Laboratory Performance of Universal Adhesive Systems for Luting CAD/CAM Restorative Materials

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Purpose: To evaluate the microshear bond strength (μSBS) of several universal adhesive systems applied on five different indirect restorative materials.

Materials and Methods: Five CAD/CAM materials were selected: 1) indirect resin composite (LAV); 2) feldspathic glass ceramic (VTR); 3) leucite-reinforced glass-ceramic (EMP); 4) lithium disilicate ceramic (EMX); 5) yttrium-stabilized zirconium dioxide (CZI). For each material, 15 blocks were cut into 4 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 (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 stored in water (37°C for 24 h) and tested in shear mode at 1.0 mm/min (mSBS). The failure pattern and μSBS were statistically evaluated (a = 0.05).

Results: LAV, VTR, and EMP showed a greater number of cohesive fractures than EMX and CZI ($p < 0.0001$). PUB was the only adhesive for which the mean μSBS reached the highest ranking of statistical significance for all five substrates. When each adhesive was compared across the five substrates, 8 out of 10 (ADU, ABU, AMB, CFU, OCU, PUB, PBE, and SBU) reached the statistically highest mean μSBS when applied on CZI.

Conclusion: The specific chemical composition of universal adhesives was not the decisive factor in the bond strength values measured for different CAD/CAM indirect materials. There was a wide variability in mean μSBS when different universal adhesives were applied to the several CAD/CAM indirect materials. Most universal adhesives bonded well to air-abraded zirconia.

Keywords: resin cement, universal dentin adhesive, CAD/CAM restorative materials, bond strength.

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During the last decade, the use of indirect dental restorations has increased, triggered by advances in manufacturing technologies and development of materials with improved mechanical properties.[6,31,42] Currently, bonded restorations are an integral part of minimally invasive dentistry due to advances in adhesion technology.

The adhesive luting procedure of tooth-colored indirect restorations is the final step in a sequence of pre-luting

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steps. Besides the strength of the restoration, the adhesion of luting cements to both substrates (ie, the dental tissues and the indirect restorative material) is essential for long-term clinical success.[55]

The intaglio surface of resin- or ceramic-based indirect restorations can be treated with sandblasting, hydrofluoric acid (HF), silane coupling agents, or with a combination of these.[65,69] The traditional silica-based ceramics or glass-matrix ceramics[28] are still extensively used for porcelain veneers, inlays/onlays, and single crowns. The adhesion of composite resin to glass-matrix ceramics is achieved through a combination of mechanical retention from HF etching and chemical adhesion provided by the silane-coupling agent.[12,35,41,66]

HF creates an etching pattern on glass-matrix ceramics similar to the microporosities created by phosphoric acid on enamel surfaces. The resulting microretention increases the ceramic bonding area for resin cements.[3,7,66] A silane coupling agent is subsequently applied to promote adhesion between the inorganic phase of the ceramic and the organic phase of the luting adhesive system through siloxane bonds.[48] In addition, silane increases the surface energy of ceramic substrates and improves cement wettability.[49,59]

Recent advances in CAD/CAM technology have changed the classical concepts of ceramic restorations. Glass-free oxide-based or polycrystalline ceramics, also known as high-strength ceramic materials, have become extremely popular. HF etching does not improve bond strengths to oxide-based or polycrystalline ceramics (ie, alumina, stabilized zirconia, and zirconia-toughened alumina or alumina-toughened zirconia),[10,28,37,38,44,46,56] since these substrates do not contain a glass matrix. Accordingly, other surface conditioning methods have been used in the last two decades to improve bond strengths of resin luting cements to polycrystalline ceramics.[10,25,38,56] Some of the newest protocols include primers or silanes mixed with functional monomers, such as the phosphate ester monomer 10-methacryloyloxydecyl dihydrogenphosphate (MDP).[17,55,72]

A novel family of adhesive systems, known as universal adhesives, has been recently introduced.[30,53,58] For enamel and dentin, universal adhesives have been recommended as etch-and-rinse (two-step) and self-etching (one- or two-step) adhesives. This versatility results from the inclusion of acidic functional monomers, such as MDP, in their composition. More recently, the indication for universal adhesives has been expanded to other substrates, including glass-matrix ceramics, oxide-based ceramics, and metal alloys without the need for additional primers.[9,39,63] While some current universal adhesives contain different functional monomers, the effectiveness of universal adhesives with or without MDP has not been thoroughly investigated.

