https://doi.org/10.4047/jap.2021.13.5.292

The effect of silane and universal adhesives on the micro-shear bond strength of current resin-matrix ceramics

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PURPOSE. The aim of this *in vitro* study was to evaluate the effect of silane and universal adhesive applications on the micro-shear bond strength (μSBS) of different resin-matrix ceramics (RMCs). **MATERIALS AND METHODS.** A total of 120 slides (14 × 12 × 1 mm) were produced from 5 different RMC materials (GC Cerasmart [GC]; Brilliant Crios [BC]; Grandio blocs [GB]; Katana Avencia [KA]; and KZR-CAD HR 2 [KZR]) and sandblasted using 50 μm Al_2O_3 particles. Each RMC material was divided into six groups according to the surface conditioning (SC) method as follows: control (G1), silane primer (G2), silane-free universal adhesive (G3), silane-containing universal adhesive (G4), silane primer and silane-free universal adhesive (G5), and silane primer and silane-containing universal adhesive (G6). Three cylindric specimens made from resin cement (Bifix QM) were polymerized over the treated surface of each slide (n = 12). After thermal cycling (10000 cycles, 5 - 55°C), μSBS test was performed and failure types were evaluated using a stereomicroscope. Data were analyzed using 2-way ANOVA and Tukey tests (α = .05). **RESULTS.** μSBS values of specimens were significantly affected by the RMC type and SC protocols ($P < .001$) except the interaction ($P = .119$). Except for G2, all SC protocols showed a significant increase in μSBS values ($P < .05$). For all RMCs, the highest μSBS values were obtained in G4 and G6 groups. **CONCLUSION.** Only silane application did not affect the μSBS values regardless of the RMC type. Moreover, the application of a separate silane in addition to the universal adhesives did not improve the μSBS values. Silane-containing universal adhesive was found to be the best conditioning method for RMCs. [J Adv Prosthodont 2021;13:292-303]

KEYWORDS
Adhesives; CAD-CAM; Resin cements; Silanes

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Received June 1, 2021 /
Last Revision September 24, 2021 /
Accepted October 18, 2021

This study was partically supported by VOCO GmbH, Cuxhaven, Germany, they kindly supplied Grandio Blocs CAD-CAM blocks and Bifix QM self-etch resin cement.

INTRODUCTION

Computer-aided design and computer-aided manufacturing (CAD-CAM) technology has been integrated into the field of dentistry in order to fabricate du-

pISSN 2005-7806 • eISSN 2005-7814

rable dental materials into restorations that better simulate the appearance of natural teeth, and at the same time reduce the manufacturing period and remove any technician-related inaccuracies.[1,2] The rapid advancements in CAD-CAM technology, along with the ever-increasing patients' esthetic expectations, have both led to the emergence of new esthetic, non-metallic restorative materials that possess different mechanical and optical properties.[1,3,4]

These esthetic restorations can be mainly divided into dental ceramics and composite resins. In comparison to composite resins, dental ceramics have greater durability, better mechanical properties, better biocompatibility, higher discoloring resistance, and superior esthetics.[5,6] However, dental ceramics are lacking in chipping resistance, which makes them prone to fracture.[7] Composite resins cause less abrasion to the opposite teeth when compared to dental ceramics.[5] Additionally, restorations made from resin composite blocks usually take less time to fabricate, cause little damage to the burs during fabrication, have a relatively simpler finishing and polishing process, and are easier to repair.[5,8] The different advantages of each material over the other have sparked the idea of combining both materials into one hybrid dental material.[3,9,10] These materials called resin-matrix ceramics (RMCs) which contain a dominant ceramic network that is reinforced by a cross-linked polymeric matrix.[11]

Among the most important factors that define and affect the properties of RMCs are: the polymerization technique, microstructure, the composition of the resin matrix, and the composition and particle size of the filler material.[12] The constant advancements in manufacturing technologies have allowed the continuous development of new RMC materials.

The adhesive connection between dental restorative materials and the adhesive cement plays a vital role in extending the lifespan and clinical success of the restoration.[5,7] However, achieving good adhesion between abutment teeth and restorative materials is one of the major challenges in modern prosthodontics. The correlation between weak adhesive connection and different mechanical and biological complications has been well-established in the literature.[13-15] Several surface conditioning (SC) methods have been suggested to modify the restoration surface and increase its porosity, in order to increase the chemical and micromechanical retention, thus enhancing the connection between the resin cement and the restorative material.[16,17]

