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Research article

Effect of trialkoxysilane/aminosilane-containing universal adhesive on resin-ceramic microtensile bond strength and ceramic wettability: An in-vitro study

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ABSTRACT

Background: The dual-silane (trialkoxysilane/aminosilane) universal adhesive (UA) is claimed for its enhanced priming capacity of glass-ceramics.

Objective: This study evaluated the effect of organofunctional trialkoxysilane- and organofunctional trialkoxysilane/aminosilane-containing UAs on the long-term resin–ceramic microtensile bond strength (μTBS) and wettability of ceramic.

Methods: Hydrofluoric acid-etched lithium disilicate discs were distributed into four groups as follows: (control), no priming was performed; (MBN), primed using a silane-based primer (Monobond N); (SBU), primed using a trialkoxysilane-containing UA (Single Bond Universal Adhesive) and (SBP), primed using a trialkoxysilane/aminosilane-containing UA (Scotchbond Universal Plus Adhesive). Ceramic discs were cemented into blocks then sectioned into micro-beams stored in distilled water at 37° for 1 year. The μTBS was evaluated followed by assessment of the failure modes. The contact angle of the two UAs was measured with a goniometer using the sessile drop technique.

Results: MBN significantly improved the resin-ceramic μTBS (31.71 ± 6.33 MPa) compared to the control group. The resin-ceramic μTBS obtained after priming using SBP (22.83 ± 3.42 MPa) was comparable to those of MBN. SBU showed significantly inferior resin-ceramic μTBS (16.02 ± 6.28 MPa) compared with MBN. Mixed failures mode patterns were the most frequent in the groups. The ceramic wettability of both UAs did not significantly differ.

Conclusion: Ceramic priming using a UA with dual-silane monomers (organofunctional trialkoxysilane/aminosilane) resulted in long-term adhesion comparable to a silane-containing primer. Incorporating aminosilane monomer in UA formulation did not affect the wetting of characteristics of the UA solution and enhanced its glass-ceramic priming capacity.

Clinical significance: The use of UA with optimized silane content as a primer for glass-ceramics simplifies clinical adhesive procedures including resin cementation and repair of ceramic restorations.

1. Introduction

Ceramic restorations are widely applied in both esthetic and functional oral rehabilitation treatments [1]. The longevity of ceramic restorations relies on multiple factors including cementation protocol utilized to obtain robust tooth–restoration adhesion. Lithium

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disilicate ceramics are rich in glass phase [2]; thus, chemicals surface treatments such as hydrofluoric acid (HF) etching is performed to break down some of the glass content of the ceramic material [3]. This increases the bondable surface area and the surface energy of the ceramic surface, promoting adequate adhesion [4]. However, to achieve chemical resin-ceramic adhesion, a priming step using a silane-based primer is required [3,5]. Silane monomer organo-functional groups which bond to resin-based substrates and a silanol group which bonds to glass-ceramics. Silane primers contain large amounts of organic solvents which the silane molecules are diluted and dissolved at a certain pH [6]. Therefore, an air-drying step is mandatory to ensure solvent evaporation after primer application [7]. Some universal adhesives (UAs) were claimed that they can be used to promote adhesion to glass-ceramics and oxide ceramics [8]. However, the use of silane-containing UAs for glass-ceramic priming could not be comparable to silane-based or silane-containing universal primers [9–12]. The chemical unstableness of the silane content within the UA formulation might be due to acidity along with the complex composition of the silane-containing UAs might be a plausible explanation for the impaired priming potential [11,13,14].