Thus, the aim of the present study was to evaluate the microshear bond strength of several universal adhesive systems on five different indirect materials. The null hypotheses tested were: 1) universal adhesives would not result in different bond strengths for each indirect substrate, and 2) a given universal adhesive would not result in different bond strengths when applied to all five indirect materials.

MATERIALS AND METHODS

Specimen Preparation

Five CAD/CAM materials were selected: 1) indirect resin composite (LAV, Lava Ultimate CAD/CAM Restorative 3M ESPE; St Paul, MN, USA); 2) feldspathic glass ceramic (VTR, Vitablocs RealLife, VITA Zahnfabrik; Bad Säckingen, Germany); 3) leucite-reinforced glass ceramic (EMP, IPS Empress CAD, Ivoclar Vivadent; Schaan, Liechtenstein); 4) lithium disilicate (EMX, IPS e.max CAD, Ivoclar Vivadent); 5) yttrium-stabilized zirconium dioxide (Y-TZP) (CZI, Ceramill Zi, Amann Girrbach; Koblach, Austria).

A total of 75 CAD/CAM blocks, 15 for each material, were used in the present study. For each material, the blocks (12 × 12 × 6 mm) were cut into 4 rectangular sections (6 × 6 × 6 mm) (n = 60 per group) using a diamond disk at slow speed (Isomet Buehler; Lake Bluff, IL, USA) under water cooling. When necessary, specimens were fired following the crystallization program recommended by the respective manufacturer, while CZI specimens were sintered according to the recommended protocol (Table 1).

Experimental Design

For each indirect material, the specimens (n = 60) were randomly assigned (http://www.sealedenvelope.com) into 10 groups according to the adhesive system used: 1. AdheSE Universal (ADU, Ivoclar Vivadent); 2. All-Bond Universal (ABU, Bisco; Schaumburg, IL, USA); 3. Ambar Universal (AMB, FGM Prod Odont; Joinville, SC, Brazil); 4. Clearfil Universal (CFU, Kuraray Noritake Dental; Tokyo, Japan); 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; Konstanz, Germany); 9. Scotchbond Universal Adhesive (SBU, 3M ESPE, also known as Single Bond Universal in some countries); 10. Xeno Select (XEN, Dentsply, also known as Prime&Bond One Select in some countries). 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)

After polyvinyl chloride (PVC) rings were filled with acrylic resin (AutoClear, DentBras; Pirassununga, São Paulo, Brazil), the specimens were embedded into the acrylic resin protruding 3 mm from the top of the PVC ring. The materials and respective surface treatments are displayed in Table 1. The universal adhesives were then applied according to the respective manufacturer's instructions (Table 2). A single operator performed all bonding procedures.

A pilot study demonstrated that the delimitation of the bonding area resulted in no significant difference in bond strengths compared to no delimitation. After the application of the adhesive system, five polyethylene transparent Tygon tubes (Tygon Medical Tubing Formulations 54-HL, Saint Gobain Performance Plastics; Akron, OH, USA) with an inter-

