



Article Silane-Containing Universal Adhesives Influence Resin-Ceramic Microtensile Bond Strength

Mohamed M. Awad ¹, Feras Alhalabi ^{1,*}, Abdullah Alshehri ¹, Mohammed Ali Salem ¹, Ali Robaian ¹, Shahad Alghannam ², Abdullah S. Alayad ³, Basil Almutairi ³ and Ali Alrahlah ^{3,4}.

- ¹ Department of Conservative Dental Sciences, College of Dentistry, Prince Sattam Bin Abdulaziz University, Al-Kharj 11942, Saudi Arabia
- ² College of Dentistry, Prince Sattam Bin Abdulaziz University, Al-Kharj 11942, Saudi Arabia
- ³ Department of Restorative Dentistry, King Saud University College of Dentistry, Riyadh 11545, Saudi Arabia ⁴ Engr Abdullah Burghan Percent Chair for Dental and Oral Pelabilitation. King Saud University
- Engr Abdullah Bugshan Research Chair for Dental and Oral Rehabilitation, King Saud University, Riyadh 11545, Saudi Arabia
- * Correspondence: f.alhalabi@psau.edu.sa

Abstract: Background: Silane-containing universal adhesives (UAs) are marketed as adhesion promotors for glass-ceramics. Objectives: This study aimed to evaluate the priming capacity of γ methacryloxypropyltrimethoxysilane (γ MPTS)-containing and γ -methacryloxypropyltriethoxysilane (yMPTES)/3-(aminopropyl)triethoxysilane (APTES)-containing universal adhesives (UAs) for lithium disilicate ceramic (LDC). Materials and Methods: Etched LDC discs were distributed into four groups according to the priming material used: (control), no priming; (MBN), LDC was primed with a universal primer (Monobond N); (SBU), yMPTS-containing UA (Single Bond Universal Adhesive) was used as a primer; and (SBP), yMPTES/APTES-containing UA (Scotchbond Universal Plus Adhesive) was used as a primer. LDC discs were cemented using a dual-cure resin cement, then sectioned into microbeams for microtensile bond strength (µTBS) evaluation. Failure modes were assessed. Results: MBN application showed the highest µTBS among all groups. γMPTES/APTES-containing UA (SBP) resulted in considerably higher μ TBS compared with γ MPTS-containing UA (SBU) or the control group. The mixed failures were the most predominant among all groups. Conclusions: The effect of silane-containing UAs on resin-ceramic μ TBS is material dependent. Although γ MPTES/APTEScontaining UA improved bonding to LDC, the priming of LDC with either of the UAs tested cannot be considered as an alternative to a separate silanization (priming) step using a universal primer.

Keywords: adhesives; ceramics; microtensile bond strength; silanes

1. Introduction

Ceramic restorations are usually cemented using adhesive or self-adhesive resin cements which can effectively bond to both tooth structure and ceramic restoration while having enhanced esthetic and mechanical properties compared with conventional cements [1]. Robust resin-ceramic bond strength (adhesion) is crucial for the clinical outcome of indirect restorations made of lithium disilicate ceramic (LDC) [2,3]. Multiple steps are required to promote adequate bonding between LDC and methacrylate-based materials, such as adhesives or resin-based cements [4,5]. Ceramic surface treatments, such as priming in addition to using a type of luting cement, can influence resin-ceramic bond strength (adhesion) [6,7]. LDC is one of the most used glass-ceramic materials, which can be supplied in a fully or partly crystallized form, which requires crystallization (firing) or heat-pressing. Despite the wide range of chemical and microstructural differences among LDC materials, such materials are generally composed of a glass matrix supported by crystals [8]; hence, etching with hydrofluoric acid (HF) is required to dissolve the glass matrix [9] and increase the surface energy and wettability [10]. This step is usually followed by a priming procedure using a silane-containing or universal primer to promote chemical



Citation: Awad, M.M.; Alhalabi, F.; Alshehri, A.; Salem, M.A.; Robaian, A.; Alghannam, S.; Alayad, A.S.; Almutairi, B.; Alrahlah, A. Silane-Containing Universal Adhesives Influence Resin-Ceramic Microtensile Bond Strength. *Coatings* 2023, *13*, 477. https://doi.org/ 10.3390/coatings13020477

Academic Editors: Lavinia Cosmina Ardelean and Laura-Cristina Rusu

Received: 26 January 2023 Revised: 13 February 2023 Accepted: 14 February 2023 Published: 20 February 2023



Copyright: © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). bonding (adhesion) between silane-treated LDC and methacrylate-based materials, such as adhesives or resin-based cements [9,11]. Dental silane primers usually contain organofunctional trialkoxysilanes, such as γ -methacryloxypropyltrimethoxysilane (γ MPTS) and γ -methacryloxypropyltriethoxysilane (γ MPTS), which are diluted and dissolved in ethanol and water at a specific pH [12]. Silane molecules chemically bond to methacrylate-based materials via the organofunctional group (methacrylate) and to HF-etched glass-ceramic via the silanol group resulting from silane activation (hydrolysis) [9,11,12]. Clinically, upon application of silane primer to a glass-ceramic restoration for a specific time (usually 60 s), an effective air-drying step is required to disperse any remaining excess of the primer solution and to evaporate the ethanol (solvent) and water content to form a homogenous silane layer onto the ceramic surface; otherwise, the resin-ceramic bond strength would be impaired [13], which would negatively affect the clinical performance of glass-ceramic restorations.

