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Impact of Recently Developed Universal Adhesives on Tensile Bond Strength to Computer-aided Design/Manufacturing Ceramics

A Liebermann • J Detzer • B Stawarczyk

Clinical Relevance

Monobond Etch & Prime and Monobond Plus showed comparable tensile bond strength results. Only a few universal adhesive systems provided reliable values for all ceramics tested. Thus, the clinician needs to individualize the selection of the adhesive system for each ceramic.

SUMMARY

Objectives: The aim of this investigation was to test the tensile bond strength (TBS) between different computer-aided-design/manufacturing (CAD/CAM) ceramics after conditioning using different universal adhesive systems and resin composite cement.

Methods and Materials: Substrates of four CAD/CAM ceramics—1) VITABLOCS Mark II, 2) Initial LRF, 3) Celtra Duo, and 4) IPS e.max CAD (N=648, n=162)—were fabricated. VITABLOCS Mark II and Initial LRF were etched using 9% hydrofluoric acid for 60 seconds, Celtra Duo for 30 seconds, and IPS e.max CAD for 20 seconds. Substrates for conditioning using Monobond Etch & Prime were untreated. The following adhesive systems were used: All-Bond Universal (ABU), Clearfil Universal Bond (CUB), G-Multi Primer (GMP), iBond Universal (IBU), Monobond Etch & Prime (MEP), Monobond Plus (MBP), One Coat 7 Universal (OCU), Prime&Bond Active (PBA), and Scotchbond Universal (SBU). Conditioned substrates were bonded using a resin composite cement (Variolink Esthetic DC), thermal cycled (20,000×, 5°C/55°C), and TBS was measured using a universal testing machine. Data were analyzed using univariate analysis with partial eta-squared, Kolmogorov-Smirnov, Kruskal-Wallis, Mann-Whitney *U*, and Spearman-Rho tests (α=0.05).

Results: ABU, MEP, and MBP obtained the significantly highest TBS, while CUB, IBU, and OCO resulted in the lowest, regardless of the CAD/CAM ceramic. SBU showed varying TBS

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results depending on the CAD/CAM ceramic used. ABU, MEP, and MBP showed no impact of CAD/CAM ceramic on TBS values. ABU, GMP, MEP, and MBP showed predominantly cohesive failure types in luting composite, while CUB and OCU demonstrated adhesive failure types.

INTRODUCTION

The ongoing optimization of computer-aided-design/manufacturing (CAD/CAM) technology has supported the further development of dental ceramics, which have seen an increase in popularity because of their esthetic and mechanical properties. Glass ceramics are often used and generally based on silicon dioxide (SiO_2) or similar forms, with the incorporation of other crystals, such as feldspar/leucite, lithium disilicate, or even zirconium dioxide (zirconia).[1] On the basis of various factors, such as fracture toughness, the Weibull modulus, and subcritical crack formation, characterization of individual ceramics is based on initial bond strengths, which can differ significantly between individual glass ceramics. Glass ceramic restorations should preferably be fixed using the adhesive technique.[2]

In adhesive dentistry, with modern, minimally invasive procedures, the treatment team has a wide range of materials and luting procedures available to fix restorations, ranging from modern single-step to multistep systems. The latest generation of universal adhesive systems, for example, raises expectations of a broad range of indications, associated simplifications, and time savings in everyday practice; however, to satisfy the notion of universality, these adhesive systems must meet a wide variety of requirements.

To produce a chemical bond on the glass ceramic surface, hydrofluoric acid etching with subsequent silanization is the method of choice.[3,4] Differences in pretreatment among glass ceramic groups are reflected in their different etching times.[5] To avoid the risk of a possible hydrofluoric acid accident, the Monobond Etch & Prime (Ivoclar Vivadent, Schaan, Liechtenstein) ceramic primer, which combines hydrofluoric acid etching and silanization steps, has been available on the market for some time now. The primer contains the conditioning agent ammonium polyfluoride for etching in addition to a silane system based on trimethoxypropyl methacrylate for silanization. There are currently only a few published *in vitro* studies that have reported good results.[6-9] Most universal adhesive systems, apart from Monobond Etch & Prime, have a similar composition to self-etch bonding systems, including water for dissociation, acid adhesive monomer, bisphenol A-glycidylmethacrylate, urethanedimethacrylate, and hydroxyethylmethacrylate monomers, as well as a solvent.[6] In addition, some available universal adhesive systems already contain a silane other than the 10-methacryloyloxydecyldihydrogenphosphate (MDP) monomer.