Table 1 Materials used, composition, and surface treatment

Material	Composition	Surface treatment
Indirect resin composite (LAV, Lava Ultimate CAD/CAM, 3M ESPE)	Bis-GMA, UDMA, bis-EMA, TEG-DMA, 80 SiO_2 (20 nm), ZrO_2 (4–11 nm), aggregated ZrO_2 /SiO_2 cluster	Sandblasted with Al_2O_3, <50 µm (2 bar, until entire bonding surface appeared matte). Ultrasonically cleaned with distilled water for 180 s. The surface was throughouly rinsed (5 ml) with ethyl alcohol (70%). Dried with oil-free air for 30 s. Silane solution: Monobond Plus (Ivoclar Vivadent) was 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.
Feldspathic glass ceramic (VTR, Vitablocs RealLife, VITA Zahnfabrik)	SiO_2, Al_2O_3, K_2O, Na_2O, CaO, TiO_2	5% hydrofluoric acid etching for 60 s (Vita Ceramics Etch; batch 42530). 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.
Leucite-reinforced glass-ceramic (EMP, 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) was 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 (EMX, 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°C–850°C for 20–31 min. 5% hydrofluoric acid etching for 20 s*. 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) was 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 (CZI, Ceramill Zi, Amann Girrbach)	$ZrO_2 + HfO_2 + Y_2O_3$: >99% Y_2O_3: 4.5–5.6% HfO_2: < 5% Al_2O_3: < 0.5% Other oxides: <0.5%	Sintered in a furnace (Ceramill Therm, Amann Girrbach; Curitiba, PR, Brazil) using a universal program (8°C/min from 200°C to 1450°C), 2 hours at a fixed temperature of 1450°C, and the correct cooling time. Sandblasted with <50-µm Al_2O_3 particles (2.8 bar, 7 s). Ultrasonically cleaned with distilled water for 180 s. The surface was throughouly rinsed (5 ml) with ethyl alcohol (70%). Dried with oil-free air for 30 s. Silane solution: Monobond Plus (Ivoclar Vivadent) was 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.

Bis-GMA: bisphenol-A diglycidylether methacrylate; UDMA: urethane dimethacrylate; bis-EMA: ethoxylated bisphenol-A dimethacrylate; TEG-DMA: triethylene glycol dimethacrylate. *Condac Porcelain Etch 5% (FGM Prod Odont; Joinville, SC, Brazil).

nal diameter of 0.8 mm and a height of 0.5 mm were positioned over the substrate. A dual-curing resin cement (NX3, Kerr; Orange, CA, USA) 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 s using an LED light-curing unit set at 1200 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 every 5 specimens. These procedures were carried out under magnifying loupes.[54]

After storage of the specimens in distilled water for 24 h at 37°C, the Tygon tubes were carefully removed with a blade, exposing the cement cylinders. Each specimen was examined under a stereomicroscope at 10X magnification. The bonded cylinder was discarded if there was evidence of porosities or gaps at the interface.

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.[64] 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) to determine the

Table 2 Adhesive system (batch number), composition, and application mode of the adhesive systems according the manufacturer's instructions

Adhesive (batch number)	Composition	Application mode*
AdheSE Universal, Ivoclar Vivadent (UO2709)	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 1200 mW/cm^2.
All-Bond Universal, Bisco (1500002859)	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 1200 mW/cm^2.
Ambar Universal, FGM (210415)	Methacrylate monomers (UDMA and 10-MDP), photoinitiators, co-initiators, stabilizers, inert silica nanoparticles and ethanol	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, Kuraray Noritake (CR0002)	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 1200 mW/cm^2.
Futurabond U, Voco (1346518)	HEMA, bis-GMA, HEDMA, acidic adhesive monomer, urethane dimethacrylate, catalyst, silica nanoparticles, ethanol	1. Apply the adhesive with a 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 1200 mW/cm^2.
One Coat 7 Universal, Coltene (F96836)	Methacrylates including 10-MDP, photoinitiators, 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 1200 mW/cm^2.
Peak Universal Bond, Ultradent (BB7D7)	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. Thin/dry for 10 s using air pressure. 3. Light cure for 10 s at 1200 mW/cm^2.
Prime&Bond Elect, Dentsply (130811)	Mono-, di- and trimethacrylate resins, PENTA diketone, organic phosphine oxide, stabilizers, cetylamine hydrofluoride, acetone, water	1. Apply a generous amount of adhesive to thoroughly wet all surfaces and leave undisturbed for 20 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 1200 mW/cm^2.
Scotchbond Universal, 3M ESPE (523652)	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 it no longer moves and the solvent is evaporated completely. 3. Light cure for 10 s at 1200 mW/cm^2.
Xeno Select, Dentsply (1401001210)	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 at least 5 s. Surface should have a uniform, glossy appearance. 3. Light cure for 10 s at 1200 mW/cm^2.
NX3, Kerr (1401001210)	Bis-GMA, UDMA, EBPADMA, TEG-DMA, inert mineral fillers, activators, stabilizers and radiopaque agent	1. 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. 2. Light cure for 20 s at 1200 mW/cm^2.
Monobond Plus, Ivoclar Vivadent (1401001210)	Ethanol, 3-trimethoxysilylpropyl 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.