To simplify dental adhesive procedures, some universal adhesives (UAs) have been marketed as effective adhesion promotors (primers) to indirect materials owing to their silane and 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) content [14]. Such adhesives are claimed to be alternatives to 10-MDP-containing primers used for polycrystalline ceramics or silane-containing primers used for glass-ceramics. Recent studies indicated that although UAs can effectively promote bonding to zirconia ceramic or indirect composite [15], their ability to promote adequate bond strength (adhesion) to glass-ceramics is limited [15–19]. Similarly, the application of such silane-containing UAs as an adhesion promoter to hybrid ceramic failed to replace the use of commercially available silane-based primers [20]. Experimentally, the fresh mixing of silane compounds such as γ MPTS with dental adhesives is more effective than adding silane compounds to an adhesive composition followed by long-term storage due to the acidity (pH) of the adhesive solution [21] and complexity of the contemporary UAs' composition [20,22]. Such adhesives contained only organofunctional trialkoxysilane compounds. Recently, Scotchbond Universal Plus Adhesive (SBP; 3M Deutschland GmbH, Neuss, Germany) was introduced to the dental market as the first UA containing both organofunctional trialkoxysilane and amino-silane compounds, such as 3-(aminopropyl)triethoxysilane (APTES). Such optimized silane content is expected to enhance the priming efficacy of SBP adhesive to glass-ceramics due to its optimized silane content (γ MPTES and APTES) [23]. Nevertheless, it is essential to compare the priming capacity of the new γ MPTES/APTES-containing UA with commercially available silane-based or universal primers used clinically. The use of SBP as an alternative to silane-based or universal primer is assumed to promote adequate resinceramic bond strength (adhesion), which can save more clinical time and the additional financial cost of silane-containing or universal primers. In addition, γ MPTES/APTEScontaining UA can be applied in more versatile clinical situations, such as in the intra-oral repair of LDC restorations. This study aimed to assess whether silane-containing UAs can influence the resin-ceramic microtensile bond strength (μ TBS). Therefore, the effect of two silane-containing UAs on the resin-ceramic µTBS was evaluated in comparison with silane-containing universal primer. The null hypotheses were that: (1) Ceramic surface treatments (primer and UAs) would have no significant effect on the resin-ceramic µTBS; (2) There would be no significant difference between the resin-ceramic μ TBS obtained after the use of either of the two silane-containing UAs tested.

2. Materials and Methods

The materials used in the study and their compositions are described in Table 1.

Material (LOT Number)	Code	Composition		
Initial LiSi Block, GC, GC corporation, Tokyo, Japan. (LOT: 2201201C)	LDC	Silicon dioxide: 81%; phosphorus oxide 8.1%; potassium oxide 5.9%; aluminum oxide 3.8%; titanium oxide 0.5%; and cerium oxide 0.6%.		
Single Bond Universal Adhesive, 3M Deutschland GmbH, Neuss, Germany. (LOT: 10608B)	SBU	10-MDP phosphate monomer, Vitrebond, copolymer, HEMA, Bis-GMA, dimethacrylate resin, camphorquinone, silane (γMPTS), ethanol, and water. 10-MDP phosphate monomer,		
Scotchbond Universal Plus Adhesive, 3M Deutschland GmbH, Neuss, Germany. (LOT: 7676509)	SBP	Vitrebond, copolymer, HEMA, dimethacrylate resin, crosslinking radiopaque monomer, dual-cure accelerator, camphorquinone, optimized silane (γMPTES/APTES), ethanol, and water.		
Monobond N, Ivoclar Vivadent, Schaan, Liechtenstein. (LOT: Z02S7Z)	MBN	Alcohol, silane methacrylate, phosphoric acid methacrylate, disulfide methacrylate.		
RelyX Universal Resin Cement, 3M Oral Care, St. Paul, USA. (LOT: 7756479)	-	Dimethacrylate monomers, phosphorylated dimethacrylate adhesion monomers, photoinitiator system, novel amphiphilic redox initiator system, radiopaque fillers, and rheological additives and pigments.		

 Table 1. Materials used in study.

Bis-GMA, bisphenol A glycidyl methacrylate; 10-MDP, 10-methacryloyloxydecyl dihydrogen thiophosphate; HEMA, 2-hydroxyethyl methacrylate; γMPTS, γ methacryloxypropyltrimethoxysilane; γMPTES, γmethacryloxypropyltriethoxysilane; APTES, 3-(aminopropyl)triethoxysilane.