The effect of different SC methods such as hydrofluoric acid etching, sandblasting with aluminum oxide (Al_2O_3) particles, tribochemical silica coating, laser irradiation, and silanization on the adhesive connection between resin cements and the restorative materials has been examined in the literature.[5,7,18-21] The SC method of choice is mainly dependent on the characteristics of the restorative material's microstructure.[7,18,22] The industrial polymerization of CAD-CAM RMC materials results in high degree of conversion (up to 96%), which in turn increases the intensity of cross-links. Therefore, there are too few free monomers on the restoration's surface to copolymerize with the monomers of the resin cement. This is the main factor that necessitates the performing of surface conditioning prior to cementation.[18]

The majority of manufacturers that produce RMC blocks and disks suggest the use of sandblasting with 25 - 50 μm Al_2O_3 particles, followed by silanization in order to increase the adhesive connection between the cement and the restoration. The combined application of appropriate surface roughening technique and suitable primer/bonding agent is known to significantly increase the strength of adhesive connection between the resin cement and the restoration surface. However, there is no consensus regarding the effectiveness of using silane with the universal adhesives as a primer with RMC materials.[18,19,23]

The application of a silane after the surface pretreatment has been suggested to increase the bond strength by promoting adhesion with silica-based materials through the hydroxyl groups available in its composition.[24] Also, the organofunctional monomeric ends of silane also enable the reaction with the methacrylate groups in the resin cement and to the polymer content of RMCs. In addition to the chemical bonds, silane can wet the treated surface, which enhances the resin ability to penetrate into the microporosities.[25-28] As an alternative to silane primers, universal adhesives can be used to form a chemical-based bond to the resin-matrix parts of RMCs. Ion-

https://doi.org/10.4047/jap.2021.13.5.292

ic interactions with the filler ceramic material through acid groups are also possible with universal adhesives since they contain such groups. Bonds to the resin-matrix can be produced through three methods; hydrogen bonds which have limited efficiency, the formation of an interpenetrating network, and the formation of strong, covalent bonds between universal adhesive monomers and double bonds still available in the substrate of the resin-matrix.[19,29-31] The incorporated silane coupling agent in the composition of universal adhesives may provide additional adhesion to the silica-based inorganic fillers.[32] However, the effectiveness of the incorporated silane is still under investigation.[32-35]

Some manufacturers of new universal adhesives claim that there is no need for a separate silane primer step to obtain reliable bonding.[36,37] Nevertheless, some studies have showed a clear effect of using a separate silane step on bond strength, regardless of using silane-containing or silane-free universal adhesives.[38] However, there is no consensus regarding the optimal SC technique, and this subject is still under research.

To the best knowledge of the authors, there is limited information about the effect of SC with silane or/and universal adhesives on the microshear bond strength (μSBS) of different RMC materials. Therefore, the purpose of this study was to assess the effect of silane and universal adhesives on the μSBS of current RMCs. The first null hypothesis tested in this study was that the application of different SC methods would not affect the μSBS between the restoration surface and the resin cement. The second null hypothesis was that the change in RMC material would not affect the μSBS between the restoration surface and the resin cement.

MATERIALS AND METHODS

The experimental design and properties of materials used in this study are presented in Fig. 1 and Table 1. For a total of 120 slides, 24 slides were obtained from each of the five following CAD-CAM RMC blocks (KZR-CAD HR 2 [KZR], Katana Avencia [KA], Grandio Blocs [GB], Brilliant Crios [BC], and GC Cerasmart [GC]). The slides (12 × 14 × 1.0 mm) were obtained by using a precision cutting machine (Isomet 1000 Precision Saw, Buehler, IL, USA) under water cooling. Each slide