The first hydrofluoric acid etching during the pretreatment also increases the available Si-OH groups on the ceramic surface.[2] A functional silane monomer such as λ-methacryloyloxypropyltrimethoxysilane, which is frequently used in dentistry, contains a methacrylate end capable of copolymerizing with the adhesive and/or luting resin composite as well as the silane group to create a covalent bond with the ceramic.[10] Two factors, water and low pH, present a risk for a hydrolysis process in the silane. For this reason, the latest-generation silane is dissolved in a mildly corrosive and anhydrous solution, resulting in a relatively long shelf life[11]; however, recent studies have clearly shown that the step of silanizing the restoration separately using a conventional silane after etching results in significantly better adhesion values.[10,12,13] Unfortunately, there are currently little data available in the literature about the different universal adhesive systems.

For this reason, this investigation analyzed the influence of various universal adhesive systems on the bond to CAD/CAM glass ceramics by measuring tensile bond strength (TBS) after thermal aging. The tested null hypotheses were as follows:

1. the tested universal adhesive systems show no comparable TBS results to the CAD/CAM ceramics, and
2. the CAD/CAM ceramics show no impact on the TBS results.

METHODS AND MATERIALS

A total of 648 rectangular specimens were produced from CAD/CAM ceramic blocks (n=162/ceramic material; n=18 for each of the 36 subgroups) under standardized conditions. Four different ceramic materials and nine diverse universal adhesive systems were analyzed (Table 1).

All of the CAD/CAM ceramic blocks were cut into five standardized ceramic discs (thickness: 2.5 mm, diameter: 18 mm) using a water-cooled (Secotom-50, Struers, Ballerup, Denmark), automatic diamond saw (Diamond Cut-off Whell M1D13, Struers).

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Table 1: *Materials, Abbreviations, Application Steps, Lot No., Manufacturers, and Compositions*

Ceramic Material	Lot No.	Manufacturer	Composition (Oxides in wt%)
VITABLOCS Mark II	JO17EC4I14MO	VITA Zahnfabrik, Bad Säckingen, Germany	SiO_2 56%-64%, Al_2O_3 20%-23%, Na_2O 6%-9%, K_2O 6%-8%, CaO 0.3%-0.6%, TiO_2 0%-0.1%
Initial LRF	1610181	GC Europe, Leuven, Belgium	No information
Celtra Duo	18026884	Dentsply Sirona, Bensheim, Germany	SiO_2, Li_2O, ZrO_2 ($\approx$10%), P_2O_5, Al_2O_3, K_2O, CeO_2, pigments
IPS e.max CAD	V50567	Ivoclar Vivadent, Schaan, Liechtenstein	SiO_2 57%-80%, Li_2O 11%-19%, K_2O 0%-13%, P_2O_5 0%-11%, ZrO_2 0%-8%, ZnO 0%-8%, Al_2O_3 0%-5%, MgO 0%-5%, coloring oxides 0%-8%

Universal Adhesives	Abbreviation	Lot No.	Manufacturer	Composition
All-Bond Universal	ABU	1600005525	Bisco Dental, Schaumburg, IL, USA	MDP, Bis-GMA, HEMA, ethanol
Clearfil Universal Bond	CUB	4J0025	Kuraray Noritake Dental Inc, Okayama, Japan	MDP, Bis-GMA, HEMA, hydrophilic aliphatic dimethacrylate, colloidal silica, silane coupling agent, dl-camphor quinone, ethanol, water
G-Multi Primer	GMP	1610071	GC Europe	Silane, MDP, MDTP
iBond Universal	IBU	10025	Heraeus Kulzer, Hanau, Germany	Acetone-water–based solution of light-activating methacrylate monomers
Monobond Etch&Prime	MEP	V26292	Ivoclar Vivadent	Silane system based on trimethoxypropylmethacrylate, ammoniumpolyfluoride, alcohol, water, food coloring, Fast Green
Monobond Plus	MBP	W02150	Ivoclar Vivadent	Phosphoric acid methacrylate, silane methacrylate, sulfide methacrylate, alcohol
One Coat 7 Universal	OCU	H39695	Coltène/Whaledent, Altstätten, Switzerland	MDP, methacrylate, photoinitiators, ethanol, water
Prime&Bond Active	PBA	1609000479	Dentsply Sirona, Bensheim, Germany	Phosphoric acid–modified acrylic resin, multifunctional acrylate, bifunctional acrylate, acid acrylate, isopropanol, water, initiators, stabilisators
Scotchbond Universal	SBU	648274	3M, Seefeld, Germany	MDP, dimethacrylate polymers, Vitrebond copolymer, filler, ethanol, water, initiators, silane

Abbreviations: Al_2O_3, aluminum trioxide; Bis-GMA, bisphenol glycidyl methacrylate; CeO_2, cerium dioxide; HEMA, hydroxyethylmethacrylate; K_2O, potassium oxide; Li_2O, lithium oxide; MDP, 10-. methacryloyloxydecyl hydrogen phosphate; MgO, magnesium oxide; Na_2O, natrium oxide; P_2O_5, phosphorus pentoxide; SiO_2, silicon dioxide; TiO_2, titanium dioxide; ZnO, zinc oxide; ZrO_2, zirconium dioxide.