2.1. Specimens' Preparation

LDC (GC InitialLiSi Block, GC Corporation, Tokyo, Japan) blocks were cut into a total of 40 (approximately 5.5 mm \times 6.5 mm) 5 mm thick discs using a water-cooled diamond cutting disc mounted on a computer-controlled precision cutter (IsoMet 5000 Linear Precision Saw, Buehler, Lake Bluff, IL, USA). The top surface of each ceramic disc was wet-polished for 1 min using 600-grit silicon carbide papers mounted on a grinding machine (Automata, Jean Wirtz, Germany) performing 200 revolutions per minute (rpm), and then the discs were subjected to ultrasonic cleaning in distilled water for 5 min using an ultrasonicator (Sonicer, Yoshida Dental Manufacturing. Co., Ltd., Tokyo, Japan). The top surface of each ceramic disc was acid-etched with 5% HF (IPS Ceramic Etching Gel, Ivoclar Vivadent, Schaan, Liechtenstein) for 20 s, then thoroughly washed with water, before being air-dried and randomly distributed into four groups according to the priming material utilized. In Group 1 (control), the LDC discs were not primed. In Group 2, a universal primer (MBN) was applied to the top surface using a microbrush according to the manufacturer's instructions, where it was left for 60 s; subsequently, any remaining

excess was dispersed with a strong stream of air for approximately 5 s. In Group 3, γ MPTScontaining UA (SBU) was mixed with one drop (1-to-1 ratio) of Scotchbond Universal DCA (3M Deutschland GmbH, Neuss, Germany) and immediately applied onto the ceramic surface with a microbrush for 20 s, before being air-dried for approximately 5 s without being light-cured. In Group 4, YMPTES/APTES-containing UA (SBP) was applied onto the ceramic surface with a microbrush for 20 s, before being air-dried for approximately 5 s without being light-cured. For each group, the two discs that received the same surface treatment were cemented using a dual-cure resin cement (RelyX Universal Resin Cement, 3M Oral Care, St. Paul, MN, USA). Equal amounts of resin cement were applied onto the HF-etched or surface-treated (primed) sides before both discs were vertically aligned using a custom-made metal cementation device; then, a cementation force of 1 kg was applied to the discs for 1 min (Figure 1) [24]. The excess cement was immediately and carefully removed with a small-size cotton pellet. The cemented blocks (n = 5/group) were light-cured from each side for 40 s. Then, additional light curing was performed from the top and bottom sides for an additional 40 s each using EliparTM S10 (3M ESPE, St. Paul, MN, USA) operated at 1000 mW/cm², as verified by a hand-held radiometer (Bluephase Meter, Ivoclar Vivadent, Austria). The light-curing tip was kept at approximately 1 mm and at 0 angle to the cemented block. Then, the cemented blocks were kept in distilled water for 24 h at 37 °C before sectioning.



Figure 1. Schematic illustration of specimens' preparation and μ TBS test. (**a**) Sectioning of ceramic blocks into discs; (**b**) acid etching with 5% HF; (**c**) priming of ceramic discs, control (no priming), MBN, SBU, SBP; (**d**) cementation of ceramic discs using RelyX Universal Resin Cement under 1 kg force; (**e**) sectioning of cemented discs into microbeams; (**f**) fixation of microbeams onto μ TBS metal jig; (**g**) using a scanning electron microscope (SEM) to evaluate the failure/fracture mode of microbeams with failure-mode patterns unidentifiable with a light microscope.

2.2. *µTBS*

The cemented ceramic blocks were sectioned into ceramic-resin-ceramic microbeams with a cross-sectional area of approximately 1 mm² (Figure 1e) using a water-cooled diamond cutting disc mounted on a low-speed precision cutter (IsoMet 1000 Precision Cutter, Lake Bluff, IL, USA) for µTBS evaluation. Thirty microbeams were generated within each group. The generated microbeams were examined with a light microscope (Hirox Co., Ltd., Tokyo, Japan) at $15 \times$ magnification before µTBS testing. Defective microbeams were excluded. A digital micrometer (Mitutoyo, Kanagawa, Japan) was used to measure the microbeam cross-sectional surface area at the interface. Then, the microbeams were fixed (glued) to a custom-made metal jig using a cyanoacrylate glue (Loctite 416, Henkel, Düsseldorf, Germany) and attached to a universal testing machine (Instron 5965, Instron Corporation, Norwood, MN, USA) supplied with a 30 kN load cell. Before testing, it was ensured that the alignment of each microbeam was parallel to the direction of the tensile force. The microbeams were stressed under tension at a crosshead speed of 0.5 mm/min until failure or debonding. The µTBS (in megaPascal (MPa)) was calculated for each microbeam by dividing the maximum force (in Newton (N)) at fracture or debonding by the cross-sectional surface area (in mm^2). The μ TBS test was performed in accordance with the protocol described by the Academy of Dental Materials [25].

2.3. Failure/Fracture Mode Assessment

Failure/fracture modes were assessed at $15 \times$ magnification using a light microscope (Hirox Co., Ltd., Tokyo, Japan). Failed/fractured microbeams with unidentifiable failure-mode patterns were ultrasonically cleaned in distilled water for 5 min and dehydrated in ascending concentrations of ethanol, before being gold sputtered for 180 s at 40 mA using a JFC-1100 Fine Coat Ion Sputter (JEOL Ltd., Tokyo, Japan). Gold-sputtered microbeams were then assessed for the failure/fracture mode at low ($75 \times$, $80 \times$) and high magnifications ($500 \times$) using a scanning electron microscope (JSM-6610LV; JEOL Ltd., Tokyo, Japan) operated at 20 kV. Failure/fracture mode patterns were classified as cohesive failure/fracture in the ceramic; adhesive failure/fracture at the resin-ceramic interface; cohesive failure/fracture the resin cement; or mixed failure, including failure/fracture at the ceramic and the resin-ceramic interface. Defective microbeams or those failed/fractured away (>2 mm) from the resin-ceramic interface were excluded.