While most silane-containing UAs contain organofunctional trialkoxysilane monomrtd, the first dual-silane UA, Single Bond Universal Adhesive (SBP), 3 M Oral Care, St. Paul, USA, has been recently introduced containing both γMPTES and aminosilane monomer (3-(aminopropyltriethoxysilane (APTES)) (Fig. 1). Owing to its dual-silane technology, SBP showed promising glass-ceramic priming capacity compared with other adhesives [15,16]. The question is that would SBP promote long-term resin-ceramic adhesion to Lithium disilicate ceramics. This study evaluated the effect UAs silane content on the resin-ceramic microtensile bond strength (μTBS) after long-term water storage. The null hypotheses were that (1), ceramic-priming using either a γ-methacryloxypropyltriethoxysilane (γMPTES)/APTES-containing UA, a γMPTS-containing universal adhesive, or a silane-based primer would not have a significant effect on the resin-ceramic adhesion, (2), the contact angle of the two silane-containing UAs will not significantly differ.

2. Methods

Table 1 describes the materials utilized in the study as well as their composition.

2.1. *Specimens' preparation*

Ceramic blocks (GC InitialLiSi Block) were sectioned into smaller discs (Fig. 2a). The prepared discs were polished flat and ultrasonically cleaned in distilled water. Hydrofluoric acid (~5 %) ceramic etchant was used for surface treatment for 20 s and then neutralized using sodium carbonate and calcium carbonate powder (Fig. 2b). This was followed by thorough washing and ultrasonic cleaning in distilled water to eliminate the residues after etching. The ceramic discs were randomly distributed based on the priming step into four groups (n = 10/group). In group 1 (control), no priming was performed; group 2 (MBN), Monobond N, Ivoclar Vivadent was used for priming of the ceramic discs as per the manufacturer's instructions.; group 3 (SBU), Single Bond Universal Adhesive, 3 M Oral Care was first mixed with the dual-cure activator and subsequently used for priming of the ceramic discs and group 4 (SBP), Scotchbond Universal Plus Adhesive, 3 M Oral Care used for priming of the ceramic discs (Fig. 2c). Both the universal adhesives were not light cured before applying RelyX Universal Resin Cement for cementation of ceramic discs. Each two ceramic discs surface-treated using the same protocol were vertically aligned and cemented together making cemented blocks (2 discs) (Fig. 2d). The cemented discs were maintained under 1 kg cementation force for 60 s [16,17]. The interfacial area of the cemented blocks was light-cured from four sides for 40 s using a light-curing unit worked at an intensity of 1000 mW/cm^2. Further light-curing from the top and bottom sides was performed for 40 s each. Then, the cemented ceramic blocks were stored in a distilled water path at 37 °C for 24 h.

2.2. *μTBS evaluation*

Micro sectioning of the blocks across the interface in the "x" and "y" directions microbeams was achieved using a 4-inch diamond cutting blade (IsoMet Blade, Buehler, Lake Bluff, USA) mounted on a low-speed precision cutting saw (IsoMet 1000 Linear Precision Saw, Buehler, Lake Bluff, USA) into microbeams with a cross-sectional area of approximately 1 (±0.2) mm^2 (Fig. 2e). The microbeams failed during sectioning (pre-test failures (PTFs)) were noticed and reported. The prepared microbeams were stored in distilled water for 1 year at 37 °C in an incubator (Fig. 2f). Chloramine-T powder (0.5 g for each 100 mL) was added to the storage distilled water to decrease the biofilm formation. The storage water was changed weekly. For μTBS evaluation, microbeam' cross-sectional surface area

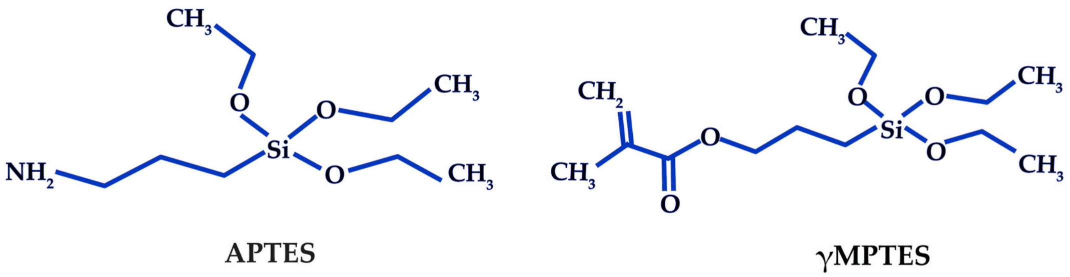


Fig. 1. The chemical structure of 3-(aminopropyl)triethoxysilane (APTES) and γ-methacryloxypropyltriethoxysilane (γMPTES).