Compared with the other three glass ceramics, IPS e.max CAD required a subsequent crystallization process. These specimens were crystallized in a ceramic furnace (Programat EP 5000, Ivoclar Vivadent) at 840°C, according to the manufacturer's instructions. The polishing of one of the two ceramic specimen surfaces was carried out using a mechanical polishing machine (Abramin, Struers) under constant water cooling and at a constant pressure of 3 bar using silicon carbide foils (SiC Foil, Struers), beginning with P500 up to P1200. The polished surfaces were etched using 9% hydrofluoric acid (Ultradent Products, South Jordan, UT, USA), with the etching time in accordance with the manufacturer's instructions: VITABLOCS Mark II and Initial LRF were etched for 60 seconds, Celtra Duo for 30 seconds, and IPS e.max CAD for 20 seconds. To preclude the risk of overetching, each specimen was etched separately and timed by a stopwatch. After etching, the hydrofluoric acid was removed with demineralized water and subsequent purification in an ultrasonic bath (Transistor/Ultrasonics, L & R, Kearny, NJ, USA). Removal was carried out for one minute with alcohol (80% ethanol; Otto Fischer, Saarbrücken, Germany) and cleaned again after-

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ward with demineralized water. Finally, the surface was air dried for 20 seconds.

An exception in the four ceramic groups was Monobond Etch & Prime, for which hydrofluoric acid etching and cleaning in the ultrasonic bath were not needed.

To complete all of the specimens, pretreatment of the etched ceramic surface using the universal adhesive systems was carried out according to the manufacturers' instructions. The individual application steps for the nine universal adhesive systems are listed below. Each adhesive system was performed for each of the four ceramic materials.

1. All-Bond Universal (ABU): Apply one to two coats of porcelain primer with a microbrush for 30 seconds and air dry. Apply ABU, air dry for five seconds, and light cure for 10 seconds.
2. Clearfil Universal Bond (CUB): Mix 1:1 bond and DC activator, apply using a microbrush, and massage in for 10 seconds. Air dry for five seconds and light cure for 10 seconds.
3. G-Multi Primer (GMP): Apply with microbrush and air dry for five seconds.
4. iBond Universal (IBU): Apply iBond Ceramic, evaporate for 20 seconds, and air dry. Apply iBond Universal, massage for 20 seconds, air dry, and light cure for 10 seconds.
5. Monobond Etch & Prime (MEP): Apply using microbrush, rub with light pressure for 20 seconds, and leave for 40 seconds. Rinse with distilled water until the green color is removed, and air dry for 10 seconds.
6. Monobond Plus (MBP): Apply using microbrush, leave for 60 seconds, and air dry.
7. One Coat 7 Universal (OCU): Apply using microbrush, rub with light pressure for 20 seconds, and air dry for five seconds. Mix 1:1 OCU and One Coat 7 Activator for five to 10 seconds and apply to the surface, air dry for five seconds, and light cure for 10 seconds.
8. Prime&Bond Active (PBA): Apply the silane and air dry for five seconds. Mix 1:1 PBA and Dentsply Self-Cure Activator for one to two seconds. Apply to ceramic for 20 seconds, air dry for five seconds, and light cure for 10 seconds.
9. Scotchbond Universal (SBU): Mix 1:1 SBU and Scotchbond Universal Activator for five seconds. Apply to ceramic, massage in for 20 seconds, air dry for five seconds, and light cure for 10 seconds.

ABU and IBU needed an additional ceramic primer, and CUB, OCU, PBA, and SBU required an additional activator. The specimens were light cured (Elipar S10, 3M, Seefeld, Germany) with a light intensity of 1200 mW/cm^2.

Thereafter, an acrylic cylinder (SD Mechatronics, Feldkirchen-Westerham, Germany) was bonded to the ceramic surfaces. This acrylic cylinder (inner diameter: 2.9 mm, wall thickness: 0.1 mm, height: 10 mm) was positioned in the middle of the ceramic surface, fixed, and gently filled with dual-curing resin composite cement (Variolink Esthetic DC, Ivoclar Vivadent, Lot No. V16615). While filling the cylinder, the syringe initially rested directly on the ceramic and was slowly pulled up during the filling process to avoid blistering. If necessary, excess material was carefully removed with a microbrush, followed by uniform light curing from all four sides for a total of 20 seconds. The bonded specimens were stored in a black storage box with distilled water after curing, while the remaining specimens of the respective group were completed.