2.4. Statistical Analysis

The sample size was calculated using G*Power software, version 3.1.9.7 (Heinrich-Heine-Universität Düsseldorf, Düsseldorf, Germany). A total of 20 cemented blocks are required to provide 5 blocks per group. This design achieves 98% power and a 5% significance level. The µTBS values of microbeams generated from the same cemented ceramic block were averaged, thus the cemented ceramic block was considered as the statistical (experimental) unit [26]. Upon the evaluation of the normality of µTBS data using the Kolmogorov–Smirnov test, the one-way analysis of variance (ANOVA) and Tukey's multiple comparison tests were applied in the statistical analysis considering the effect of the priming material on the resin-ceramic µTBS, with *p* < 0.05 considered as being statistically significant. The statistical analysis was performed using R software version 4.1.2 (R Foundation for Statistical Computing, Vienna, Austria).

3. Results

3.1. µTBS

The means \pm standard deviations (SDs) µTBS for the tested groups are presented graphically and numerically in Figure 2. Universal primer (MBN) application showed the highest µTBS (32.21 \pm 4.49 MPa) among all groups. γMPTES/APTES-containing UA (SBP) resulted in significantly higher µTBS (20.41 \pm 6.26 MPa) compared with γMPTS-containing UA (SBU) (10.65 \pm 3.5 MPa) or the control group (8.47 \pm 3.11 MPa). There was

no statistically significant difference between γ MPTS-containing UA (SBU) and the control group. The results of Tukey's multiple comparisons statistical analysis are illustrated in Table 2.



Figure 2. The means \pm standard deviations (SDs) of resin-ceramic microtensile bond strength (µTBS) (expressed in MPa) of Group 1 (control), no priming; Group 2, primed with MBN; Group 3, primed with SBU; and Group 4, primed with SBP. Different superscript letters indicate statistically significant difference (p < 0.05) between groups.

Pair-Wise Groups Comparisons	Mean Difference	Lower Bound	Upper Bound	<i>p</i> -Value	Significance
Control-MBN	23.739	15.581	31.897	< 0.001	*
Control-SBU	2.181	-5.977	10.339	0.869	
Control-SBP	11.942	3.784	20.100	0.003	*
MBN-SBU	21.557	13.399	29.715	< 0.001	*
MBN-SBP	11.797	3.639	19.955	0.004	*
SBU-SBP	-9.761	-17.919	-1.603	0.017	*

Table 2. Results of Tukey's multiple comparisons statistical analysis of µTBS data.

*: indicates statistically significant difference.

3.2. Failure Mode Evaluation

The frequencies of failure modes (expressed as percentages) recorded for the tested μ TBS microbeams are presented in Figure 3. The mixed failures were the most predominant among all groups at 60% (15 microbeams) for the control group, 80% (20 microbeams) for MBN, 68% (17 microbeams) for SBU, and 72% (18 microbeams) for SBP. Adhesive failures occurred next to mixed failures, while cohesive failures were the least among all groups. No cohesive failures in ceramic, or pretest failures, were recorded for any group. The representative SEM photomicrographs of the recorded failure modes are presented in Figure 4. Most of the mixed failure-mode patterns involved an adhesive interfacial failure at the ceramic side and a cohesive failure within the resin cement (Figure 4c,d).



Figure 3. Frequencies of failure modes (expressed as percentages) recorded for tested µTBS microbeams of following groups: control (no priming), MBN, SBU, and SBP.



Figure 4. Representative SEM photomicrographs at low and high magnifications of tested microbeams. (a), at $75 \times$ and (b), at $500 \times$: adhesive failure; (c), at $75 \times$ and (d), at $500 \times$: mixed failure; (e), at $80 \times$ and (f), at $500 \times$: cohesive failure in resin cement. Cr: ceramic surface; gray arrow: ceramic surface covered by adhesive layer; white arrow: adhesive layer, and black arrow: resin cement.

4. Discussion

Bond strength evaluations can be utilized to assess the effectiveness of adhesive procedures and materials [27] before clinical studies can be conducted. This study was designed to evaluate the effect of priming LDC with silane-containing UAs on resin-ceramic bond strength. In contrast to a previous study [23] in which a shear (macro) bond strength test was used to evaluate the bonding of light-cured (polymerized) silane-containing UAs, the current study utilized the μ TBS test due to its better discriminative ability, more even stress distribution during testing, and less cohesive substrate failures compared with macro bond strength tests [28,29]. In addition, the ceramic discs were primed with UAs before they were cemented (under force) using a dual-cure resin-based cement to simulate the intra-oral cementation procedure.

The first null hypothesis was that ceramic surface treatments (universal primer or UAs) would have no significant effect on the resin-ceramic μ TBS. The results of this study indicated that despite γ MPTS-containing UA (SBU) having no effect on the resinceramic μ TBS, both the universal primer (MBN) and γ MPTES/APTES-containing UA (SBP) considerably improved the resin-ceramic μ TBS. Thus, the first hypothesis was rejected.

