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High Bond Durability of Universal Adhesives on Glass Ceramics Facilitated by Silane Pretreatment

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Clinical Relevance

Additional silane treatment prior to the application of universal adhesive is necessary to improve the bonding longevity to lithium disilicate glass ceramic.

SUMMARY

Objective: This study aimed to investigate the long-term effectiveness of ceramic–resin bonding with universal adhesives in non–silane-pretreated and silane-pretreated modes after 10,000 cycles of thermal aging.

Methods and Materials: All Bond Universal, Adhese Universal, Clearfil Universal Bond, and Single Bond Universal were selected. Etched lithium disilicate glass ceramics were prepared, randomly assigned to groups, and pretreated with or without ceramic primer containing silane coupling agent prior to the application of universal adhesive (ie, silane-pretreated or non–silane-pretreated mode). The shear bond strength (SBS), microleakage, and field-emission scanning electron microscopy images of the ceramic–resin interfaces were examined after 24 hours of water storage or 10,000 thermal cycles. Light microscopy and confocal laser scanning microscopy (CLSM) were performed to analyze marginal sealing ability.

Results: SBS and microleakage percentage were significantly affected by bonding procedure (non–silane-pretreated or silane-pretreated mode) and aging (24 hours or 10,000 thermal cycles). After the universal adhesives in the non–silane-pretreated mode were aged, SBS significantly decreased and microleakage percentage increased. By contrast, the SBS of

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Adhese Universal, Clearfil Universal Bond, and Single Bond Universal decreased, and the microleakage percentage of all of the adhesives increased in the silane-pretreated mode. However, after aging, the SBS of the silane-pretreated groups were higher and their microleakage percentages lower than those of the non–pretreated groups. In the non–silane-pretreated mode, adhesive failure was dominant and gaps between composite resin and the adhesive layer were significant when observed with CLSM.

Conclusions: The simplified procedure reduced the ceramic–resin bonding effectiveness of universal adhesives after aging, and additional silane pretreatment helped improve the long-term durability.

INTRODUCTION

Dental glass ceramic has gained considerable interest from both dentists and patients because of outstanding esthetics and biocompatibility. Lithium disilicate glass ceramic, which is a third-generation ceramic system, was introduced in 1998 for single- and multiple-unit frameworks[1] and is a widely used dental restorative material.[2] A glass ceramic restorative material is generally bonded to tooth substrates by using an adhesive system. Although the properties of current adhesives have been remarkably enhanced,[3-5] glass ceramic–resin bonding is limited by factors such as bulk fractures and marginal deficiencies.[6] Therefore, effective and stable bonding are key elements in the application of ceramic restorations and have been intensively explored in clinical practice.

Etching with hydrofluoric acid and application of a ceramic primer containing a silane coupling agent is an effective approach for glass ceramic bonding because of the silica base of glass ceramics.[7] The glass matrix is dissolved by the hydrofluoric acid and a porous structure for micromechanical retention is produced. Silane is an effective adhesion promoter[8] that reacts and couples with inorganic and organic materials.[9] However, the clinical application of glass ceramic treatment is impeded by time-consuming and complex steps. Thus, an efficient and simplified method must be established in the adhesive protocol, not only to maintain stable bonding, but also to reduce clinical operation time.

Universal adhesive has emerged as a promising bonding candidate because it can be applied to various substrates, such as enamel, dentin,[10,11] zirconia ceramics,[12] and glass ceramics.[13] For a simplified glass ceramic bonding procedure, some universal adhesive manufacturers claim that additional silane pretreatment is unnecessary because a silane coupling agent is incorporated in their products. Although universal adhesives have the advantage of convenient use, this material cannot guarantee the long-term clinical success of adhesive bonding for restorations. Furthermore, the durability and efficiency traits of adhesive bonding have yet to be enhanced. Therefore, in-depth *in vitro* and *in vivo* studies and clinical trials must be conducted to verify the longevity of glass ceramic–resin bonds produced by universal adhesives.