All specimens were stored for 24 hours at 37°C in distilled water in an incubator (Heracell 150, Kulzer, Hanau, Germany) and then subjected to thermal cycling (20,000×, 5°C/55°C; THE-1100 thermal cycler, SD Mechatronics). The specimens underwent a relaxation time of two hours before being further measured by TBS test.

The measurement of TBS was carried out with a universal testing machine (1445 Zwick/Roell, Zwick GmbH & Co. KG, Ulm, Germany). For the standardized method, the settings were adjusted on the machine (forward speed: 5 mm/min; force: 1 N). The required tensile force was determined using a 500-N load cell, which was connected to the collets of the testing machine. The calculation of tensile strength was analyzed using the following equation: TBS (MPa) = fracture load (N)/bonding area (mm^2). The fracture types were analyzed macroscopically. Three types were distinguished: 1) adhesive fracture (smooth fracture between luting resin composite and CAD/CAM ceramic), 2) cohesive fracture luting resin composite (fracture within the luting resin composite), and 3) cohesive fracture ceramic (fracture within the CAD/CAM ceramic). After failure type analysis, representative specimens were sputter coated with gold-palladium for 100 seconds with a sputter coater (SCD 005, Bal-Tec, Liechtenstein) and analyzed using scanning electron microscopy (SEM; LEO 1430, Zeiss, Germany) operating at 10 kV with a working distance of 12.7-12.9 mm.

The Kolmogorov-Smirnov test was used for testing the normality of the data distribution.

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Table 2: *Descriptive Statistics (Mean ± Standard Deviation) of TBS Values for Each CAD/CAM Ceramic and Universal Adhesive System (UAS), Separately*

	ABU	CUB	GMP	IBU
VITABLOCS Mark II	35.7 ± 4.2^{aC}	13.8 ± 9.3^{bA*}	36.4 ± 6.7^{cC}	13.8 ± 11.1^{cA}
Initial LRF	36.7 ± 3.2^{aD}	0.0 ± 0.0^{aA*}	34.8 ± 5.0^{bcD}	9.6 ± 12.8^{abcB*}
Celtra Duo	35.0 ± 5.4^{aCD*}	21.5 ± 6.5^{cB}	30.2 ± 7.9^{bC}	2.7 ± 3.8^{abA}
IPS e.max CAD	34.0 ± 8.2^{aD*}	1.3 ± 5.4^{aA}	23.9 ± 8.9^{aC}	1.8 ± 5.4^{aA*}

^{abc} Letters indicate significant differences between the CAD/CAM ceramics within the UAS.
^{ABCD} Letters indicate significant differences between the UAS within the CAD/CAM ceramics.
* Not normally distributed.

Univariate analysis of variance with partial eta-squared, Kruskal-Wallis, and Mann-Whitney U-tests were used to evaluate the data and to determine the significant differences between the groups. In the Spearman-Rho test, a correlation between tensile strength and fracture images was evaluated. The Statistical Package for the Social Sciences v. 24 (SPSS Inc, Chicago, IL, USA) was used (α=0.05).

RESULTS

The Kolmogorov-Smirnov test indicated a higher rate of violation of the normality assumption for TBS (99%; Table 2), which might also be attributed to single statistical outliers. Therefore, for all further statistical tests, the nonassumption of normal distribution was used. The highest impact on TBS was exerted by the universal adhesive system (partial eta-squared η_P^2=0.711, p<0.001), followed by interactions between the universal adhesive system and CAD/CAM ceramic (η_P^2=0.256, p<0.001), as well as solely by the CAD/CAM ceramic (η_P^2=0.202, p<0.001).

The univariate analysis interaction (universal adhesive system vs CAD/CAM ceramic) was significant (p<0.001). Therefore, the fixed effects of the universal adhesive system and CAD/CAM ceramic could be compared directly, as the higher-order interactions were found to be significant. Consequently, several different analyses were computed, which were divided by levels of universal adhesive system and CAD/CAM ceramic, depending on the hypothesis of interest.

Impact of the Universal Adhesive System on the TBS Values

ABU, MEP, and MBP obtained the significantly highest TBS, regardless of the CAD/CAM ceramic (p<0.001). In VITABLOCS Mark II, Initial LRF, Celtra Duo, GMP, and PBA were in the same values range with the universal adhesive systems above (p>0.05).

In specimens bonded on IPS e.max CAD, PBA showed significantly lower values than GMP (p<0.001), while GMP showed lower values than ABU, MEP, and MBP (p<0.001). IBU, OCU, and CUB revealed the lowest TBS (p<0.001). In contrast, ABU combined with Celtra Duo had the highest TBS, although it was in the value range with the lowest TBS when combined with IPS e.max CAD. The detailed significant differences are shown using capital letters in Table 2.