Silane-containing universal primers, such as MBN, would not promote adequate adhesion to glass-ceramics without a prior HF etching step [30]. That is why the effect of MBN or silane-containing UAs should not be evaluated without prior HF etching. The application of HF on glass-ceramics results in the formation of tetrafluorosilane, which reacts with HF to form a soluble hydrofluorosilicic acid [12]. Then, a considerable amount of the glass matrix at the etched ceramic surface is dissolved and can be easily rinsed away with water. This not only results in the creation of surface microirregularities but also results in the formation of hydroxyl groups on the etched ceramic surface [9]. Upon the surface treatment of HF-etched glass-ceramic with silane-containing primers, a condensation reaction between such hydroxyl groups and the silanol groups of activated silane occurs, representing one aspect of ceramic bonding [9,31]. The other aspect is the reaction between organofunctional groups in the silane molecule and functional (methacrylate) groups of the methacrylate-based materials, such as resin-based cements or adhesives [9,31]. The universal primers utilized in this study, such as MBN, contain large percentages of organic solvents, such as ethanol or acetone [11,32]. Upon air-drying the primer layer applied on the ceramic surface, much of the solvent will be evaporated, resulting in the formation of a silane layer on the ceramic surface, which is a crucial factor affecting the resin-ceramic bond strength [31]. In contrast, compared with MBN, the composition of silane-containing UAs is much more complex because they are primarily manufactured to bond resin-based composite materials to the tooth structure [33]. To specify, other components within the UA composition, such as 10-MDP, Bis-GMA, HEMA, dimethacrylate resin, crosslinker monomers, and other components, cannot be eliminated after the application of UA. Thus, it is assumed that despite the ability of UAs to flow effectively into the microirregularities created by HF etching [21], the formation of a silane layer deposited onto the ceramic surface seems less likely, even with the effective air-drying of such UAs [20]. This can partly explain the inferior priming capacity of UAs compared with MBN. Meanwhile, UAs are acidic in nature [14,34], which might have affected the chemical stability of silane molecules within the UAs' formulations, impairing their priming potential [21] and resulting in a low resin-ceramic bond strength. γ MPTS-containing UA (SBU) did not have any impact on resin-ceramic bonding. This is in accordance with a recent meta-analysis that indicated that γMPTS-containing UAs do not have a priming capacity similar to silane-containing or universal primers [35]. In contrast, YMPTES/APTES-containing UA (SBP) not only significantly improved the resin-ceramic µTBS compared with the control group but also resulted in significantly higher µTBS than the γMPTS-containing UA (SBU). Therefore, the second null hypothesis, that there would be no significant difference between the resin-ceramic µTBS obtained after the use of either of the two silane-containing UAs tested, was also rejected. The different bond strengths obtained can be explained—in part—by the better priming capacity of SBP due to its optimized (YMPTES/APTES) silane technology. Combining the effects of the silanol-groups of both γ MPTES and APTES molecules, as well as the amino groups of the APTES molecule, means they can simultaneously interact with the hydroxyl groups on the glass-ceramic surface [36], increasing UA's priming capacity; thus, the resin-ceramic μ TBS is enhanced. The APTES molecule within SBP forms hydrogen bonds at the HF-etched glass-ceramic surface and reacts with silanol groups, resulting in the formation of amino-silanol groups [37]. In addition, the APTES molecule can act as a siloxane intermolecular crosslinker [36], which can increase the density of the silane interaction layer and enhance the bonding to the glass-ceramic material [23]. Moreover, the Si–O–C₂H₅ group within the γ MPTES molecule in SBP hydrolyzes slower than the Si–O–CH₃ group within the γ MPTS molecule in SBU [38], which may have decreased the dehydration condensation of silanol groups [23]. Another plausible explanation for the superior resin-ceramic μ TBS obtained when SBP is used as a primer may be its improved dual-cure compatibility with the luting resin cement used (RelyX Universal Resin Cement). However, further in-depth investigations are required to prove this speculation.

Despite the precision of the μ TBS test employed in this study, it is a technique-sensitive method associated with the generation of interfacial stress during specimens' sectioning. Such stress can result in failure of low-performance adhesive interfaces or brittle substrates [29]. Multiple factors, such as specimens' alignment in relation to the direction of tensile force, shape and dimensions, gripping jig, and testing machine, can also influence the outcome of the μ TBS evaluation [25,39]. The outcome (μ TBS data) should be interpreted in combination with the corresponding failure modes because a statistical evaluation of µTBS data can be affected by the inclusion or censoring microbeams with specific failure modes [40,41]. Both a light microscope and SEM were utilized to assess the failure mode patterns of the tested microbeams. The fractographic evaluation using SEM is more illustrative and can be performed in higher magnifications compared with using a light microscope [25]. We found that mixed failure patterns (Figure 4c,d) were the most predominant in all the groups. The control (no priming) group presented a higher incidence of adhesive failures compared with the other groups, which may be due to the relatively low μ TBS. The failure mode patterns of the tested groups were affected by the setup of the μ TBS test. Although sectioning the cemented ceramic blocks into microbeams is laboratory-intensive and technique-sensitive work, testing the ceramic-resin-ceramic microbeams employed in this study not only had the advantage of solely evaluating the resin-ceramic interface [24] but also eliminated the incidence of cohesive failures within the ceramic material, which can be explained by the high mechanical properties of LDC [42] and the homogenous stress distribution at the interface. Overall, the γ MPTES/APTEScontaining UA (SBP) showed promising performance as an LDC primer before cementation with a dual cure resin cement. However, this study has some limitations, including the lack of long-term artificial aging (water storage and thermocycling) of the microbeams and the use of one ceramic material and one resin cement. Moreover, mechanical loading before bond strength evaluation cannot be performed because of the geometry of the cemented blocks. Thus, the results of this study should be considered with much caution. In fact, in-depth chemical analyses of γ MPTES/APTES ratio applied in SBP in addition to the reaction between YMPTES/APTES-containing UA (SBP) and HF-etched glass-ceramic are required to confirm its positive impact on resin-ceramic bond strength.