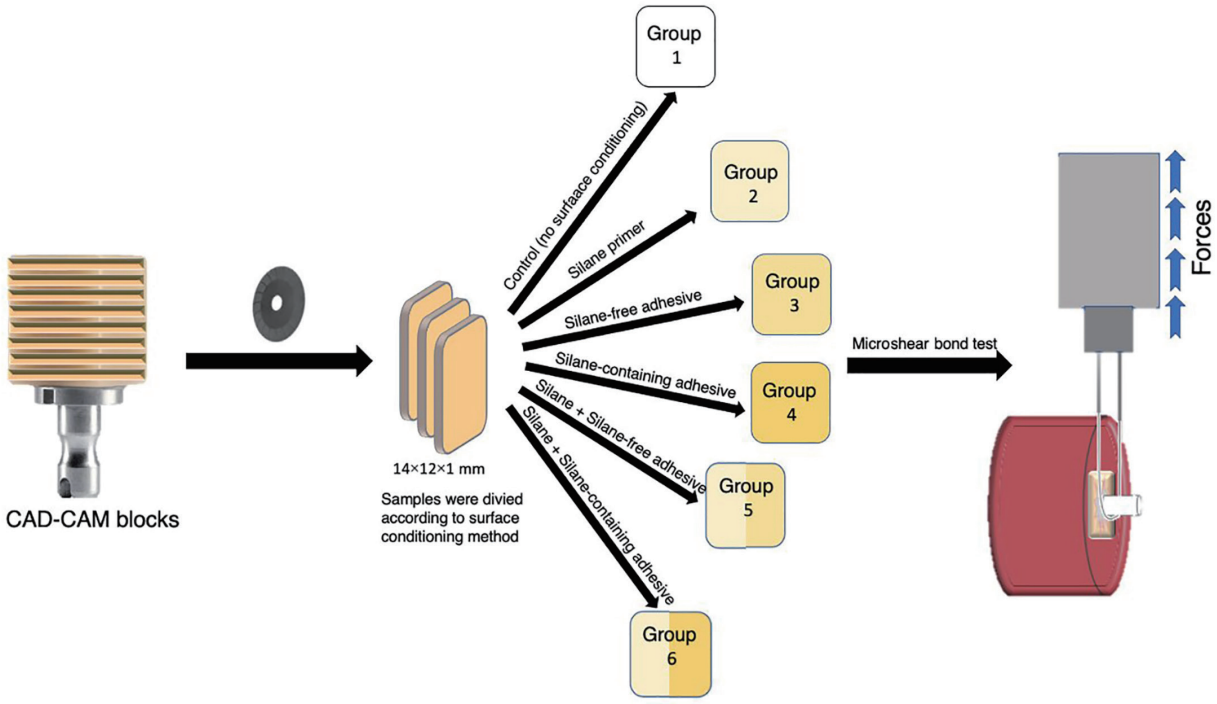

Fig. 1. Schematic setup of the experiment.

Table 1. Materials used in the current study

Material	Brand	Chemical composition	Manufacturer	Batch number
CAD-CAM resin matrix ceramic block	KZR-CAD HR2	Organic part: UDMA, TEGDMA İnorganic part: 75 wt% aggregated SiO_2-Al_2O_3-ZrO_2 (200 - 600 nm), fluoride sustained release filler (700 nm), cluster (1 - 20 mm)	Yamakin Co., Ltd, Kochi, Japan	20031706
	Katana Avencia	Organic part: UDMA, methacrylate monomers Inorganic part: 62 wt% SiO_2 (40 nm), Al_2O_3 (20 nm)	Kuraray Noritake Dental, Tokyo, Japan	000759
	Grandio Blocs	Organic part: methacrylate monomers Inorganic part: 86 wt% filler	VOCO GmbH, Cuxhaven, Germany	2041123
	Brilliant Crios	Organic part: Bis-GMA, Bis-EMA, TEGDMA Inorganic part: 71 wt% barium glass (< 1 μm), amorphous silica SiO_2 (< 20 nm)	Coltène/Whaledent, Altstätten, Switzerland	J22933
	GC Cerasmart	Organic part: Bis-MEPP, UDMA, DMA Inorganic part: 71 wt% barium glass (300 nm), SiO_2 (20 nm)	GC Dental Products, Leuven, Belgium	1805021
Silane coupling agent	Ultradent Silane	Methacryloxypropyltrimethoxy silane < 10%, isopropyl alcohol < 95%	Ultradent Products, South Jordan, UT, USA	BJ282
Universal adhesive	One Coat 7	Ethanol, HEMA, UDMA, hydroxypropylmethacrylate, MMA-modified polyacrylyic acid, amorphous silicic, water. pH: 2.8	Coltène/Whaledent, Altstätten, Switzerland	J01042
	Scotchbond Universal	HEMA, 10-MDP, dimethacrylate resins, Vitrebond™ copolymer, silane, filler, ethanol, water, initiators. pH: 2.7	3M Oral Care, St. Paul, MN, USA	90228A
Self-etch resin cement	Bifix QM	Bis-GMA, benzoylperoxide, amines, barium-aluminiumboro-silicate glass (71 - 73 wt%)	VOCO GmbH, Cuxhaven, Germany	2014181