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; EBPADMA (ethoxylated bisphenol A-dimethacrylate); TEG-DMA (triethylene glycol dimethacrylate). *The intensity of light curing was standardized for all materials.

Table 3 **Number (%) of specimens according to fracture mode**

Adhesive system	Feldspathic glass ceramic (VTR, Vita Mark II)			Indirect resin composite (LAV, Lava Ultimate CAD/CAM)			Leucite-reinforced glass ceramic (EMP, IPS Empress CAD)			Lithium disilicate glass ceramic (EMX, IPS e.max CAD)			Yttrium-stabilized zirconium dioxide (CZI, 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 (27)	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)

Abbreviations: A/M: adhesive/mixed fracture mode; CR: cohesive in resin cement; CC: cohesive in indirect restorative material

shear bond strength. After testing, the specimens were examined under an optical microscope (SZH-131, Olympus; Tokyo, Japan) at 100X 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.

Statistical Analysis

The data were first analyzed using the Kolmogorov-Smirnov test to assess whether the data followed a normal distribution, and Barlett's test for equality of variances to determine if the assumption of equal variances was valid.[51] 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. One-way ANOVA was used to analyze the μSBS data for each indirect material (adhesive), as well as the μSBS data for each adhesive separately (indirect material). The failure pattern of indirect materials was compared using the chi-squared test; the Tukey's post-hoc test was used at $\alpha = 0.05$.

RESULTS

Thirty bonded cylinders were tested for each group. Although the majority of specimens showed adhesive/mixed failures (Table 3), for some materials a lower percentage of cohesive failure in resin cement or in indirect material (Table 3) occurred. For instance, 3% to 37% of cohesive fractures (CC) occurred in the LAV substrate. Likewise, there were 10% to 40% and 0% to 40% of cohesive fractures in the substrate for VTR and EMP, respectively. The CC for LAV, VTR, and EMP were statistically similar ($p > 0.15$) and statistically different than those of EMX and CZI ($p < 0.0001$).

When each substrate was compared across different adhesives (Table 4), PUB was the only adhesive for which the mean μSBS reached the highest ranking of statistical significance among all adhesives for all five CAD/CAM substrates (Table 4). ABU reached the highest ranking of statistical significance for all substrates except for EMX, for which the μSBS with OCU and PUB exceeded that of ABU (Table 4).

For VTR, the application of ADU, ABU, AMB, and FBU resulted in statistically significantly higher mean μSBS than did CFU, OCU, and XEN ($p < 0.001$; Table 4). PBE and SBU yielded intermediate values.

For LAV, the highest mean μSBS values were observed using ABU, AMB, PUB, OCU, and SBU. These means were statistically significantly higher than those of CFU and XEN ($p < 0.004$; Table 4). Intermediate results were found for ADU, FBU, and PBE.

When universal adhesives were applied to EMP, higher μSBS means were measured for AMB, PUB, and SBU, which were statistically higher than those of CFU, FBU, OCU, PBE and XEN ($p < 0.003$; Table 4).