Impact of CAD/CAM Ceramic on the TBS Values

In ABU (p=0.534), MEP (p=0.708), and MBP (p=0.273), no impact of CAD/CAM ceramic on TBS values was observed. Within specimens conditioned with CUB, the highest TBS values were tested on Celtra Duo ceramic, followed by VITABLOCS Mark II (p<0.001). Specimens bonded on Initial LRF showed no bond (0 MPa).

Within GMP and IBU, the highest TBS showed specimens bonded on VITABLOCS Mark II, followed by Initial LRF and Celtra Duo. The lowest TBS was observed in combination with IPS e.max CAD (p<0.001).

Within OCU, significantly higher TBS was observed for VITABLOCS Mark II and Celtra Duo compared with IPS e.max CAD and Initial LRF (p<0.001).

Within PBA, specimens bonded on IPS e.max CAD showed the significantly lowest TBS compared with the remaining tested ceramics (p<0.001).

Within SBU, the highest TBS was found with Celtra Duo and the lowest IPS e.max CAD (p<0.001).

TBS showed a significant positive Pearson correlation with fracture type (r^2=–0.656, p<0.001). With an improvement in TBS values, the number of

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Table 2: *Descriptive Statistics (Mean ± Standard Deviation) of TBS Values for Each CAD/CAM Ceramic and Universal Adhesive System (UAS), Separately (ext.)*

	MEP	MBP	OCU	PBA	SBU
VITABLOCS Mark II	33.4 ± 6.3^{aC}	31.9 ± 6.4^{aBC}	10.2 ± 8.4^{bA}	30.6 ± 9.5^{bBC}	24.6 ± 5.5^{bcB}
Initial LRF	$35.8 \pm 6.4^{aD*}$	35.9 ± 2.5^{abD}	$0.02 \pm 0.1^{aA*}$	$31.3 \pm 8.6^{bD*}$	$19.3 \pm 5.6^{bC*}$
Celtra Duo	35.2 ± 3.5^{aCD}	$33.8 \pm 8.6^{abCD*}$	16.4 ± 12.1^{bB}	33.7 ± 6.6^{bC}	29.3 ± 7.9^{cC}
IPS e.max CAD	$33.9 \pm 9.7^{aD*}$	32.5 ± 7.1^{aD}	$2.0 \pm 6.7^{aA*}$	15.4 ± 12.2^{aB}	$7.8 \pm 11.9^{aAB*}$

cohesive fracture types increased. ABU, GMP, MEP, and MBP showed predominantly cohesive failure types in luting resin composite, while CUB and OCU fractured adhesively (Table 3; Figures 1 and 2).

SEM Images

SEM images of the different fracture types were observed, and both cohesive fracture types are presented in Figures 1 and 2.

Distinctly irregular surfaces with partly visible streaks and minor structural defects were detected. Figure 1 shows the interface between the universal adhesive structures with the remaining luting composite of cohesive failure within the luting material, whereas Figure 2 represents the cohesive failure within the ceramic surface with visible ceramic structures.

DISCUSSION

The large number of glass ceramics and the wide range of different adhesives, as well as the new development of universal adhesive systems for ceramic pretreatment, make clinical decision making more difficult. In this investigation, the choice of the adhesive system and glass ceramic showed a significant impact on TBS values. Therefore, both null hypotheses were rejected.

Each bond generally consists of three sides: 1) substrate, 2) luting material, and 3) tooth surface. However, the present TBS test can investigate only one side of bonding, which was the bond between the glass ceramic with an applied universal adhesive and the luting material. After testing geometrical specimens, the standard procedure is to check clinic-related geometries, such as the crown TBS test, and should be the next step for future investigations.

Generally, the highest TBS was observed after glass ceramic conditioning using ABU, MEP, and MBP. These results were independent of the CAD/CAM glass ceramics used. Similar values with the TBS test have been obtained in other studies.[12,14] A prerequisite for a good bond between luting resin composite and glass ceramic is the creation of a microretentive surface, which offers high wettability for the subsequently applied adhesive.[2,15-17] MEP combines the hydrofluoric acid etching and silanization steps and contains ammonium polyfluoride for the etching effect and trimethoxypropyl methacrylate for silanization. When comparing the etching patterns between MBP and MEP, MEP generally resulted in a less roughened surface than the hydrofluoric acid etching.[6] MBP showed good long-term bonding properties on different restorative materials.[6,7,12,14] In this study, MEP showed comparable results (mean and standard deviation) to MBP, regardless of the glass ceramic used (see Table 2; VITABLOCS Mark II: MEP 33.4±6.3, MBP 31.9±6.4; Initial LRF: MEP 35.8±6.4, MBP 35.9±2.5; Celtra Duo: MEP 35.5±3.5, MBP 33.8±8.6; IPS e.max CAD: MEP 33.9±9.7, MBP 32.5±7.1). These results have been confirmed by another study that performed testing with IPS e.max CAD (MBP 26.53±6.33 MPa, MEP 23.52±8.41 MPa)[7]; however, very little clinical data on MEP are currently available. One study showed satisfactory results for this adhesive after six months of clinical testing,[8,9] but further studies are needed to confirm these outcomes. Currently, there have been no investigations on MEP with other glass ceramics, except in combination with IPS e.max CAD. Thus, direct comparisons are not possible.