5. Conclusions

The effect of silane-containing UAs on resin-ceramic μ TBS is material-dependent. γ MPTS-containing UA (SBU) did not affect the resin-ceramic μ TBS. Although γ MPTES/ APTES-containing UA (SBP) improved bonding to LDC compared with γ MPTS-containing UA (SBU) or the control group (no priming), it resulted in considerably lower μ TBS than a universal silane-containing primer (MBN). The priming of LDC with either of the UAs tested cannot be considered as an alternative to a separate silanization (priming) step using a universal silane-containing primer. **Author Contributions:** Conceptualization, M.M.A.; methodology, M.M.A.; software, A.A. (Ali Alrahlah); validation, A.S.A.; formal analysis, M.M.A.; investigation, B.A. and S.A.; resources, A.A. (Abdullah Alshehri); data curation, M.M.A.; writing—original draft preparation, M.M.A. and F.A.; writing—review and editing, M.A.S.; visualization, M.M.A. and A.R.; supervision, A.A. (Ali Alrahlah); project administration, F.A.; funding acquisition, A.A. (Abdullah Alshehri). All authors have read and agreed to the published version of the manuscript.

Funding: The authors are grateful to the Deanship of Scientific Research, King Saud University, for funding this study through the Vice Deanship of Scientific Research Chairs and the Research Chair for Dental and Oral Rehabilitation, Engineer Abdullah Bugshan.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: The data presented in this study are available upon request from the corresponding author.

Conflicts of Interest: The authors declare no conflict of interest.