Thermal cycling with simulated intraoral temperature is commonly used to evaluate the bond durability and degradation of an adhesive system.[14,15] In particular, a widely accepted criterion is that 10,000 cycles are equivalent to the clinical physiological aging of one year.[16] To the extent of our knowledge, although numerous studies have investigated the longevity of dentin-resin bonding produced by universal adhesives,[17-19] few studies have explored the ceramic–resin bonding durability with universal adhesives. Limited reports have verified whether silane incorporated in universal adhesives is a sufficient substitute for additional silane pretreatment in long-term applications.

Therefore, this study aimed to evaluate the effect of additional silane pretreatment on the durability of ceramic–resin bonding with universal adhesives after 10,000 thermal cycles of aging. Two silane-containing and two silane-free universal adhesives were used in the present study. Our tested null hypotheses were as follows: 1) there is no significant difference in the bonding performance of universal adhesive on lithium disilicate glass ceramics between non–silane-pretreated and silane-pretreated modes and 2) there is no significant difference in ceramic–resin bonding with universal adhesive after 24 hours of water storage and after 10,000 thermal cycles.

METHODS AND MATERIALS

Specimen Preparation and Experimental Design

Two hundred forty-two lithium disilicate glass ceramic specimens (e.max CAD, Ivoclar Vivadent, Schaan, Liechtenstein) with dimensions of 8 mm × 7 mm × 2 mm were prepared by using a low-speed cutting device (Isomet, Buehler, Evanston, IL, USA). The specimens were sintered in accordance with the manufacturer's instructions. The ceramic specimens were ground flat using silicon carbide (SiC) paper through 3000-grit and polished with a soft cloth and 0.5-µm-grit diamond paste in distilled water. The ceramics were etched with 9% hydrofluoric acid (Ultradent Products, Inc, South Jordan, UT, USA) for 90 seconds and thoroughly rinsed with water for 15 seconds.

A total of 240 etched ceramic blocks were randomly divided into four universal adhesives groups: All Bond Universal (ABU; Bisco, Schaumburg, IL, USA), Adhese Universal (ADU; Ivoclar Vivadent), Clearfil Universal Bond (CUB; Kuraray Noritake Dental, Tokyo, Japan), and Single Bond Universal (SBU; 3M ESPE, St. Paul, MN, USA). The compositions of the adhesives are listed in Table 1. The etched blocks were directly treated with adhesive (ie, non–silane-pretreated mode, eg, ABU, n=30) or pretreated with ceramic primer containing silane (RelyX ceramic primer; 3M ESPE) for 20 seconds, followed by adhesive treatment (ie, silane-pretreated mode, eg, S-ABU, n=30). The bonding procedure of adhesive systems (Table 1) was conducted strictly in accordance with the manufacturer's instruction. The adhesive was light polymerized with a high-intensity light-emitting diode curing light (1100 mW/cm^2; Ivoclar Vivadent).

Shear Bond Strength

After light polymerizing each adhesive, a Tygon tube (inner diameter: 3 mm; height: 4 mm) was placed on the ceramic surface. Then, the composite resin (Charisma, Heraeus Kulzer, Hanau, Germany) was placed into the tube with 2 layers up to a height of 4 mm. Each resin increment of 2 mm was light-polymerized for 20 seconds using a light-emitting diode curing light (1100 mW/cm^2; Ivoclar Vivadent). Prior to shear bond strength (SBS) testing, the tubes were removed to reveal the composite cylinders. Eighty etched ceramic specimens were randomly selected from each adhesive in the non–silane-pretreated (eg, ABU, n=10) and silane-pretreated modes (eg, S-ABU, n=10), and these specimens were used for SBS evaluation with storage in distilled water (37°C) for 24 hours. Another 80 specimens were randomly selected from each adhesive in the non–silane-pretreated (eg, ABU, n=10) and silane-pretreated modes (eg, S-ABU, n=10), and these specimens were treated with 10,000 thermocycles between two water baths at 5°C and 55°C, with a dwell time of 60 seconds for each temperature and an exchange time of 10 seconds between the baths (Thermo-cycler, Thermo Fisher Scientific, Waltham, MA, USA).