For EMX, only two universal adhesives (OCU and PUB) showed statistically significantly higher mean μSBS when compared with all other adhesive systems ($p < 0.0001$; Table 4). The lowest mean μSBS was mediated by XEN. In-

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Table 4 Means ± standard deviations of microshear bond strength (µSBS) of universal adhesives bonded to indirect restorative materials

Adhesive system	Feldspathic glass ceramic (VTR, Vita Mark II)	Indirect resin composite (LAV, Lava Ultimate CAD/CAM)	Leucite-reinforced glass-ceramic (EMP, IPS Empress CAD)	Lithium disilicate glass-ceramic (EMX, IPS e.max CAD)	Yttrium-stabilized zirconium dioxide (CZI, Ceramill Zi)
ADU	31.1 ± 1.9^{Aa}	24.2 ± 1.9^{BCc}	27.3 ± 0.8^{ABb}	26.1 ± 1.2^{Bbc}	31.2 ± 1.4^{Ba}
ABU	28.7 ± 1.7^{Ab}	28.8 ± 1.3^{Ab}	27.5 ± 1.2^{ABb}	24.3 ± 0.9^{BCc}	34.9 ± 0.9^{Aa}
AMB	27.3 ± 1.9^{Abc}	26.8 ± 2.2^{ABc}	28.3 ± 2.2^{Ab}	26.4 ± 1.4^{Bc}	32.1 ± 1.9^{ABa}
CFU	21.4 ± 2.1^{Cc}	22.8 ± 0.5^{Cc}	24.1 ± 0.9^{Cb}	21.0 ± 1.5^{Cc}	29.5 ± 3.0^{Ba}
FBU	28.4 ± 2.3^{Aa}	25.5 ± 1.3^{Bb}	25.9 ± 1.3^{Bb}	28.0 ± 1.6^{ABa}	26.1 ± 1.1^{Cab}
OCU	22.5 ± 1.0^{Cd}	26.3 ± 1.4^{ABc}	21.5 ± 0.9^{CDd}	28.2 ± 0.7^{Ab}	36.2 ± 0.9^{Aa}
PUB	26.1 ± 1.9^{ABc}	26.5 ± 1.6^{ABc}	28.4 ± 1.1^{Ac}	30.3 ± 0.4^{Ab}	33.1 ± 0.7^{Aa}
PBE	23.6 ± 1.1^{BCc}	25.6 ± 2.9^{Bb}	25.8 ± 1.1^{Bb}	21.9 ± 2.2^{Cc}	33.7 ± 0.9^{Aa}
SBU	23.9 ± 0.9^{BCc}	28.4 ± 2.3^{Aab}	29.4 ± 1.1^{Aa}	27.5 ± 1.5^{Bb}	30.2 ± 2.1^{Ba}
XEN	20.8 ± 0.8^{Cb}	22.4 ± 1.4^{Ca}	19.2 ± 3.0^{Db}	18.1 ± 1.2^{Db}	19.5 ± 0.4^{Dd}

*Different superscript capital letters (in each column for indirect material) and different lowercase letters (in each line for adhesive material) indicate statistically significant differences (one-way ANOVA, Tukey's test, $p < 0.05$)

termediate results were found for ADU, ABU, AMB, CFU, PBE, and SBU.

When the universal adhesives were applied to CZI, the mean µSBS with ABU, AMB, OCU, PUB, and PBE were statistically significantly higher than those with FBU and XEN ($p < 0.001$; Table 4). XEN resulted in significantly lower mean µSBS than any of the other adhesives.

When each adhesive was compared across the 5 substrates, 8 out of 10 adhesives (ADU, ABU, AMB, CFU, OCU, PUB, PBE, and SBU) reached the highest mean µSBS when applied on CZI. However, ADU reached statistically similar mean µSBS for VTR and CZI.