UDMA, urethane dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; wt, weight; SiO_2-Al_2O_3-ZrO_2, zirconia aluminosilicate; SiO_2, silica; Al_2O_3, alumina; bis-GMA, bisphenol-A-glycidyl methacrylate; Bis-EMA, ethoxylated bisphenol dimethacrylate; Bis-MEPP, 2,2-Bis(4-methacryloxypolyethoxyphenyl) propane; DMA, dimethacrylate; HEMA, hydroxethyl methacrylate; MMA, methyl-methacrylate; 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate

was fixed to the base of a cylinder mold made from auto polymerizing acrylic resin (Ortho-jet, Lang Dental, Wheeling, IL, USA) using a cyanoacrylate adhesive (Model Repair, Dentsply-Sankin, Tokyo, Japan). The bonding surface of each slide was ground using a 600-grit silicon carbide paper (Siawat WA, Sia Abrasives, Liechtenstein, Switzerland) mounted on a surface abrasion and polishing machine (Phoenix Beta, Buehler, IL, USA) under water cooling. The slides were then cleaned in distilled water for 10 minutes using an ultrasonic machine (Biosonic Ultrasonic Cleaner UC1-110, Coltene Whaledent, OH, USA) and dried using compressed air (Fig. 1, Table 1).

The slides were sandblasted for 20 seconds using 50 μm Al_2O_3 particles (Korox 50, Bego, Bremen, Germany) at 2 bar pressure from a vertical distance of 10 mm. All slides were ultrasonically cleaned in distilled water and dried using compressed air. The slides were randomly divided into 6 subgroups depending on the SC protocol as follows:

Group 1 (G1; control): Silane/universal adhesive was not used.

Group 2 (G2; silane): Silane (Ultradent Silane, Ultradent products GmbH, Cologne, Germany) was applied passively onto the bonding surface using an applicator brush. After waiting for 60 seconds, the remaining excessive silane was gently removed using compressed air for 5 seconds.

Group 3 (G3; silane-free universal adhesive): Silane-free universal adhesive (One Coat 7 Universal Resin, Coltene/Whaledent, Langenau, Germany) was applied by rubbing onto the bonding surface for 20 seconds with an applicator brush and was gently air-dried for 5 seconds. The bonding surface was polymerized using a quartz-tungsten halogen light device (Hilux Ultra Plus, Benlioglu Dental, Ankara, Turkey) for

https://doi.org/10.4047/jap.2021.13.5.292

20 seconds.

Group 4 (G4; silane-containing universal adhesive): Silane-containing universal adhesive (3M™ Scotchbond™ Universal Adhesive, 3M Oral Care, St. Paul, MN, USA) was applied onto the bonding surface by using the same procedure as mentioned in the G3.

Group 5 (G5; silane + silane-free universal adhesive): Silane was applied using the same procedure performed in G2. Subsequently, silane-free universal adhesive was applied on the bonding surface, using the same approach as mentioned in the G3.

Group 6 (G6; silane + silane-free universal adhesive): Silane was applied using the same procedure performed in G2. Subsequently, silane-containing universal adhesive was applied on the bonding surface, using the same procedure as mentioned in the G4.

After SC applications, three polyvinyl microtubes (cylinder-shaped, transparent) with an inner diameter of 1.0 mm and height of 1.0 mm were fixed with flowable composite onto the bonding surface of each slide. Each microtube was gently filled with a dual-cured resin cement (Bifix QM, Voco Gmbh, Cuxhaven, Germany) and light polymerization of the resin cement was achieved with an 800 mW/cm^2 intensity quartz-tungsten halogen light device for 20 seconds per side. Accordingly, three resin specimens were composed on each slide (n = 12 per material per group), for a total of 360 specimens. All specimens were held in 37°C distilled water for 24 hours to make sure polymerization is completed.

The polyvinyl microtubes on the specimens were carefully removed using a sharp surgical blade. Premature failures in specimens among all groups took place during polyvinyl tube removal, and were excluded from the study. New specimens were prepared to replace the excluded ones. The replacement process included repeating all preparation steps from the beginning in order to ensure standardization. All specimens were thermally aged (MTE 101, MOD Dental, Esetron Smart Robotechnologies, Ankara, Turkey) for 10000 cycles in a 5°C to 55°C water bath with 20 seconds dwell time. μSBS test was performed by using a universal testing machine (EZtest-500 N Shimadzu, Kyoto, Japan). A metal wire with a diameter of 0.2 mm was wrapped around at the base of resin cement cylinder,[39] and a shear force with a speed of 0.5 mm/min was applied until failure occurred. The force value (N) at which the failure occurred was divided by the space of the bonding surface (mm^2), in order to acquire μSBS values (MPa). The bond surfaces of specimens were observed under a stereomicroscope (Leica S8 APO, Leica Microsystems GmbH, Wetzlar, Germany) at × 40 magnification to determine failure types. Failure types were categorized into adhesive, cohesive, and mixed (both adhesive and cohesive).