All specimens were placed in a steel fixture in a universal testing machine (ZY-100K, Yangzhou, Jiangsu, China) and subjected to shear stress at a constant crosshead speed of 1 mm/min.[20,21] The peak load that created a fracture was recorded. SBS (MPa) was calculated by dividing the peak load during interface failure by the adhesive bonding area.

Failure Mode Analysis

After debonding, the fractured samples were sputter coated with Au–Pd alloy (JFC-1600, JEOL, Tokyo, Japan) and analyzed by field-emission scanning electron microscopy (FESEM; Zeiss, Sigma, Germany) at 24×, 5000×, and 20,000× magnifications. The failure modes of the fractured interfaces were classified as adhesive failures on the ceramic surface or cohesive failures in the adhesive or composite resin.[22,23] Image J analysis software (National Institutes of Health Frederick, MD, USA) was used to calculate the adhesive and cohesive failure percentages of the fracture surfaces.

Microleakage Evaluation

Forty etched ceramic specimens were randomly selected from each adhesive in the non–silane-pretreated (eg, ABU, n=5) and silane-pretreated modes (eg, S-ABU, n=5), and these specimens were used for microleakage evaluation with storage in distilled water (37°C) for 24 hours. Another 40 specimens were randomly selected from each adhesive in the non–silane-pretreated (eg, ABU, n=5) and silane-pretreated modes (eg, S-ABU, n=5), and these specimens were treated with 10,000 thermocycles. After the adhesive was light polymerized, 2-mm-thick composite resin was placed onto the ceramic and polymerized for 40 seconds using a light-emitting diode curing light (1100 mW/cm^2; Ivoclar Vivadent).

The bonded specimens were sectioned longitudinally across the interface, obtaining slabs with dimensions of 7 mm × 4 mm × 1 mm. Peripheral slabs with excessive or insufficient resin amount at the interface were abandoned. Finally, 80 slabs were collected, stored in distilled water (37°C) for 24 hours or subjected to 10,000 thermocycles, coated with two consecutive nail varnish layers (except for an area of approximately 0.5 mm away from the interface), and immersed in 0.5% basic fuchsin solution[24] at 37°C for 24 hours. Afterward, the

Table 1: *Composition and Application Procedures of the Universal Adhesive Systems Used in This Study*

Universal Adhesive (Batch No.)	PH	Composition	Application Procedures
All-Bond Universal-ABU (1200006111)	3.2	Bis-GMA, HEMA, MDP, initiators, ethanol, water	Apply for 10-15 seconds, mild air dry for at least 10 seconds, light cure for 10 seconds
Adhese Universal-ADU (SS4248)	2.5-3.0	Bis-GMA, HEMA, MDP, CQ, D3MA, MCAP, highly dispersed silicon dioxide, ethanol, water	Apply for 20 seconds, mild air dry for five seconds, light cure for 10 seconds
Clearfil Universal Bond-CUB (01416)	2.3	Bis-GMA, HEMA, MDP, CQ, colloidal silica, silane coupling agent, ethanol, water, hydrophilic aliphatic dimethacrylate	Apply for 10 seconds, mild air dry for five seconds, light cure for 10 seconds
Single Bond Universal-SBU (D-82229)	2.7	HEMA, MDP, silane, filler, dimethacrylate resins, initiators, Vitrebond copolymer, ethanol, water	Mix Single Bond Universal dual cure activator with Single Bond universal in a ratio of 1-to-1, apply adhesive mixture for 20 seconds, dry gently for five seconds, light cure for 10 seconds

Abbreviations: Bis-GMA, bisphenol A diglycidylmethacrylate; CQ, camphorquinone; D3MA, decandiol dimethadrylate; HEMA, 2-hydroxyethyl methacrylate; MCAP, methacrylated carboxylic acid polymer; MDP, 10-methacryloyloxydecyl dihydrogen phosphate.

specimens were fixed on glass slides,[25] subsequently polished with SiC paper and distilled water through 5000-grit, and cleaned ultrasonically. The ceramic–resin interface was examined with light microscopy (LM; DP72, Olympus, Tokyo, Japan). Ten images with 100× magnification were randomly captured for each subgroup (eg, ABU-24h). For quantitative analysis, Image J analysis software was employed to determine the microleakage percentage at the adhesive interface.