For ABU, higher mean µSBS were measured with CZI, while the lowest mean µSBS were obtained with EMX. Intermediate results were found for VTR, LAV, and EMP. For both AMB and CFU, the lowest mean µSBS values were obtained on VTR, LAV, and EMX. The lowest mean µSBS for OCU were measured on VTR and EMP. For PUB, the lowest mean µSBS were measured on VTR, LAV, and EMP. XEN bonded better to LAV than to any other substrate.

DISCUSSION

Generation of a rough surface for interfacial interlocking is crucial to increase the surface area, resulting in higher bond strengths due to mechanical retention.[47] HF etching is the recommended treatment for glass-matrix ceramics, as adhesive cementation of etched glass-matrix ceramics increases their resistance to fracture.[23,71] For Vitablocs feldspathic ceramic (VTR), 5% HF etching times from 30 s to 120 s have been recommended,[16] while for lithium disilicate ceramic, 20 s is recommended.[52] For Y-TZP, the absence of a glass phase makes HF etching unnecessary.

For glass-matrix ceramics, air abrasion with alumina particles increases surface roughness to a greater extent than does HF acid etching. However, air abrasion results in ceramic specimens with lower immediate intrinsic strength[2] due to the reduction in flexural strength caused by air abrasion on feldspathic ceramics[23] as well as on lithium disilicate ceramics.[52]

LAV is a "tooth-shade resin material" according to the Food and Drug Administration classification.[24] Being a resin-based material, the respective manufacturer recommends treatment of the intaglio surface with airborne particle abrasion with ≤ 50 µm alumina particles, without any chemical etching. Air abrasion with alumina particles improves the fracture resistance of LAV compared to polished LAV specimens,[15] which may be a result of better mechanical interlocking of the bonding resins into the LAV microporosities and better stress distribution at higher loads.

Frankenberger et al[26] measured microtensile bond strengths of LAV luted with a dual-curing adhesive and respective dual-curing resin cement (Prime&Bond XP DC and Calibra, Dentsply). The surface treatment was identical to the technique used in our study, that is, air abrasion with 50-µm alumina particles. After 10,000 thermal cycles, higher bond strengths were obtained in the air-abrasion group compared to the air abrasion + Monobond Plus group, which denotes the importance of air abrasion in establishing reliable mechanical interlocking with LAV intaglio surfaces. On the other hand, the application of Monobond Plus (silane + MDP solution) on air-abraded LAV surfaces may not have had any additional effect compared to air abrasion only. In our study, the MDP solution was applied to all substrates to standardize the experimental procedure. In the case of LAV, air abrasion alone might have resulted in higher bond strengths.

It has been reported that SBU bonds better than FBU to air-abraded LAV surfaces,[67] which was confirmed in our study. In another study,[21] LAV surfaces were treated with 9% HF for 60 s or with air abrasion with 110-µm alumina particles, with or without additional conventional silane application, followed by a dual-curing resin cement without an intermediary adhesive. At 24 h, microtensile bond strengths were similar for all surface treatments. At 30 days, bond strengths decreased significantly for all groups. The magnitude of bond strengths obtained in that study[21] were lower than the bond strengths obtained in our study, which may be a consequence of the different experimental methodology, specifically, the lack of an adhesive material, the different testing setup, and the larger alumina particles. Larger particles may have caused more damage to the LAV surface than the smaller alumina particles used in the present study.