Table 1
Materials utilized in the study.

Material	Description	Composition
Initial LiSi Block, GC, Tokyo, Japan.	Fully crystalline lithium disilicate ceramic	SiO_2 81 %, P_2O 8.1 %, K_2O 5.9 %, Al_2O_3 3.8 %, TiO_2 0.5 % and CeO_2 0.6 %
Single Bond Universal Adhesive, 3 M Oral Care, St. Paul, USA.	Silane-containing universal adhesive	10-MDP, Vitrebond copolymer, dimethacrylate resin, HEMA, bisphenol A glycidyl methacrylate, photo initiator, silane, organic solvent, water.
Scotch bond Universal Plus Adhesive, 3 M Oral Care, St. Paul, USA.	Silane-containing universal adhesive	10-methacryloyloxydecyl dihydrogen phosphate, Vitrebond copolymer, dimethacrylate resin, HEMA, crosslinking monomer, dual-cure accelerator, photo initiator, optimized silane monomers, organic solvent, water.
Monobond N, Ivoclar Vivadent, Schaan, Liechtenstein.	Silane-containing universal primer	Silane methacrylate, Disulfide methacrylate, Phosphoric acid methacrylate Alcohol.
RelyX Universal Resin Cement, 3 M Oral Care, St. Paul, USA.	Dual-cure resin-cement	Dimethacrylates, photo initiator, phosphorylated dimethacrylates, redox initiator system, fillers and rheological modifiers, pigments.

