10.1016/j.dental.2016.07.002

17. Yao C, Yu J, Wang Y, Tang C, & Huang C (2018) Acidic pH weakens the bonding effectiveness of silane contained in universal adhesives *Dental Materials* **34(5)** 809-818. doi: 10.1016/j.dental.2018.02.004

18. Gutierrez MF, Perdigão J, Malaquias P, Cardenas AM, Siqueira F, Hass V, Reis A, & Loguercio AD (2020) Effect of methacryloyloxydecyl dihydrogen phosphate-containing silane and adhesive used alone or in combination on the bond strength and chemical interaction with zirconia ceramics under thermal aging *Operative Dentistry* **45(5)** 516-527. doi: 10.2341/18-093-L

19. Inokoshi M, Kameyama A, De Munck J, Minakuchi S, & Van Meerbeek B (2013) Durable bonding to mechanically and/or chemically pre-treated dental zirconia *Journal of Dentistry* **41(2)** 170-179 doi: 10.1016/j.jdent.2012.10.017

20. Shimada Y, Yamaguchi S, & Tagami J (2002) Micro-shear bond strength of dual-cured resin cement to glass ceramics *Dental Materials* **18(5)** 380-388. doi: 10.1016/s0109-5641(01)00054-9

21. Cuevas-Suárez CE, de Oliveira da Rosa WL, Vitti RP, da Silva AF, & Piva E (2020) Bonding strength of universal adhesives to indirect substrates: A meta-analysis of *in vitro* studies *Journal of Prosthodontics* **29(4)** 298-308. doi: 10.1111/jopr.13147

22. Cardenas AM, Siqueira F, Hass V, Malaquias P, Gutierrez MF, Reis A, Perdigão J, & Loguercio A (2017) Effect of MDP-containing silane and adhesive used alone or in combination on the long-term bond strength and chemical interaction with lithium disilicate ceramics *Journal of Adhesive Dentistry* **19(3)** 203-212. doi: 10.3290/j.jad.a38414

23. Maier E, Bordihn V, Belli R, Taschner M, Petschelt A, Lohbauer U, & Zorzin J (2019) New approaches in bonding to glass-ceramic: Self-etch glass-ceramic primer and universal adhesives *Journal of Adhesive Dentistry* **21(3)** 209-217. doi: 10.3290/j.jad.a42546

24. Elsayed A, Younes F, Lehmann F, & Kern M (2017) Tensile Bond Strength of So-called Universal Primers and Universal Multimode Adhesives to Zirconia and Lithium Disilicate Ceramics *Journal of Adhesive Dentistry* **19(3)** 221-228. doi: 10.3290/j.jad.a38436

25. Kim RJ-Y, Woo J-S, Lee I-B, Yi Y-A, Hwang J-Y, & Seo D-G (2015) Performance of universal adhesives on bonding to leucite-reinforced ceramic *Biomaterials Research* **19** 11. doi: 10.1186/s40824-015-0035-1

26. Awad MM, Albedaiwi L, Almahdy A, Khan R, Silikas N, Hatamleh MM, Alkhtani FM, & Alrahlah A (2019) Effect of universal adhesives on microtensile bond strength to hybrid ceramic *BMC Oral Health* **19(1)** 178. doi: 10.1186/s12903-019-0865-7

27. Attia A & Kern M (2011) Long-term resin bonding to zirconia ceramic with a new universal primer *Journal of Prosthetic Dentistry* **106(5)** 319-327. doi: 10.1016/S0022-3913(11)60137-6

28. Oyagüe RC, Monticelli F, Toledano M, Osorio E, Ferrari M, & Osorio R (2009) Effect of water aging on microtensile bond strength of dual-cured resin cements to pre-treated sintered zirconium-oxide ceramics *Dental Materials* **25(3)** 392-399. doi: 10.1016/j.dental.2008.09.002

29. Bömicke W, Schürz A, Krisam J, Rammelsberg P, & Rues S (2016) Durability of resin-zirconia bonds produced using methods available in dental practice *Journal of Adhesive Dentistry* **18(1)** 17-27. doi: 10.3290/j.jad.a35517

30. Nagarkar S, Theis-Mahon N, & Perdigão J (2019) Universal dental adhesives: Current status, laboratory testing, and clinical performance *Journal of Biomedical Materials Research Part B: Applied Biomaterials* **107(6)** 2121-2131. doi: 10.1002/jbm.b.34305

31. Van Meerbeek B, Yoshihara K, Van Landuyt K, Yoshida Y, & Peumans M (2020) From buonocore's pioneering acid-etch technique to self-adhering restoratives. A status perspective of rapidly advancing dental adhesive Technology *Journal of Adhesive Dentistry* **22(1)** 7-34. doi: 10.3290/j.jad.a43994

32. Van Landuyt KL, Snauwaert J, De Munck J, Peumans M, Yoshida Y, Poitevin A, Coutinho E, Suzuki K, Lambrechts P, & Van Meerbeek B (2007) Systematic review of the chemical composition of contemporary dental adhesives *Biomaterials* **28(26)** 3757-3785. doi: 10.1016/j.biomaterials.2007.04.044

33. Maravić T, Comba A, Cunha SR, Angeloni V, Cadenaro M, Visinitini E, Navarra CO, Salgarello S, Breschi L, & Mazzoni A (2019) Long-term bond strength and endogenous enzymatic activity of a chlorhexidine-containing commercially

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-08-30 via free access

available adhesive *Journal of Dentistry* **84** 60-66. doi: 10.1016/j.jdent.2019.03.004

34. Van Loghum BS (2014) Fact file: XENO®Select *Tandartspraktijk* **35(5)** 50-51. doi.org/10.1007/s12496-014-0061-5

35. Chen Y, Tay FR, Lu Z, Chen C, Qian M, Zhang H, Tian F, & Xie H (2016) Dipentaerythritol penta-acrylate phosphate - an alternative phosphate ester monomer for bonding of methacrylates to zirconia *Scientific Reports* **6** 39542. doi: 10.1038/srep39542

36. Triwatana P, Visuttiwattanakorn P, Suputtamongkol K, & Butrangamdee N (2016) Bond strength and failure characteristics of zirconia-based dental ceramic to resin cements *Mahidol Dental Journal* **36** 13-23.