Confocal Laser Scanning Microscopy

CUB (silane-containing universal adhesive) was evaluated. One etched ceramic block directly received CUB, whereas another block was pretreated with additional silane and then the CUB applied. Rhodamine B was added to the adhesive for confocal microscopy evaluation.[26] After application of 2-mm of composite resin, the specimens were sectioned into 7-mm × 4-mm × 1-mm slabs with a low-speed diamond saw. The peripheral slabs were discarded, and a middle slab was collected from each group. After being stored in distilled water at 37°C for 24 hours and subjected to 5000 and 10,000 thermocycles, the slabs were observed using a Fluoview Ver.4.2 confocal laser scanning microscope (CLSM; FV1000, Olympus). The same observation place was strictly implemented for each slab under the above three conditions. Emission fluorescence was conducted at 559 nm. Topographic single projection was established from the serial images obtained at a depth of 20 μm.[27] The captured images were analyzed with Imaris 7.2.3 software (v.7.2.3, Bitplane, Zurich, Switzerland). The configuration was standardized at the same level for the entire investigation.

Statistical Analysis

SBS and microleakage percentage were presented as mean and standard deviation values. SBS and microleakage percentage were separately analyzed for each universal adhesive by conducting two-way analysis of variance (ANOVA), with "bonding procedure" and "aging" as independent variables. Tukey post hoc test was applied for multiple comparisons. The significance level was set at $\alpha = 0.05$. Statistical analysis was calculated using SPSS version 23.0 (SPSS, Chicago, IL, USA).

RESULTS

SBS

Figure 1A shows the SBS results for the four different universal adhesives bonded to lithium disilicate glass ceramics. For each adhesive, two-way ANOVA revealed that the SBS was significantly affected by additional silane pretreatment and thermocycling aging ($p<0.001$). The interaction between the two factors was significant (for ABU, CUB, and SBU, $p<0.001$; for ADU, $p=0.018$). For pairwise comparisons within the factor silane pretreatment strategy, after 24 hours of water storage, the SBS of each universal adhesive in the silane-pretreated mode significantly differed from those in the non–silane-pretreated mode ($p<0.05$). After aging, the SBS of the four adhesives under silane-pretreated mode was significantly higher than those of the non–pretreated ones ($p<0.001$). For pairwise comparisons within the factor aging, the SBS of all four adhesives in the non–silane-pretreated mode significantly decreased ($p<0.001$). No significant differences were observed in S-ABU between the 24-hour soaked samples and those subjected to 10,000 thermocycling aging ($p=0.84$); by contrast,