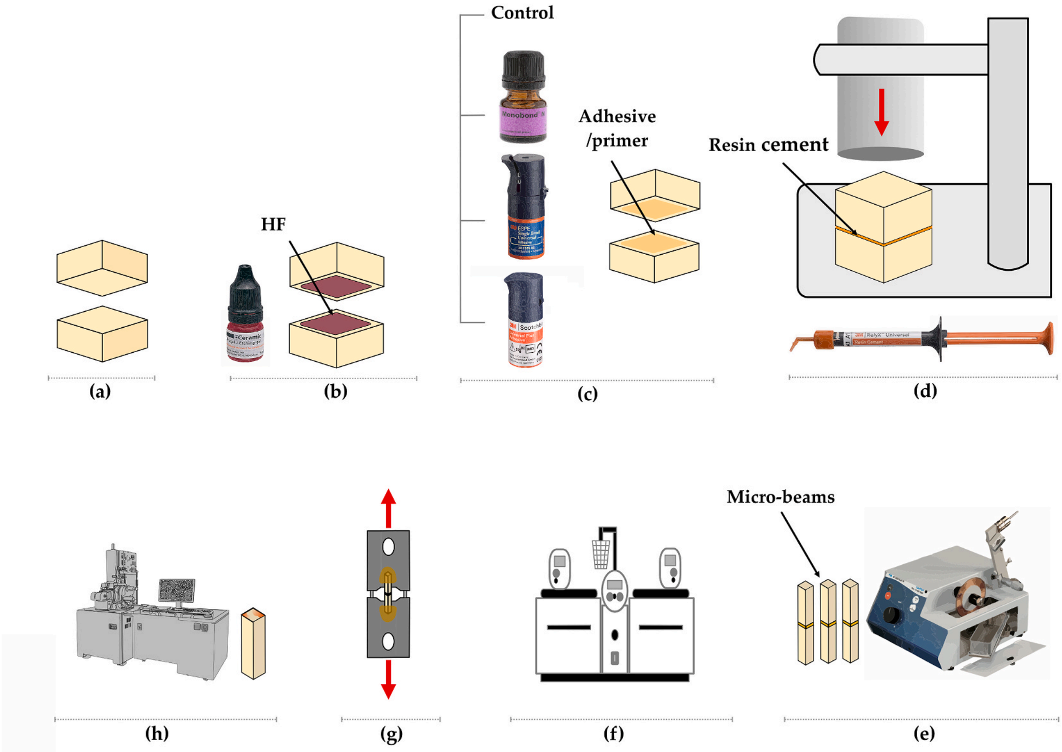


Fig. 2. Schematic diagram illustrating the steps of specimens' preparation for microtensile bond strength evaluation. (a): ceramic discs; (b) surface treatment of the ceramic surface with ~5 % hydrofluoric acid (HF); (c): ceramic priming either with silane-containing universal primer or with silane containing universal adhesives); (d): cementation (under force) of two ceramic discs; (e): sectioned of bonded specimens into micro-beams; (f): water storage of the obtained-microbeams; (g): microtensile bond strength evaliatopm; (h): failure mode assessment; red arrow: direction of force.

was determined using a digital micrometer (Mitutoyo, Kanagawa, Japan). The microbeam was aligned straight and glued (fixed) onto a metal jig mounted on a universal testing machine equipped with a load cell of 1-kN capacity (Fig. 2g). The microbeam was subjected to a tensile force at a 1 mm/min crosshead speed. Calculation of the resin–ceramic μTBS was done by dividing the force recorded at failure by the predetermined cross-sectional surface area.

2.3. *Assessment of failure mode*

A light microscope was used to determine the type of failure mode that occurred during μTBS testing. Tested microbeams with failure mode patterns which cannot be identified using the light microscope were viewed with a scanning electron microscope (SEM) at low and high magnifications (Fig. 2h). In addition, representative microbeams were selected for the most frequent failure mode patterns and examined using SEM. The noticed failure mode patterns were classified as interfacial (adhesive) failures that include debonding, failure or fracture of the microbeam at the interface and mixed failures that include debonding, failure or fracture of the microbeam at the ceramic and the resin-cement combined with interfacial (adhesive) failures. No cohesive failures were reported.

2.4. *Contact angle (CA) measurement*

Ceramic blocks (GC InitialLiSi Block) were cut into a total of fifteen (14 mm × 12 mm) 2 mm thick discs using a high-speed automatic precision cutting saw. The ceramic discs were polished, cleaned, acid-etched with hydrofluoric acid, and washed exactly as described for the *μTBS* specimens' preparation. Lithium disilicate ceramic discs were randomly distributed into three groups ($n = 5$); distilled water (control), SBU and SBP. The contact angles of distilled water, SBU and SBP to Lithium disilicate ceramic surfaces were measured with a goniometer (CA Goniometer (model no. 190-F2), ramé-hart instrument Co., Succasunna, NJ, USA) using the sessile drop technique. A drop (3 μl) of water and UAs was applied on LCD and CA was measured after 2 s. Two measurements were performed for each Lithium disilicate ceramic disc.

2.5. *Statistical analysis*

Calculation of the sample size was performed using G*Power software, version 3.1.9.7 (Heinrich-Heine-Universität Düsseldorf, Düsseldorf, Germany), indicating 10 samples per group to achieve 98 % power and a 5 % significance level. To statistically analyze μTBS data, the block was considered as the experimental unit. Therefore, the microbeams obtained from the same block were averaged while accounting values corresponding to PTFs as 0 MPa. After assessment of the data normality, one-way analysis of variance (ANOVA) followed by Tukey's HSD ($\alpha = 0.05$) were used for the analysis of the μTBS considering the impact of the ceramic priming on the resin–ceramic bond strength and effect the priming material on the contact angle.

3. Results

3.1. *μTBS*

The μTBS data showed normal distribution. The result of one-way ANOVA for the μTBS is presented in Table 2. The ceramic priming step had a statistically significant ($p < 0.001$) effect on the obtained resin-ceramic μTBS. Differences between the groups' means were detected using Tukey's multiple comparisons test (Table 3).