An analyzing software program (IBM SPSS Statistics v26, IBM Corp., Chicago, IL, USA) was used for statistical analysis of the data. The Shapiro-Wilk test (> .05) was used to determine the normality of data distribution. The influence of RMC type and SC method on μSBS values was analyzed using a 2-way analysis of variance (ANOVA). Subsequently, Tukey's Honestly Significant Difference post hoc test was used for multiple comparisons. *P* value of < .05 was accepted as statistically significant for all analyses.

RESULTS

Results obtained by two-way ANOVA test stated that the mean μSBS values were significantly affected by RMC type (*P* < .001) and SC method (*P* < .001), except for RMC type × SC method interaction (*P* = .119) (Table 2). Descriptive statistics and Tukey multiple comparisons in terms of mean μSBS values are shown in Table 3.

Regarding the effect of RMC type, the highest mean μSBS values were observed in KZR (48.79 ± 12.43) and GB (48.79 ± 10.16), while the lowest value was observed in GC (32.25 ± 10.82). Multiple comparisons indicated insignificant differences between KZR, KA, and VG (*P* > .05). However, BC and GC both showed significantly lower mean μSBS values when compared to the other 3 RMC materials (*P* < .05). The difference between BC and GC in terms of mean μSBS values was insignificant (*P* > .05). Depending on the RMC type, the μSBS values changed in meaning as follows: KZR = VG = KA > GC = BC.

Considering the effect of SC method, the highest mean μSBS value has been found in G4 (51.97 ± 12.77) and the lowest in G1 (32.13 ± 11.35). All SC method groups showed a significant increase in μSBS

Table 2. Two-way ANOVA results of μSBS values

Source	Type III sum of squares	df	Mean square	F	Sig.
RMC material type (A)	14462.133	4	3615.533	34.624	.000
SC method (B)	11504.937	5	2300.987	22.035	.000
A * B	2940.126	20	147.006	1.408	.119

μSBS, microshear bond strength; RMC, resin-matrix ceramic; SC, surface conditioning; df, degree of freedom; F, variance analysis test statistics. $P < .05$ indicates a significant difference.

Table 3. Mean and standard deviations of μSBS values (MPa) of RMC materials treated with different surface conditioning methods to resin cement

Surface conditioning method	Resin-matrix ceramic material type						
	KZR-CAD HR2	Katana Avencia	Grandio Block	Brilliant Crios	GC Cerasmart	Total	RSD
G1	39.90 ± 5.42	35.51 ± 11.62	40.24 ± 9.01	21.02 ± 5.83	23.98 ± 7.78	32.13 ± 11.35^{a}	35.33%
G2	42.84 ± 11.95	38.24 ± 14.47	46.81 ± 4.16	23.86 ± 7.16	26.51 ± 5.63	35.65 ± 12.85^{a,b}	36.04%
G3	53.24 ± 13.05	42.06 ± 10.35	53.38 ± 8.30	28.13 ± 6.94	32.30 ± 7.55	41.82 ± 13.89^{c}	33.21%
G4	64.44 ± 9.02	52.06 ± 10.32	55.74 ± 8.91	45.44 ± 8.21	42.14 ± 14.67	51.97 ±12.77^{d}	24.57%
G5	44.46 ± 7.32	47.60 ± 14.94	48.14 ± 10.18	35.37 ± 7.49	29.71 ± 6.74	41.06 ± 11.92^{b,c}	29.03%
G6	47.86 ± 9.88	50.94 ± 15.36	48.41 ± 12.95	45.85 ± 16.43	38.85 ± 9.25	46.38 ±13.15^{c,d}	28.35%
Total	48.79 ± 12.43^{A}	44.40 ± 13.90^{A}	48.79 ± 10.16^{A}	33.28 ± 13.32^{B}	32.25 ± 10.82^{B}	41.50 ± 14.16	34.12%
RSD	25.48%	31.31%	20.82%	40.02%	33.55%		

μSBS, microshear bond strength; G1, control group; G2, silane; G3, silane-free universal adhesive; G4, silane-containing universal adhesive; G5, silane + silanefree universal adhesive, G6: silane + Silanecontaining universal adhesive; RSD, Relative standard deviation; Different superscript uppercase letters indicate statistical significance among μSBS values obtained from different material types; Different superscript lowercase letters indicate statistical significance among μSBS values obtained from different surface conditioning methods.

values compared to the control group except the G2. G2 had significantly lower mean μSBS value than the other SCM groups except for G5. The differences between the G3, G5, and G6 were insignificant ($P > .05$). G4 had significantly higher μSBS mean value than the other SCM groups except for G6. Depending on the SC method, the μSBS values changed in meaning as follows: G1 = G2 ≤ G5 = G3 = G6 ≤ G3.