References

- 1. van den Breemer, C.R.; Gresnigt, M.M.; Cune, M.S. Cementation of Glass-Ceramic Posterior Restorations: A Systematic Review. *BioMed Res. Int.* 2015, 2015, 148954. [CrossRef] [PubMed]
- Politano, G.; Van Meerbeek, B.; Peumans, M. Nonretentive Bonded Ceramic Partial Crowns: Concept and Simplified Protocol for Long-lasting Dental Restorations. J. Adhes. Dent. 2018, 20, 495–510. [CrossRef] [PubMed]
- Phark, J.-H.; Duarte, S., Jr. Microstructural considerations for novel lithium disilicate glass ceramics: A review. J. Esthet. Restor. Dent. 2022, 34, 92–103. [CrossRef] [PubMed]
- Awad, M.M.; Alqahtani, H.; Al-Mudahi, A.; Murayshed, M.S.; Alrahlah, A.; Bhandi, S.H. Adhesive Bonding to Computer-aided Design/ Computer-aided Manufacturing Esthetic Dental Materials: An Overview. J. Contemp. Dent. Pract. 2017, 18, 622–626. [CrossRef] [PubMed]
- Malysa, A.; Wezgowiec, J.; Grzebieluch, W.; Danel, D.P.; Wieckiewicz, M. Effect of Thermocycling on the Bond Strength of Self-Adhesive Resin Cements Used for Luting CAD/CAM Ceramics to Human Dentin. Int. J. Mol. Sci. 2022, 23, 745. [CrossRef]
- 6. Malysa, A.; Wezgowiec, J.; Orzeszek, S.; Florjanski, W.; Zietek, M.; Wieckiewicz, M. Effect of Different Surface Treatment Methods on Bond Strength of Dental Ceramics to Dental Hard Tissues: A Systematic Review. *Molecules* **2021**, *26*, 1223. [CrossRef]
- Akar, T.; Dündar, A.; Kırmalı, Ö.; Üstün, Ö.; Kapdan, A.; Er, H.; Kuştarcı, A.; Er, K.; Yılmaz, B. Evaluation of the shear bond strength of zirconia to a self-adhesive resin cement after different surface treatment. *Dent. Med. Probl.* 2021, 58, 463–472. [CrossRef]
- Lubauer, J.; Belli, R.; Peterlik, H.; Hurle, K.; Lohbauer, U. Grasping the Lithium hype: Insights into modern dental Lithium Silicate glass-ceramics. *Dent. Mater.* 2022, 38, 318–332. [CrossRef]
- Tian, T.; Tsoi, J.K.-H.; Matinlinna, J.P.; Burrow, M.F. Aspects of bonding between resin luting cements and glass ceramic materials. Dent. Mater. 2014, 30, e147–e162. [CrossRef]
- 10. Della Bona, A.; Anusavice, K.J.; Mecholsky, J.J., Jr. Failure analysis of resin composite bonded to ceramic. *Dent. Mater.* **2003**, *19*, 693–699. [CrossRef]
- Matinlinna, J.P.; Lassila, L.V.; Özcan, M.; Yli-Urpo, A.; Vallittu, P.K. An introduction to silanes and their clinical applications in dentistry. *Int. J. Prosthodont.* 2004, 17, 155–164. [PubMed]
- Lung, C.Y.; Matinlinna, J.P. Aspects of silane coupling agents and surface conditioning in dentistry: An overview. *Dent. Mater.* 2012, 28, 467–477. [CrossRef] [PubMed]
- Shen, C.; Oh, W.S.; Williams, J.R. Effect of post-silanization drying on the bond strength of composite to ceramic. J. Prosthet. Dent. 2004, 91, 453–458. [CrossRef] [PubMed]
- Nagarkar, S.; Theis-Mahon, N.; Perdigão, J. Universal dental adhesives: Current status, laboratory testing, and clinical performance. J. Biomed. Mater. Res. B Appl. Biomater. 2019, 107, 2121–2131. [CrossRef] [PubMed]
- 15. Cuevas-Suárez, C.E.; de Oliveira da Rosa, W.L.; Vitti, R.P.; da Silva, A.F.; Piva, E. Bonding Strength of Universal Adhesives to Indirect Substrates: A Meta-Analysis of in Vitro Studies. *J. Prosthodont.* **2020**, *29*, 298–308. [CrossRef]
- Kalavacharla, V.K.; Lawson, N.C.; Ramp, L.C.; Burgess, J.O. Influence of Etching Protocol and Silane Treatment with a Universal Adhesive on Lithium Disilicate Bond Strength. *Oper. Dent.* 2015, 40, 372–378. [CrossRef]
- Makishi, P.; Andre, C.B.; Silva, J.L.; Bacelar-Sa, R.; Correr-Sobrinho, L.; Giannini, M. Effect of Storage Time on Bond Strength Performance of Multimode Adhesives to Indirect Resin Composite and Lithium Disilicate Glass Ceramic. *Oper. Dent.* 2016, 41, 541–551. [CrossRef]
- 18. Yao, C.; Yang, H.; Yu, J.; Zhang, L.; Zhu, Y.; Huang, C. High Bond Durability of Universal Adhesives on Glass Ceramics Facilitated by Silane Pretreatment. *Oper. Dent.* **2018**, *43*, 602–612. [CrossRef]