The μTBS means of the tested groups are presented in Table 4 and Fig. 3. In contrast to both SBU and SBP, only priming of ceramic discs using a universal silane-containing primer (MBN) markedly increased the resin-ceramic μTBS, obtaining a μTBS mean of 31.71 ± 6.33 MPa compared with both the control and SBU groups. No statistically significant difference was detected between the μTBS SBP (22.83 ± 3.42 MPa) and MBN.

3.2. *Assessment of failure mode*

Fig. 4 illustrates the incidence (expressed as percentages) of the failure mode patterns recorded. For all the tested groups, mixed failures (Fig. 5c and d) were the most predominant while adhesive failures (Fig. 5a and b) came second to mixed failures with no cohesive failures were noticed within ceramic or resin cement. A total of 2 PTFs were recorded only for SBU group. Representative SEM images at different magnifications of the failure modes patterns observed are shown in Fig. 5.

3.3. *Contact angle (CA)*

The mean ± standard deviation (SD) and representative images of contact angle for each group are presented in Figs. 6 and 7a–c. No statistically significant difference was detected between SBU and SBP ($p = 0.768$).

4. Discussion

Two UAs were used as alternatives to a silane-based universal primer in this study. The μTBS test was employed in the current study because of its ability to discriminate between tested groups [18]. The experimental set-up of this study involved simulation of the intraoral cementation procedure the ceramic discs were cemented and maintained under force [16,17]. The ceramic surface treatment steps including polishing, HF etching, priming and cementation were performed by one operator to ensure standardization of the procedures [19]. It was hypothesized that ceramic priming would not have a significant impact on adhesion to a glass-ceramic material. The present study showed that the resin–ceramic adhesion was significantly improved when the ceramic priming was done using a silane-based primer. Ceramic priming using a γMPTS -containing UA (SBU) yielded significantly lower μTBS than those recorded for

Table 2
One-way ANOVA results of the resin-ceramic microtensile bond strength.

	Df	Sum Sq	Mean Sq	F Value	*p*-value
Ceramic priming	3	756.2	252.05	9.826	<0.001*
Residuals	16	410.4	25.65		

Table 3
Tukey's HSD results of the resin-ceramic microtensile bond strength.

Pair-wise groups comparisons	Mean Difference	Lower bound	Upper bound	*p*-value
Control–SBP	5.367	−3.797	14.532	0.368
Control–SBU	−1.442	−10.607	7.722	0.969
Control–MBN	14.243	5.079	23.407	0.002[a]
SBU–SBP	−6.810	−15.974	2.355	0.187
MBN–SBP	8.876	−0.289	18.040	0.059
MBN–SBU	15.685	6.521	24.849	<0.001[a]

[a] : indicates statistically significant difference.

Table 4
The mean ± standard deviation (SD) μTBS (in mega Pascals (MPa)) recorded after long-term water storage for the tested groups.

Group	μTBS (MPa)	
	Mean ± SD^1	(PTFs^2/n^3)
Control (no priming)	17.46 ± 3.38	(0/30)
MBN	31.71 ± 6.33	(0/30)
SBU	16.02 ± 6.28	(2/28)
SBP	22.83 ± 3.42	(0/30)

1 = standard deviation; *2* = pre-test failures; *3* = total number of micro-beams tested.