The number of failure modes of RMC materials with different SC method applications is shown in Table 4. Failure analysis indicated more adhesive failures were observed with lower μSBS values while cohesive and mixed failures were observed with higher μSBS values (Table 4) (Fig. 2).

DISCUSSION

This *in vitro* study investigated the effects of silane and two universal adhesives on the adhesive strength between dual-cure resin cement and different CAD-CAM resin-matrix ceramic materials. The results of two-way ANOVA indicated a statistically significant effect of SC method and RMC type on the mean μSBS values. Therefore, both null hypotheses were rejected.

Dimethacrylate resin and silica fillers are the basic components of resin-based materials, and therefore there is a convergence of composition between RMCs and the resin cement. This provides a degree of compatibility between the two materials. However, industrially polymerized RMC materials have too few carbon-carbon double bonds (C=C) available on their surface, which may necessitate the need for pretreatment.[19] The manufacturers of all RMC materials in this study recommend sandblasting with Al_2O_3 particles. Additionally, sandblasting with 50 μm Al_2O_3 particles at a pressure of 1 - 2 bar have demonstrated reliable results in the literature.[19,40] Advantages like extending

https://doi.org/10.4047/jap.2021.13.5.292

Table 4. The number of failure modes of specimens after μSBS test

RMC material type	SC group	Failure type		
		Adhesive	Cohesive cement\ ceramic	Mix
KZR CAD HR 2	G1	9	0	3
	G2	11	0	1
	G3	1	0	11
	G4	0	1\4	7
	G5	5	0	7
	G6	1	1\6	4
Katana Avencia	G1	8	0\2	2
	G2	9	0\2	1
	G3	2	0\3	7
	G4	0	2\9	1
	G5	2	1\4	5
	G6	0	0\10	2
Grandio Block	G1	9	0	3
	G2	10	0	2
	G3	4	0	8
	G4	0	1\7	4
	G5	1	0	11
	G6	1	0\5	6
Brilliant Crios	G1	10	1\0	1
	G2	8	0\1	3
	G3	0	0\4	8
	G4	0	3\6	3
	G5	2	0\1	9
	G6	0	2\9	1
GC Cerasmart	G1	9	0	3
	G2	11	0	1
	G3	1	1\0	10
	G4	0	0\6	6
	G5	2	1\1	8
	G6	0	2\5	5

μSBS, microshear bond strength; RMC, resin-matrix ceramic; SC, surface conditioning; G1, control group; G2, silane; G3, One coat 7; G4, 3M Scotchbond; G5, silane + One coat 7, G6, silane + 3M Scotchbond.

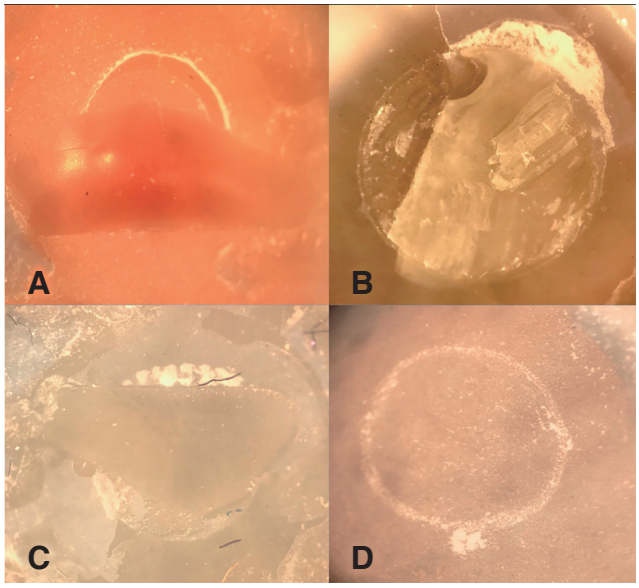

Fig. 2. Failure modes. (A) mixed failure, (B) cohesive cement failure, (C) cohesive ceramic failure, (D) adhesive failure.

the bonding surface, improving the micromechanical bond, clearing surface contamination, and eliminating the smear layer resulting from the milling process can all be acquired through sandblasting.[19,41] However, sandblasting with particles of greater size and/or with higher pressure can result in damaging the surface and thus leading to negative effects on the bond strength.[19,40] Therefore, all tested RMCs were sandblasted by using 50 μm Al_2O_3 particles at a pressure of 2 bar, thus complying with the parameters suggested by the literature.[19,40,41]