The present study did not investigate the topography of the surfaces after the respective etching process but instead examined the bond between luting materials and the CAD/CAM ceramic surface as well as the fracture patterns. In the present study, despite different etching patterns, MEP and MBP showed very similar results regarding the two parameters including four diverse CAD/CAM glass ceramics. Ammonium polyfluoride, as included in MEP, is a known alternative to established hydrofluoric acid etching. With the help of an SEM investigation in another study, it was found that although ammonium polyfluoride can exert an influence on the ceramic surface, the microretention formed is not comparable to that formed by hydrofluoric acid. The test specimens etched with ammo-

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Table 3: *Relative Frequency of Failure Types (%) With 95% Confidence Intervals (95% CI) for Each CAD/CAM Ceramic and Universal Adhesive System, Separately*

UAS	Adhesive Failure	Cohesive Failure, Ceramic	Cohesive Failure, Luting Composite
VITABLOCS Mark II			
ABU	0 (0; 18)	0 (0; 18)	100 (80; 100)
CUB	100 (80; 100)	0 (0; 18)	0 (0; 18)
GMP	0 (0; 18)	5.56 (0; 28)	94.44 (71; 100)
IBU	77.78 (51; 94)	0 (0; 18)	22.22 (5; 48)
MEP	0 (0; 18)	11.11 (0; 35)	88.89 (64; 99)
MBP	0 (0; 18)	5.56 (0; 28)	94.44 (71; 100)
OCU	94.44 (71; 100)	0 (0; 18)	5.56 (0; 28)
PBA	38.89 (16; 65)	0 (0; 18)	61.11 (34; 83)
SBU	88.89 (64; 99)	0 (0; 18)	11.11 (0; 35)
Initial LRF			
ABU	0 (0; 18)	0 (0; 18)	100 (80; 100)
CUB	100 (80; 100)	0 (0; 18)	0 (0; 18)
GMP	5.56 (0; 28)	0 (0; 18)	94.44 (71; 100)
IBU	77.78 (51; 94)	5.56 (0; 28)	16.67 (2; 42)
MEP	0 (0; 18)	0 (0; 18)	100 (80; 100)
MBP	0 (0; 18)	0 (0; 18)	100 (80; 100)
OCU	100 (80; 100)	0 (0; 18)	0 (0; 18)
PBA	22.22 (5; 48)	0 (0; 18)	77.78 (51; 94)
SBU	100 (80; 100)	0 (0; 18)	0 (0; 18)
Celtra Duo			
ABU	0 (0; 18)	0 (0; 18)	100 (80; 100)
CUB	94.44 (71; 100)	0 (0; 18)	5.56 (0; 28)
GMP	16.67 (2; 42)	0 (0; 18)	83.33 (57; 97)
IBU	100 (80; 100)	0 (0; 18)	0 (0; 18)
MEP	0 (0; 18)	0 (0; 18)	100 (80; 100)
MBP	0 (0; 18)	0 (0; 18)	100 (80; 100)
OCU	83.33 (57; 97)	0 (0; 18)	16.67 (2; 42)
PBA	0 (0; 18)	0 (0; 18)	100 (80; 100)
SBU	44.44 (20; 70)	0 (0; 18)	55.56 (29; 79)
IPS e.max CAD			
ABU	0 (0; 18)	0 (0; 18)	100 (80; 100)
CUB	100 (80; 100)	0 (0; 18)	0 (0; 18)
GMP	50 (25; 74)	0 (0; 18)	50 (25; 74)
IBU	94.44 (71; 100)	0 (0; 18)	5.56 (0; 28)
MEP	5.56 (0; 28)	0 (0; 18)	94.44 (71; 100)
MBP	0 (0; 18)	0 (0; 18)	100 (80; 100)
OCU	100 (80; 100)	0 (0; 18)	0 (0; 18)
PBA	66.67 (39; 87)	0 (0; 18)	33.33 (12; 60)
SBU	88.89 (64; 99)	0 (0; 18)	11.11 (0; 35)