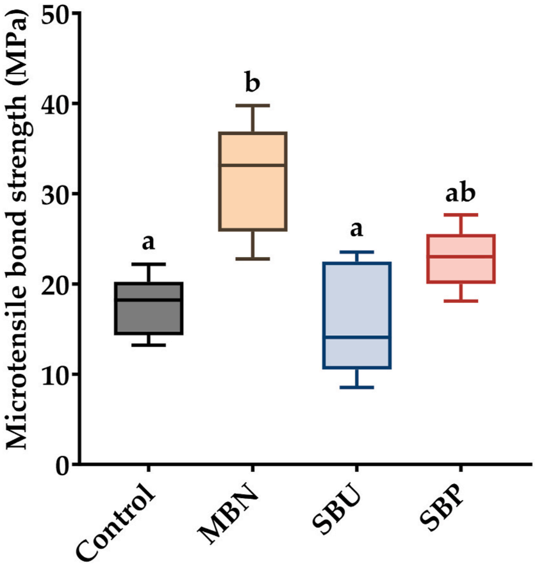


Fig. 3. Box-whisker plots presenting the micro-tensile bond strength (μTBS) (in mega Pascals (MPa)) of the control group (no priming); MBN group, priming with (Monobond N); SBU group, priming with Single Bond Universal Adhesive and SBP group, priming with Scotch bond Universal Plus Adhesive. The median μTBS for each group is indicated by the horizontal line within each box. Statistically significant difference between the groups is indicated with different superscript letters.

the MBN. Therefore, the first hypothesis was not accepted. Interestingly, the μTBS obtained after ceramic priming using a γMPTES/APTES-containing UA (SBP) was statistically comparable to MBN.

Prior to the priming step, the ceramic surface had to be acid-etched using HF. This resulted in partial dissolution of the glassy matrix within the ceramic, creating a microscopically porous and rough surface favorable for adhesion [3]. Furthermore, HF can enhance the density of the hydroxyl groups at the ceramic surface [6]. Silane-based primers contain low amounts of prehydrolyzed silane monomers and substantial amounts of organic solvents [5,20]. The adhesion between silane primers and glass ceramic relies, in part, on a condensation reaction between hydroxyl groups of the ceramic and the silanol groups of the silane monomer [3,21]. In addition, silane monomer' organofunctional groups bonds with the methacrylate-based cement used for cementation [3,21]. This ensures adequate resin-ceramic adhesion. However, what explains the superior priming capacity of the universal silane-containing primer is that after air-drying and solvent evaporation, a homogenous silane layer is formed and deposited on the ceramic surface, significantly improving resin ceramic adhesion [21]. This is not assumed to occur with the UAs, regardless of their silane content [16]. In fact,

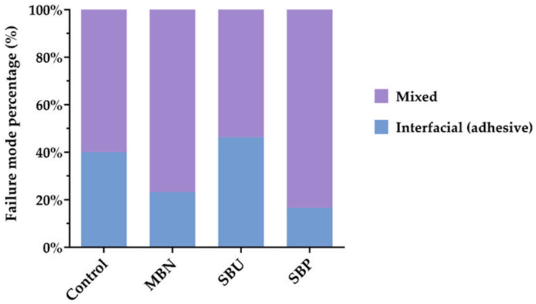


Fig. 4. The incidence (frequency) of different failure mode patterns observed for the microbeams of the tested groups: control (no priming), Monobond N (MBN), Single Bond Universal Adhesive (SBU) and Scotch bond Universal Plus Adhesive (SBP).