The bifunctional monomer of silane is able to react with the methacrylate groups in the resin cement and to the polymer content of RMCs. Additionally, the hydroxyl groups in the silane are able to interact with the inorganic composition of RMCs. Silane also wet the treated ceramic surface, which enhances the resin ability to penetrate into the microporosities, thus creating both mechanical and chemical bonds.[26-28,42] All the aforementioned effects propose that surface conditioning with silane can enhance bond strength between the resin cement and RMCs. However, insignificant difference was found between the control group and silane group in this study, which is in agreement with previous studies.[18,37] This result may be due to the active reaction of the silane agent that increases proportionally with the exposed inorganic area. However, after surface treatment of dispersed filler RMCs, a thin layer of resin stays over the RMC surface. As a conclusion, this layer may inhibit the silane adhesion to silanol (OH) groups of the ceramic surface fillers.[24]

In the present study, two universal adhesives were

tested, silane-containing universal adhesive (Scotchbond 3M) and silane-free universal adhesive (One Coat 7). The results have demonstrated that the use of universal adhesives significantly improved the bond strength between RMCs and resin cement. This higher μSBS results can be attributed to several factors; first, the low pH degree helps the initial chemical reaction to start over the surface of RMC materials. Second, the presence of methacryloyloxydecyl dihydrogen phosphate (10-MDP) monomer improves reaction stability and protects the adhesive interface against hydrolytic degeneration due to its hydrophobic nature, and its phosphate ester group provides adhesion to the passive hydroxyl groups in metal and non-glass ceramic substrates, which are zirconia and alumina fillers in the RMCs used in this study.[34,43-45] Third, the modified methyl-methacrylate (MMA) with polyacrylic acid monomer dissolves and activates the resin located at the surface, which create a suitable medium for free carbon double bonds to polymerize with carbon compounds located in the universal adhesive.[19,46] Also, the relatively low molecular weight of MMA can help with the integration of the resin primer into the roughened resin matrix of the RMC materials, which in turn can increase the bond strength.[47] In addition, the incorporation of water in universal adhesives helps with the activation process by ionizing the acidic monomers.[48]

SC with Scotchbond 3M yielded the highest μSBS values among all groups, with statistically significant difference compared with One Coat 7. The incorporated silane coupling agent in the composition of Scotchbond 3M may have provided additional adhesion to the silica-based inorganic fillers. Also, the presence of a light activated polymerization initiator may have contributed to the bonding by increasing the efficiency of the incorporated silane. There are reports in the literature that the combined use of photo-polymerized adhesives and silane coupling agents can augment bonding proficiency.[32] However, the effectiveness of the incorporated silane is still under investigation. While some studies reported that the acidity of silane containing resin primer may accelerate the reaction between the substructure SiO_2 groups and the silane functional groups to create siloxane bonds,[32,33] other studies reported that the high acidity of universal adhesives may decrease or eliminate the silane efficiency by inducing hydrolysis and dehydration condensation.[34,35] Another explanation of the difference in bonding performance between Scotchbond 3M and One Coat 7 is the possible difference in concentration of 10 MDP between the two adhesives. The wt.% of 10 MDP has direct correlation with bond strength to zirconia,[49] and although information about the exact concentrations of 10 MDP in either adhesive is not available, it can be hypothesized that One Coat 7 does not contain the optimal concentration of 10 MDP needed to promote adequate reaction with the zirconia substrates located in RMCs. In the present study, the highest bond strength was obtained in Scotchbond 3M applied KZR group. The reason for this situation may be related to the content of zirconia aluminosilicate fillers of KZR, which can provide chemical bonds with the phosphate ester group of the 10-MDP monomer. The application of an additional silane agent before using universal adhesives did not improve the bond strength of RMC materials in the present study.

All RMC materials used in this study underwent the same SC methods. However, the results showed that the type of RMC had a significant influence on the adhesive performance, where KZR and GB materials had the highest mean μSBS values. Many studies mentioned that the bond strength of CAD-CAM RMC materials varies between different material types.[20,22,40,50,51] The variations in the microstructures that include different filler sizes and ratios, and the chemical composition of each material can affect the bonding characteristics.[24,52]

In this study, all tested RMC materials are considered as dispersed filler structures because their inorganic fillers are dispersed in their resin-matrix.[53] Dispersed fill structures are divided into three types according to their maximum filler size: micron-dispersed filler structure, submicron-dispersed filler structure, and nano-dispersed filler structure.[24] Due to the fact that KA and GB are characterized by nanometer-sized fillers, these RMCs are classified as having nano-dispersed filler structure. KZR, GC, and BC materials have both nano and submicron sized fillers and are classified as submicron-dispersed filler structures. Three factors seem to affect adhesive strength:

https://doi.org/10.4047/jap.2021.13.5.292

microstructure, the inorganic content ratio, and the inorganic filler chemical composition. According to the microstructure size, the silane efficiency increases as follows: micron structure > submicron structure > nano-size structure.[24] It also seems that silanization effect increases proportionally with the inorganic content ratio as the high filler content increases the exposed surface area after surface treatment which increases silane efficiency.[19] The difference in inorganic materials can also have a direct relationship with silane efficiency.[24]