- Yao, C.; Zhou, L.; Yang, H.; Wang, Y.; Sun, H.; Guo, J.; Huang, C. Effect of silane pretreatment on the immediate bonding of universal adhesives to computer-aided design/computer-aided manufacturing lithium disilicate glass ceramics. *Eur. J. Oral Sci.* 2017, 125, 173–180. [CrossRef]
- Awad, M.M.; Albedaiwi, L.; Almahdy, A.; Khan, R.; Silikas, N.; Hatamleh, M.M.; Alkhtani, F.M.; Alrahlah, A. Effect of universal adhesives on microtensile bond strength to hybrid ceramic. *BMC Oral Health* 2019, *19*, 178. [CrossRef]
- Yoshihara, K.; Nagaoka, N.; Sonoda, A.; Maruo, Y.; Makita, Y.; Okihara, T.; Irie, M.; Yoshida, Y.; Van Meerbeek, B. Effectiveness and stability of silane coupling agent incorporated in 'universal' adhesives. *Dent. Mater.* 2016, 32, 1218–1225. [CrossRef] [PubMed]
- 22. Alrahlah, A.; Awad, M.M.; Vohra, F.; Al-Mudahi, A.; Al Jeaidi, Z.A.; Elsharawy, M. Effect of self etching ceramic primer and universal adhesive on bond strength of lithium disilicate ceramic. *J. Adhes. Sci. Technol.* **2017**, *31*, 2611–2619. [CrossRef]
- Yao, C.; Ahmed, M.H.; De Grave, L.; Yoshihara, K.; Mercelis, B.; Okazaki, Y.; Van Landuyt, K.L.; Huang, C.; Van Meerbeek, B. Optimizing glass-ceramic bonding incorporating new silane technology in an experimental universal adhesive formulation. *Dent. Mater.* 2021, *37*, 894–904. [CrossRef] [PubMed]
- Südbeck, J.M.; Jacker-Guhr, S.; Lührs, A.K. Do Different Pretreatment Methods Influence the Microtensile Bond Strength of Composite Cements to Silicate Ceramic? J. Adhes. Dent. 2021, 23, 335–345. [CrossRef] [PubMed]
- Armstrong, S.; Breschi, L.; Özcan, M.; Pfefferkorn, F.; Ferrari, M.; Van Meerbeek, B. Academy of Dental Materials guidance on in vitro testing of dental composite bonding effectiveness to dentin/enamel using micro-tensile bond strength (μTBS) approach. *Dent. Mater.* 2017, *33*, 133–143. [CrossRef]
- Eckert, G.J.; Platt, J.A. A statistical evaluation of microtensile bond strength methodology for dental adhesives. *Dent. Mater.* 2007, 23, 385–391. [CrossRef]
- 27. El Zohairy, A.A.; De Gee, A.J.; Mohsen, M.M.; Feilzer, A.J. Microtensile bond strength testing of luting cements to prefabricated CAD/CAM ceramic and composite blocks. *Dent. Mater.* **2003**, *19*, 575–583. [CrossRef]
- 28. De Munck, J.; Mine, A.; Poitevin, A.; Van Ende, A.; Cardoso, M.V.; Van Landuyt, K.L.; Peumans, M.; Van Meerbeek, B. Metaanalytical review of parameters involved in dentin bonding. *J. Dent. Res.* **2012**, *91*, 351–357. [CrossRef]
- 29. Salz, U.; Bock, T. Testing adhesion of direct restoratives to dental hard tissue—A review. *J. Adhes. Dent.* **2010**, *12*, 343–371. [CrossRef]
- 30. Huang, T.H.; Kao, C.T. The shear bond strength of composite brackets on porcelain teeth. *Eur. J. Orthod.* **2001**, *23*, 433–439. [CrossRef]
- Matinlinna, J.P.; Lung, C.Y.K.; Tsoi, J.K.H. Silane adhesion mechanism in dental applications and surface treatments: A review. Dent. Mater. 2018, 34, 13–28. [CrossRef] [PubMed]
- 32. Zakir, M.; Ashraf, U.; Tian, T.; Han, A.; Qiao, W.; Jin, X.; Zhang, M.; Tsoi, J.K.-H.; Matinlinna, J.P. The Role of Silane Coupling Agents and Universal Primers in Durable Adhesion to Dental Restorative Materials—A Review. *Curr. Oral Health Rep.* **2016**, *3*, 244–253. [CrossRef]
- 33. Chen, C.; Niu, L.N.; Xie, H.; Zhang, Z.Y.; Zhou, L.Q.; Jiao, K.; Chen, J.H.; Pashley, D.H.; Tay, F.R. Bonding of universal adhesives to dentine–Old wine in new bottles? *J. Dent.* 2015, 43, 525–536. [CrossRef] [PubMed]
- 34. Papadogiannis, D.; Dimitriadi, M.; Zafiropoulou, M.; Gaintantzopoulou, M.D.; Eliades, G. Universal Adhesives: Setting Characteristics and Reactivity with Dentin. *Materials* **2019**, *12*, 1720. [CrossRef]
- Lima, R.B.W.; Muniz, I.A.F.; Campos, D.E.S.; Murillo-Gómez, F.; Andrade, A.K.M.; Duarte, R.M.; de Souza, G.M. Effect of universal adhesives and self-etch ceramic primers on bond strength to glass ceramics: A systematic review and meta-analysis of in vitro studies. J. Prosthet. Dent. 2022. [CrossRef]
- Acres, R.G.; Ellis, A.V.; Alvino, J.; Lenahan, C.E.; Khodakov, D.A.; Metha, G.F.; Andersson, G.G. Molecular Structure of 3-Aminopropyltriethoxysilane Layers Formed on Silanol-Terminated Silicon Surfaces. J. Phys. Chem. C 2012, 116, 6289–6297. [CrossRef]
- 37. Park, S.-J.; Jin, J.-S.; Lee, J.-R. Influence of silane coupling agents on the surface energetics of glass fibers and mechanical interfacial properties of glass fiber-reinforced composites. *J. Adhes. Sci. Technol.* **2000**, *14*, 1677–1689. [CrossRef]
- Xiong, M.; Zhang, K.; Chen, Y. ATRP of 3-(triethoxysilyl)propyl methacrylate and preparation of "stable" gelable block copolymers. *Eur. Polym. J.* 2008, 44, 3835–3841. [CrossRef]
- Roeder, L.; Pereira, P.N.; Yamamoto, T.; Ilie, N.; Armstrong, S.; Ferracane, J. Spotlight on bond strength testing–unraveling the complexities. *Dent. Mater.* 2011, 27, 1197–1203. [CrossRef]
- 40. Comba, L.; Tichy, A.; Bradna, P. The Effect of Censoring on the Statistical Evaluation of Composite-to-Composite Bond Strength. *J. Adhes. Dent.* **2020**, 22, 183–193. [CrossRef]
- 41. Franz, A.; Lettner, S.; Watts, D.C.; Schedle, A. Should statistical analysis of bond-strength data include or exclude cohesive failures? *Dent. Mater.* **2022**, *38*, 1942–1946. [CrossRef] [PubMed]
- da Silva, S.E.G.; de Araújo, G.M.; Souza, K.B.; Moura, D.M.D.; Aurélio, I.L.; May, L.G.; Vila-Nova, T.E.L.; Zhang, Y.; de Assunção, E.S.R.O. Biaxial flexure strength and physicochemical characterization of a CAD/CAM lithium disilicate ceramic: Effect of etching time, silane, and adhesive applications. *Clin. Oral Investig.* 2022, 26, 6753–6763. [CrossRef] [PubMed]

Disclaimer/Publisher's Note: The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.