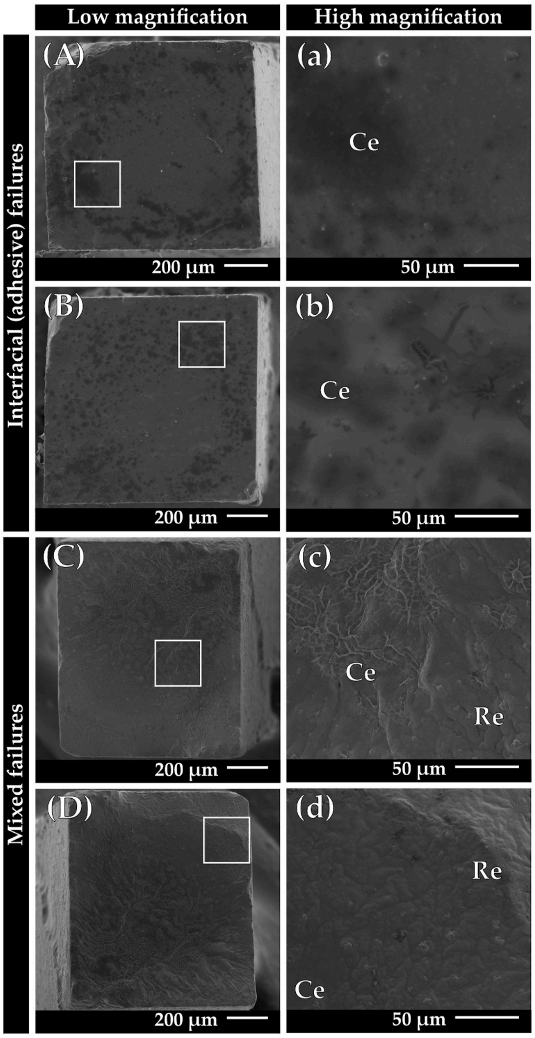
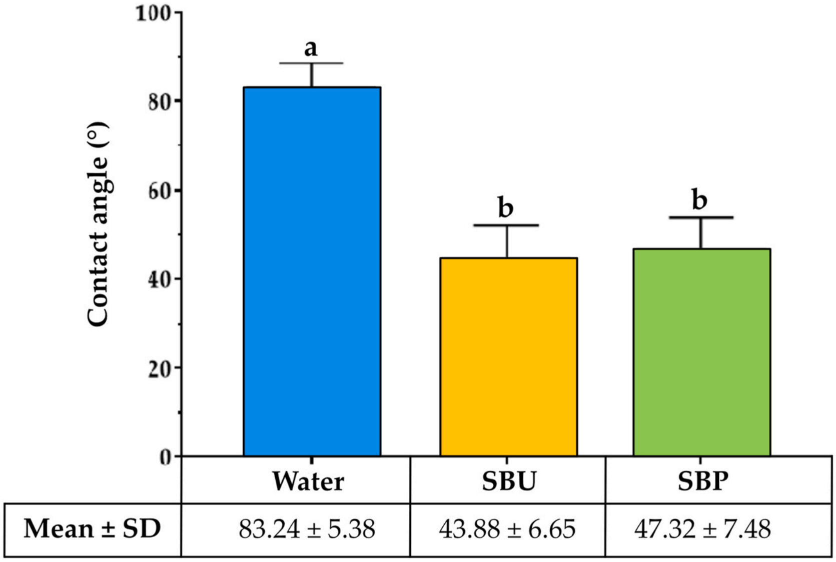


Fig. 5. SEM images captured of the tested microbeams at 70 ×, 85 × and × 500 magnifications. A–a, B–b: interfacial (adhesive) failures; C–c, D–d: mixed failure; Ce: Ceramic; Re: resin-cement.

air-drying of adhesives for shorter periods cannot ensure optimum water and solvent evaporation due to the compositional complexity of adhesives [22,23], adversely affecting the physico-mechanical properties of adhesive material. Furthermore, the residual water content within the adhesive layer in addition to the hydrophilic monomers such as HEMA would make the adhesive layer more prone to hydrolysis overtime [24]. This, in part, can explain the inferior adhesion promoted by the UAs compared to the universal primer.



	Water	SBU	SBP
Mean ± SD	83.24 ± 5.38	43.88 ± 6.65	47.32 ± 7.48

Fig. 6. The means ± standard deviations (SDs) of the contact angle on etched lithium disilicate ceramic discs, as measured for distilled water; SBU and SBP. Statistically significant difference between the groups is indicated with different superscript letters.

Fig. 7. Representative images of contact angle measurement on etched lithium disilicate ceramic discs, as measured for high purity water (a); SBU (b) and SBP (c).

Additionally, UAs acidic monomers such as 10-MDP to simultaneously etch and infiltrate into tooth structure [8,25]. Such acidity could adversely affect the chemical stability of the silane monomers in the UAs' solutions [13], minimizing their priming efficiency and, in turn, the obtained resin-ceramic bond strength [11,16]. Although UAs can, to some extent, infiltrate into the micro-porous ceramic surface created by HF etching [13], such infiltration might be impaired by the high viscosity of the UA solutions compared to the universal primer.