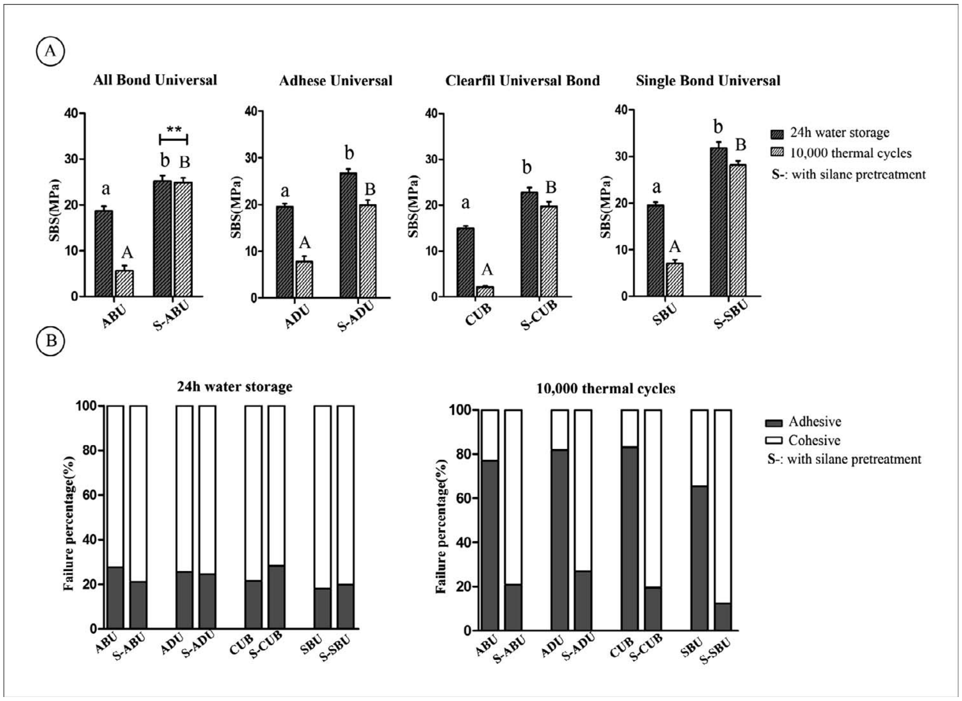

Figure 1. *Effectiveness of bonding strategy and aging on SBS of lithium disilicate glass ceramic with four universal adhesives. (A): SBS was analyzed separately for each adhesive using two-factor ANOVA. For each adhesive, columns labeled with the same lowercase letters or uppercase letters are not significantly different in pairwise comparisons within "bonding strategy" (*p*>*0.05), and columns distinguished with asterisks are not significantly different in pairwise comparisons within "aging" (*p*>*0.05). (B): Failure mode distribution in the study groups. Failure within the ceramic was classified as adhesive failure mode, whereas failure within the resin or the adhesive was regarded as cohesive failure mode.*

the performance of S-ADU, S-CUB, and S-SBU declined in SBS ($p<0.05$).

Failure Mode Analysis of Debonded Specimens

Figure 1B shows the failure percentage of the different groups in various modes after SBS testing. For immediate groups, the predominant failure mode of the specimens was cohesive failure, regardless of whether an additional silane pretreatment was performed or not. Nevertheless, for aging groups, adhesive failure increased for all groups in the non–silane-pretreated mode, whereas cohesive failure was the predominant mode for groups in the silane-pretreated mode.

Figure 2 provides representative FESEM images of debonded specimens in ABU, S-ABU, SBU, and S-SBU groups after 24 hours of SBS testing. An insignificant difference in morphological appearance was observed between silane-pretreated and non-pretreated groups. The debonded specimens displayed predominant cohesive failure at low magnification, and the adhesives and the composites resins were visible at high magnification.

Representative FESEM images of debonded specimens in ADU, S-ADU, CUB, and S-CUB groups after SBS testing of 10,000 runs with thermocycling are shown in Figure 3. The debonded specimens of the groups in the non–silane-pretreated mode exhibited predominant adhesive failure at low magnification. The fracture mainly exposed the ceramic crystals. Particularly, at high magnification, the crystal structures displayed a round pattern that differed from the etched ceramic surface. For groups in the silane-pretreated mode, FESEM images showed dominant cohesive failure. The bond appeared to fracture underneath the adhesives and the resins. In addition, cleavages and small voids were observed in the debonded interface of the S-CUB group at high magnification.

Microleakage

The results of microleakage percentage for the four different universal adhesives bonded to lithium disilicate glass ceramics are presented separately in Figure 4A. For each adhesive, two-way ANOVA showed that the microleakage percentage was considerably affected by silane pretreatment strategy and thermocycling aging ($p<0.001$). Nevertheless, the interaction between the two factors was insignificant ($p>0.05$). Then, experiments for main effects were conducted. During a 24-hour evaluation, the microleakage percentages of the CUB and SBU groups in the silane-pretreated mode significantly differed from those in non–silane-pretreated mode ($p<0.05$). After aging, the microleakage percentages of all universal adhesives in the silane-pretreated mode were significantly lower than those of the non–silane-pretreated ones ($p<0.05$). In addition, the percentage of all adhesives significantly increased