The bonding sequence for LAV recommended by the respective manufacturer includes SBU combined with the dual-curing resin cement Lava Ultimate (3M ESPE). SBU is a light-curing universal adhesive that does not require a dual-curing activator for indirect restorations as long as it is used with RelyX Ultimate. According to the manufacturer's information, this resin cement contains the activator for the adhesive. If combined with any other dual-curing composite resin material, SBU must be mixed with Scotchbond Universal DCA (dual-curing activator) (3M ESPE) at a 1:1 ratio.[1] In our study, SBU was used with the amine-free, dual-curing resin cement NX3 (Kerr), without mixing SBU with its own dual-curing activator. Nonetheless, the mean µSBS values for SBU were relatively high, ranging from 23.9 MPa to 30.2 MPa for the five CAD/CAM substrates. For LAV, SBU achieved the highest statistical ranking (letter A in LAV column, Table 4), which makes the use of the SBU DCA activator unnecessary when SBU is combined with NX3. In fact, most dual-curing resin cements are based on a peroxide-amine redox system, unless they are amine-free, such as RelyX Ultimate and NX3, which may explain the apparent compatibility of all universal adhesives with NX3 used in our study. The pH of SBU is 2.7, which is slightly more acidic than the pH of adhesives for which the respective manufacturers do not recommend a dual-curing activator – ABU (pH = 3.1 to 3.2), AMB (pH = 2.6 to 3.0) and ADU (pH = 2.5 to 3.0).[14,22,57] The lowest pH of the universal adhesives used in this study belongs to XEN (1.6),[19] which may explain why its mean µSBS ranked the lowest for all five substrates. In fact, its manufacturer does not recommend XEN for indirect restorations.

Glass-matrix ceramics, especially the feldspathic type, are stiff and brittle with low fracture toughness and high susceptibility to failure in the presence of flaws,[18,29,62] usually caused by internal tension stresses at the lower ceramic surface that causes cracks in the material.[70] VTR (100 to 150 MPa) and EMP (120 to 180 MPa)[5,13,29] have lower flexural strength than does EMX (360 to 417 MPa),[5,29,73,74] making VTR and EMP more susceptible to fracture than EMX. Both VTR and EMP are also prone to cohesive failure, such as chipping.[29,52] This susceptibility may explain the relatively higher number of cohesive failures in the VTR and EMP groups in our study, as well as that previously reported for EMP bonded with ABU and SBU.[40] The similar pattern of cohesive failures for LAV, EMP, and VTR was somewhat unexpected. The Young's modulus for LAV is 12.8 GPa,[15,20,61] while that for EMP is 62 to 68 GPa[29,33,73] and 26 to 45 GPa for VTR,[5,27,29] which suggests that the stress distribution in LAV specimens under load is somewhat similar to that of stiffer ceramics such as VTR and EMP. In fact, LAV possesses a fracture toughness of 2.0 MPa/m^2, which is higher than that of EMP (1.3 to 2.1 MPa/m^2),[13,20] and Vitablocs feldspathic ceramic (1.3 MPa/m^2),[13] indicating that LAV may have a higher resistance to catastrophic crack propagation under an applied stress, while being less brittle as revealed by the respective modulus of elasticity.

The difference between the flexural strengths of EMP (120 to 180 MPa)[5,13,29] and EMX (360 to 417 MPa)[29,73,74] may explain the higher number of cohesive fractures in the EMP than in EMX specimens. There may be a threshold of physical properties, including flexural strength, between those of EMP and those of EMX, above which the occurrence of cohesive fractures in the substrate is minimal. While radial cracks start from the adhesive interface of monolithic glass-matrix ceramic restorations,[8] adhesive cementation compensates for the low mechanical strength of feldspathic ceramic (such as VTR) and leucite-reinforced ceramic (such as EMP), making them stronger materials. For lithium disilicate (such as EMX), cementation with adhesive techniques is less crucial as a reinforcement technique than for other glass-matrix ceramics,[8] as a result of the much-improved physical properties of lithium disilicate compared to feldspathic and leucite-reinforced ceramics. Bindl et al[8] measured similar fracture loads for lithium disilicate crowns whether they were cemented adhesively or with zinc phosphate.

According to the manufacturer, the recommended luting protocol for EMX involves etching with ≈ 5% HF for 20 s, followed by the application of the primer Monobond Plus (MBP) without the need for applying a layer of adhesive on the intaglio surface. In our study, a universal adhesive was used after drying the MBP solution. Lise et al[43] concluded that the mean µSBS values of a conventional dual-curing resin cement to EMX were not significantly different if an extra layer of adhesive was used compared to MBP without adhesive. For this particular material (EMX), the application of an adhesive may not be crucial, yet it does not seem to reduce bond strengths.