It is noteworthy that the UA with the dual-silane (γMPTES/APTES) content, in contrast to its precursor UA that contains one type of silane (γMPTS) molecules, resulted in obtaining resin-ceramic adhesion comparable to the silane-containing universal primer. These findings can, in part, be explained by the optimized silane technology utilized in SBP. This is believed to enhance priming efficiency due to increasing the available silanol groups of the two silane monomers (γMPTES and APTES). In addition, the amino groups of APTES silane molecule could intensify the priming efficiency by the simultaneous interaction with hydroxyl groups formed on the HF-etched ceramic surface [26]. APTES also has a siloxane intermolecular crosslinking effect [26]. This might optimize the silane interaction layer and, in turn, obtained resin-ceramic adhesion [15]. On the other hand, the inferior priming capacity of γMPTS-containing UA can not only be elucidated by the lack of ATPES molecules and the deteriorated chemical stability of the silane molecules within the acidic UA formulation, but also with the chemical in compatibility between the UA and the used resin cement.

Despite the advantages of the μTBS test, the results should be elucidated along with the failure modes recorded. To specify, the main outcome of μTBS results might be influenced by the including the bond strength values of microbeams that failed cohesively [27]. The failure modes patterns of tested microbeams as well as pretest failures (PTFs) should be considered during the interpretation of the bond (adhesion) strength data. The percentage of PTFs might be a sign of inferior resin-ceramic adhesion, particularly on the long-term basis. PTFs should be included in the statistical analysis and considered as 0 MPa [28]. Similarly, the inclusion or censoring of the bond strength values corresponding to cohesively failed microbeams can influence the statistical analysis of μTBS data [27]. Fortunately, only a few microbeams failed during micro-sectioning. The fractographic analysis of the microbeams revealed a high incidence of mixed failures followed by adhesive failures. The preparation of the μTBS microbeams may be related to the failure mode types occurred before and during testing. The μTBS samples employed in the current study consisted of ceramic part cemented to another ceramic part using a dual-cure resin cement (Fig. 1). This would not only ensure a uniform and void-free layer of the cement used, but also can minimize the incidence of cohesive failures during μTBS testing [16,17]. Furthermore, structure of the microbeams also give chance for solely evaluating resin-ceramic interface [16,17]. As for the contact angle measurement, although the Bis-GMA monomer in SBU might reduce the contact angle of the adhesive solution minimizing ceramic wettability [29], the contact angle measurements of both UAs did not significantly differ in this study (Figs. 6 and 7a–c). Thus, the second hypothesis had to be accepted. Overall, it was found that γMPTES/APTES-containing UA (SBP) presented promising ceramic priming efficiency. The bonding performance of the two universal adhesives tested in this study coincides with a similar previous one in which short-term (immediate) bond strength evaluation was attempted [16]. Despite that, the main outcome of this study must be considered with caution due to the lack of detailed chemical analysis of the interaction between the dual silane containing UA and lithium disilicate ceramics.

5. Conclusion

Both organofunctional trialkoxysilane- and organofunctional trialkoxysilane/aminosilane-containing UAs did not significantly improve long-term resin-ceramic adhesion. Incorporating organofunctional trialkoxysilane and aminosilane molecules in UA formulation did not affect the wetting of characteristics of the UA solution and enhanced its glass-ceramic priming capacity, with resin-ceramic bond strength comparable to universal silane-containing primer. Priming of glass-ceramic using an organofunctional trialkoxysilane-containing UA resulted in suboptimal resin-ceramic μTBS (adhesion) compared to priming of glass-ceramic using a silane-based primer.

Data availability statement

The data associated with the current study have not been deposited into a publicly available repository. Data will be made available from the corresponding author on request.

CRediT authorship contribution statement

Ali Robaian: Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Software, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:Ali Robaian reports a relationship with Prince Sattam bin Abdulaziz University that includes: employment. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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