37. Luque-Martinez IV, Perdigão J, Muñoz MA, Sezinando A, Reis A, & Loguercio AD (2014) Effects of solvent evaporation time on immediate adhesive properties of universal adhesives to dentin *Dental Materials* **30(10)** 1126-1135. doi: 10.1016/j.dental.2014.07.002

38. Cho B-H & Dickens SH (2004) Effects of the acetone content of single solution dentin bonding agents on the adhesive layer thickness and the microtensile bond strength *Dental Materials* **20(2)** 107-115. doi: 10.1016/s0109-5641(03)00071-x

39. Dentsply FAQs. Available at http://www.dentsplysirona.com. Accessed March 10 2020.

40. Xie H, Li Q, Zhang F, Lu Y, Tay FR, Qian M, & Chen C (2016) Comparison of resin bonding improvements to zirconia between one-bottle universal adhesives and tribochemical silica coating, which is better? *Dental Materials* **32(3)** 403-411. doi: 10.1016/j.dental.2015.12.014

41. Shen C, Oh W-S, & Williams JR (2004) Effect of post-silanization drying on the bond strength of composite to ceramic *Journal of Prosthetic Dentistry* **91(5)** 453-458. doi: 10.1016/S0022391304001301

42. Yao C, Yang H, Yu J, Zhang L, Zhu Y, & Huang C (2018) High bond durability of universal adhesives on glass ceramics facilitated by silane pretreatment *Operative Dentistry 43(6)* 602-612. doi: 10.2341/17-227-L

43. Yao C, Zhou L, Yang H, Wang Y, Sun H, Guo J, & Huang C (2017) Effect of silane pretreatment on the immediate bonding of universal adhesives to computer-aided design/computer-aided manufacturing lithium disilicate glass ceramics *European Journal of Oral Sciences* **125(2)** 173-180. doi: 10.1111/eos.12340

44. Inokoshi M, De Munck J, Minakuchi S, & Van Meerbeek B (2014) Meta-analysis of bonding effectiveness to zirconia ceramics *Journal of Dental Research* **93(4)** 329-334. doi: 10.1177/0022034514524228

45. Piascik JR, Swift EJ, Braswell K, & Stoner BR (2012) Surface fluorination of zirconia: Adhesive bond strength comparison to commercial primers *Dental Materials* **28(6)** 604-608. doi: 10.1016/j.dental.2012.01.008

46. Nagaoka N, Yoshihara K, Feitosa VP, Tamada Y, Irie M, Yoshida Y, Van Meerbeek B, & Hayakawa S (2017) Chemical interaction mechanism of 10-MDP with zirconia *Scientific Reports* **7** 45563. doi: 10.1038/srep45563

47. Lung CYK & Matinlinna JP (2012) Aspects of silane coupling agents and surface conditioning in dentistry: An overview *Dental Materials* **28(5)** 467-477. doi: 10.1038/srep45563

48. Perdigão J, Gomes G, & Lee IK (2006) The effect of silane on the bond strengths of fiber posts *Dental Materials* **22(8)** 752-758. doi: 10.1016/j.dental.2005.11.002

49. Yang L, Chen B, Xie H, Chen Y, Chen Y, & Chen C (2018) Durability of resin bonding to zirconia using products containing 10-methacryloyloxydecyl dihydrogen phosphate *Journal of Adhesive Dentistry* **20(4)** 279-287. doi: 10.3290/j.jad.a40989

50. Chen L, Shen H, & Suh BI (2013) Effect of incorporating BisGMA resin on the bonding properties of silane and zirconia primers *Journal of Prosthetic Dentistry* **110(5)** 402-407. doi: 10.1016/j.prosdent.2013.04.005

51. Dimitriadi M, Panagiotopoulou A, Pelecanou M, Yannakopoulou K, & Eliades G (2018) Stability and reactivity of γ-MPTMS silane in some commercial primer and adhesive formulations *Dental Materials* **34(8)** 1089-1101. doi: 10.1016/j.dental.2018.05.006

52. Ambar Universal APS Dental Adhesive. Available at https://fgmus.com/product/ambar-universal-aps/. Accessed July 19 2020.

53. Yoshida K, Tsuo Y, & Atsuta M (2006) Bonding of dual-cured resin cement to zirconia ceramic using phosphate acid ester monomer and zirconate coupler *Journal of Biomedical Materials Research Part B: Applied Biomaterials* **77(1)** 28-33. doi: 10.1002/jbm.b.30424

54. Chen Y, Lu Z, Qian M, Zhang H, Xie H, & Chen C (2017) Effect of 10-methacryloxydecyl dihydrogen phosphate concentration on chemical coupling of methacrylate resin to yttria-stabilized zirconia *Journal of Adhesive Dentistry* **19(4)** 349-355. doi: 10.3290/j.jad.a38893

55. Llerena-Icochea AE, Costa RM, Borges A, Bombonatti Jfs, & Furuse AY (2017) Bonding Polycrystalline zirconia with 10-MDP-containing adhesives *Operative Dentistry* **42(3)** 335-341. doi: 10.2341/16-156-L

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-08-30 via free access