When universal adhesives were compared by substrate in our study (Table 4), all adhesives attained their significantly highest µSBS when applied to Y-TZP (CZI), with the exception of XEN, which bonded significantly better to LAV than to the other substrates. This observation reflects the increasing relevance of adhesion to Y-TZP in clinical practice. While bond strengths to HF-etched glass-matrix ceramics have long been considered the strongest in dental adhesion testing, bonding strengths to physico-chemically conditioned Y-TZP are currently reaching similar values.

Zirconia has the highest flexural strength (900 to 1300 MPa)[29,73] and the highest fracture toughness (9 to 10 MPa/m^2)[60] of all substrates tested in the present study. Bond strengths to Y-TZP increase significantly when the surface is roughened with air-abrasion methods and the use of an MDP-based primer or universal adhesive bonding solution.[9,17,46,63] The hypothesis that chemical bonds form between MDP solutions and Y-TZP substrates was confirmed by Chen et al,[17] using time-of-flight secondary ion mass spectroscopy (TOF-SIMS). When dual-curing resin cements were used on zirconia surfaces that had not been air abraded, Maeda et al[45] measured higher bond strengths when a specific MDP-containing zirconia primer was used, which confirms the chemical bonding ability described by Chen et al.[17] However, micromechanical retention plays a more prominent role than chemical bonding in adhesion to Y-TZP.[4] A 58% lower tensile bond strength was obtained with SBU on intact (19.1 MPa) than on air-abraded zirconia (33.8 MPa), whereas for an MDP-based silane primer (Monobond Plus), the difference was even more significant.[4] Monobond Plus (MBP) is a solution of 4 wt% adhesive monomers (3-trimethoxysilylpropyl methacrylate and MDP) in 96 wt% ethanol.[34,35] Different silane solutions that contain MDP have been shown to increase adhesion to Y-TZP surfaces even after water storage for 150 days with thermocycling.[9]

The fact that PUB was the only adhesive for which the mean µSBS reached the highest ranking of statistical significance among all adhesives for all five CAD/CAM substrates was unanticipated. This performance may have been a result of the higher viscosity of PUB compared to that of other universal adhesives, which might be responsible for better physical properties. PUB is the only adhesive in the present study that contains chlorhexidine (CHX) in a concentration of 0.2%. Cadenaro et al[11] reported that the incorporation of CHX in adhesive blends is detrimental to the physical properties of the adhesives in concentrations >1% CHX. Islam et al[32] reported that adding 0.5% CHX to a commercial primer used in a two-step self-etching adhesive did not result in any statistical difference from the control group for bond strengths and elastic modulus of the bonded interfaces. Therefore, 0.2% CHX in PUB may not affect its physical properties.

One of the limitations of this study is that the use of a resin cement on the intaglio surfaces does not reflect what may occur clinically, where either tooth structure or core build-up material is also bonded to the intaglio surface. The stress patterns developed at the adhesive interface are certainly different when the resin cement is applied to the intaglio surface using a narrow, transparent matrix, such as a Tygon tube.

The presence of MDP in the universal adhesives did not have a significant influence on the microshear bond strength values measured. Although the mean bond strengths of universal adhesives varied widely with the CAD/CAM material used, most universal adhesives reached the highest statistical ranking when applied to air-abraded zirconia. Further studies should focus on the potential of chemical adhesion between the different CAD/CAM materials and the functional monomers used in recent adhesive materials.

CONCLUSIONS

The first null hypothesis was rejected, as the mean microshear bond strengths of universal adhesives varied widely with the CAD/CAM material used. The second null hypothesis was partially rejected, as only one universal adhesive, PUB, reached the highest ranking of statistical significance among all adhesives for all five CAD/CAM materials.

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Clinical relevance: Universal adhesives may be used as primers for air-abraded zirconia. Peak Universal Bond may be recommended as the most consistent universal adhesive in terms of mean µSBS for all the CAD/CAM restorative materials used in this study.