Although GB is mainly considered a nanodispersed filler structure, its high inorganic composition (86 wt%) may have contributed to the higher bond strength value compared to the BC (70.7 wt%) and GC (71 wt%) in this study. However, insignificant differences were found between bond strength values of KZR (75 wt%), GB (86 wt%), and KA (62 wt%), which contains different inorganic filler wt%. The bond strengths of RMCs not only may be affected by the inorganic wt% but the type and the size of inorganic fillers may also have an effect on the bond strengths.

KZR contains a fluoride-releasing filler (700 nm), zirconium silicate, and submicron zirconia aluminosilicate (SiO_2-Al_2O_3-ZrO_2) nanoclusters (200 - 600 nm) aggregated with silica nanoparticles (20 nm). The sandblasting of KZR, along with the presence of silica particles in the internal structure may have played a role in improving the ability of zirconium particles to form reliable bonds with the silane agent.[54] According to Lucsanszky & Ruse,[4] the release of fluoride increases in wet environment, which can change the internal morphology and properties of ceramics. It was also observed that the internal composition of KZR become rougher after aging. In the present study, the appliance of 1 year aging may have affected the bonding interface in KZR, which may have resulted in stronger bond strength. The larger filler's size, the variation in the molecular types, and aging are factors that can explain the superior results of KZR over the other groups.

In a study by Ferracane,[55] it was indicated that the aging process led to silane hydrolysis which occurred in the interface between the filler and the matrix. Additionally, it was noted that there is an additional increase in silane hydrolysis with zirconia-containing composites. In the present study, it can be expected, according to the aforementioned information, that zirconium silicate containing KZR would have low bond strength values. However, the results in the present study presented that zirconium silicate containing KZR showed higher bond strength than other dispersed-filled RMC materials used in this study except for GB.

Despite KA being a nano-dispersed filler structure with lower filler wt%, it showed higher μSBS values than GC and BC groups, both of which being submicron-dispersed filler structures. This result can be explained by the different inorganic filler compositions, mainly the presence of alumina filler particles in KA composition. The negligible difference in results between GC and BC can be attributed to the similarities in inorganic structure, including filler's size, type, and wt%.

A high relative standard deviation was observed for all groups (Table 3), indicating a greater level of dispersion around the mean. The inconsistencies in the data are mainly related to the non-uniform stress distribution observed with the μSBS test.[56] Microbond tests were mainly developed in order to efficiently evaluate the adhesive surface by reducing the bonding surface area, thus decreasing the incidence of cohesive failures and lowering data dispersion.[57,58] However, variables such as grinding flaws during surface preparation of the slides, flaws in the adhesive layer (e.g. air gaps), the thickness of the adhesive layer, the use of resin cement with low modulus of elasticity for specimen preparation, flaws in the specimens (e.g. air gaps), and possible vibrations and slight changes in angle during force application will result in nonuniform stress distribution in the specimens and therefore influence data dispersion.[59] Another factor is the exercised pressure during the removal process of polyethylene tubes, which is transmitted to the resin cylinder introducing prestresses and microcracks that affect the test values, further increasing data disparity.[58,59]

The present study has several limitations: Only one micromechanic SC method was used, the surface topographies of the roughened specimens with sandblasting were not analyzed, only self-etch resin cement was used as the luting agent, the self-adhesive

resin cement was not investigated, SEM examination was not applied after μSBS test, and preparing specimens with flat surfaces are not similar to anatomical surfaces of natural teeth. Long-term laboratory and clinical studies are needed in order to further understand the role of surface treatment in the adhesive connection between RMC materials and the adhesive cements.

CONCLUSION

With the limitations of this *in vitro* study, the following conclusions were drawn: material type and surface conditionong method significantly affected the bond strength of resin-matrix ceramic materials. KZR CAD HR2 and Grandio Block yielded the highest bond strength values among all tested materials. Surface conditioning only with silane agent did not improve the bond strength. Application of universal adhesives increased the bond strength. Scotchbond 3M universal adhesive was found to be the best conditioning method for resin-matrix ceramic materials. The application of a separate silane step showed insignificant effect on the bond strength, regardless of the type of universal adhesive.

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