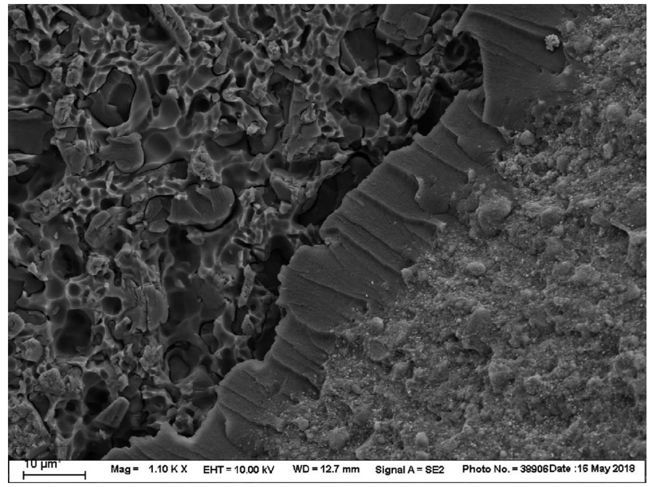

Figure 1: *SEM picture of cohesive fracture type within the resin luting composite (1100x magnification).*

nium polyfluoride showed a relatively smooth homogeneous surface, whereas those etched by hydrofluoric acid gave an irregular character, characterized by a three-dimensional lattice with pores and furrows.[17] Further studies are required to confirm the extent to which the new adhesive behaves under clinical stress over a longer period of time. A striking feature of the present study was the combination of the ceramic Initial LRF with the universal adhesive CUB. In this investigation, debonding (0.00 MPa) occurred in all 18 test specimens during thermocycling. Even with the universal adhesive, OCU almost exclusively had a loss of the bond (0.02 MPa) after aging occurred. To the authors' best knowledge, there are no studies on ceramic LRF or any detailed information on its composition, which makes it

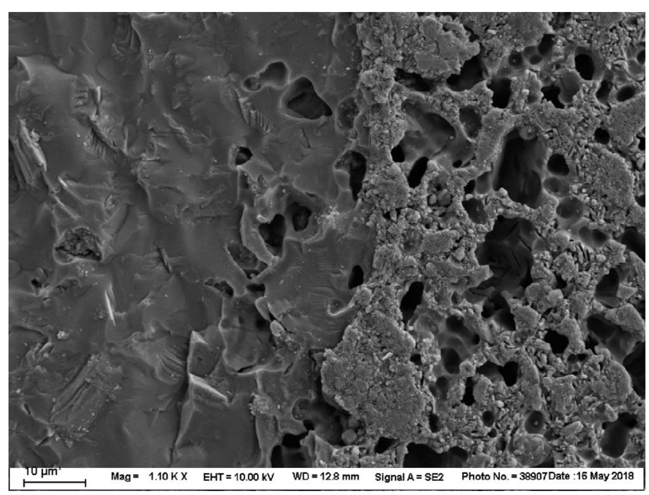

Figure 2: *SEM picture of cohesive fracture type within the glass ceramic (1100x magnification).*

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difficult to make comparisons with this ceramic. The question arises whether undesirable chemical interactions occurred in these cases and whether the composition of the adhesive, such as the viscosity, played a role, or if the luting cement used had an influence on the adhesion.[18] Further investigation of this connection is thus of great importance.

Another interesting point is the completely controversial performance of the silane-free adhesive ABU,[11,19] which, in the presented results, achieved among the highest adhesion values with all ceramic materials. In contrast, there are two studies that reported an initial strength but reported debonding in all specimens during thermal cycling.[12,19] Separate application of a corresponding special silane should be carried out when luting to glass ceramics. This was used in the current study, but such a separate application was not clearly described in either of the studies mentioned above, which could be a possible explanation for the loss of adhesion in all specimens. After closer examination of the silane content, silane-containing and silane-free universal adhesives with separate silanization were generally able to achieve comparable bond strengths in the present study. Nevertheless, the existence of silane in an adhesive should not automatically imply good adhesion to the material, as presented by the example of the CUB tested, which achieved no satisfactory strength values, with the exception of Celtra Duo. In addition to its technical sensitivity, CUB could also be responsible for its results due to the short processing time of 90 seconds and the mixture with an activator. In addition to CUB, OCU, PBA, and SBU also required an additional activator in order to be used with the dual-curing luting cement. Activators ensure chemically initiated curing with co-initiators, such as the sulfinic salt solutions contained in them.

Herein, the luting resin composite Variolink Esthetic DC was chosen to focus on the influence of the universal adhesive systems on the TBS of the four different ceramics. Compatibility with other manufacturers' products may have had little effect, but the necessary additional recommended products, such as activators or separate silanes, have been used. Since Variolink Esthetic is considered to be an "amine-free" luting resin composite and because the polymerization is based on hydroperoxide and thiourea, it would theoretically be possible to dispense it with an activator. Many studies demonstrated that an additional mixture with an activator dilutes the composition of some adhesives in such a way that adhesion is adversely affected.[20,21] In the case of CUB and OCU, both achieved significantly lower adhesion despite the use of an activator, which could explain the present results. This claim is consistent with findings of a recent study,[22] in which the added activator influenced the material-dependent micro-shear bond strength. In connection with the investigated OCU adhesive and its performance in the present study, silane and the activator could also have led to these results. In this case, the fact that this adhesive does not contain silane or a separate silane application appears to be very noticeable. The missing mediator function of the silane for chemical adhesion to the ceramic surface as well as the thinning of the adhesive could have influenced the results. Similar observations for OCU have already been reported.[22,23]

In summary, only one resin composite cement, Variolink Esthetic DC, was examined in this study. To make sweeping statements about the universal adhesives, further investigations with additional resin composite-based materials are necessary.

The etching of the glass ceramic is dependent on different factors, such as etching gel type, the etching duration, the ceramic microstructure, and its composition.[24-26] The ceramics investigated in this study were monochromatic feldspar ceramics (VITABLOCS Mark II), leucite-reinforced feldspar ceramics (Initial LRF), zirconium oxide–reinforced lithium-silicate ceramics (Celtra Duo), and lithium disilicate ceramics (IPS e.max CAD), with different percentages of etchable glass content, in which the etching time, except for MEP, was taken into account. Hydrofluoric acid leaves etching patterns on the ceramic surface, and the glass and crystalline phases are partially dissolved out, while a crystalline structure is exposed.[27-29] In addition to an increase in surface area, there is an increase in the surface roughness and wettability.[25,26] The generally good results of VITABLOCS Mark II in the present study could have been influenced by deep microstructures and the resulting large mechanical anchoring. A previous study confirmed that bond strength essentially depends on the adhesive system used and that the ceramic exerts a much smaller influence.[9] As with the adhesives ABU and MBP, MEP was independent from ceramic type in this study.

The results of the present study, which demonstrated that the bonding on Celtra Duo had a higher TBS compared with IPS e.max CAD, were also reported in another study.[16] The functional phosphoric acid group contained in the MDP molecule has the ability to undergo ionic interactions with the zirconia present with metal oxides, such as the glass

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ceramic in this case.[30,31] The proportion of zirconium oxide in Celtra Duo was higher (approximately 10%) compared with IPS e.max CAD (0%-8%), which leads to suspicions of different adhesion values between these two materials. The results in this study showed higher bonding for adhesives, which included MDP molecules such as ABU, CUB, and GMP, or phosphoric acid methacrylates, such as MBP. The universal adhesive system SBU, which included MDP, showed controversial results but demonstrated differences in adhesion between the CAD/CAM ceramics tested. For example, high TBS values were found for Celtra Duo, while low TBS values were found for IPS e.max CAD. After conditioning with IBU and OCU or SBU for some CAD/CAM ceramics, the lower bonding values compared with the other groups could be explained by the fact that despite the presence of acidic monomers (4-META or MDP), influencing factors, such as a low pH, lack of silane, and admixing with an activator, could have already had an impact.

CUB, IBU, and OCU, which achieved the lowest adhesion values, showed almost exclusively adhesive fractures. This can be interpreted as adhesive failure, since the flexural strengths of the ceramic as well as the luting resin composite used were stronger than TBS between the ceramic and universal adhesive system. ABU achieved the best adhesion values in the present study and showed 100% cohesive fractures in the luting resin composite for all four ceramics. The adhesive systems GMP, MEP, MBP, and PBA also showed predominantly cohesive fractures within the composite. The different interfaces of universal adhesive/luting material were clearly visible, especially the fractured ceramic surface (Figures 1 and 2). It can be assumed that the bond strength is higher than the intrinsic strength of the substrates or resin composite cement and therefore results in cohesive fracture types after testing. This means that the high bond strength values in the case are supported by the cohesive fractures. Thus, the true bond strength value may actually be higher. In contrast, the SBU, CUB, OCU, and IBU adhesives mainly led to adhesive fractures, indicating lower bond strengths. Thus, fracture types are created directly in the interface and provide the true bond strength values.

To gain knowledge about an expected *in vitro* behavior, thermal aging by means of water storage or thermal load change was used in the present study and demonstrated to be a common method. The longevity of the bond between the luting resin composite and the ceramic could be affected by storage times and conditions that mimic oral conditions.[32] It has been shown that hydrolysis processes within the resin composite occur during the storage of resin composites in water[33]; however, a clinical trial with a controlled standardized study design should be used to evaluate the long-term clinical performance as well.

CONCLUSION

Within the limitations of this *in vitro* study, the following can be concluded:

1. ABU, MBP, and MEP showed the highest TBS results, independent of the ceramic tested.
2. MBP and MEP presented similar stability outcomes concerning TBS values.
3. Not all universal systems can be used for each glass ceramic. SBU, for example, showed different results for the glass ceramics tested.
4. The use of universal adhesives combined with ceramic primers or activators is technique sensitive.

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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.

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