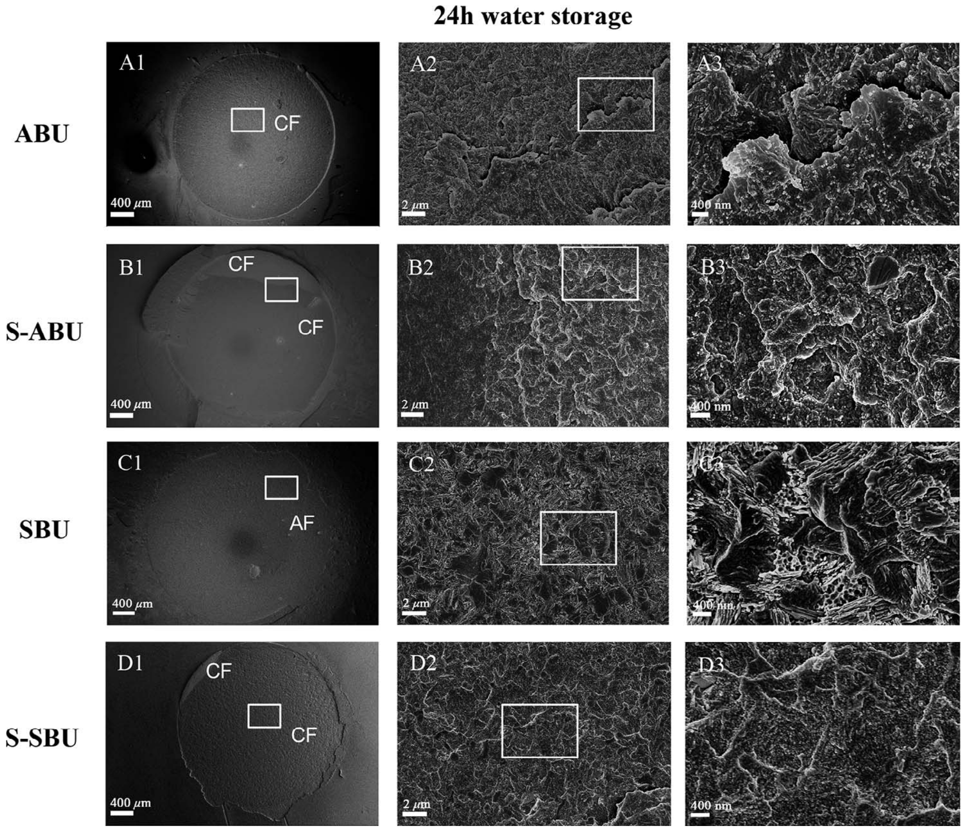

Figure 2. *Representative FESEM images of fracture surface after 24 hours of water storage. (A1): Overview (24×) in All Bond Universal (ABU in non–silane-pretreated mode). (A2-A3): High magnification (5000× and 20,000×) of the cohesive failure part. (B1): Overview in S-ABU (All Bond Universal in silane-pretreated mode). (B2-B3): High magnification of the cohesive failure part. (C1): Overview in SBU. (C2-C3): High magnification of the failure part. (D1): Overview in S-SBU. (D2-D3): High magnification of the cohesive failure part. AF, adhesive failure; CF, cohesive failure.*

after aging compared with 24-hour immediate bonding ($p<0.05$), regardless of whether silane pretreatment was implemented. For each adhesive, the subgroup in the non–silane-pretreated mode after aging (eg, ABU-10,000 thermal cycles) had the highest microleakage percentage.

Figure 4B shows the representative LM images of the ABU and S-ABU groups after 10,000 thermal cycles. Compared with the nonpretreatment group, the pretreatment group exhibited lower degrees of basic fuchsin along the ceramic–resin interface, and the ratio between the stained layer and the bond interface was less than 50%.

CLSM Images

Strong red fluorescence signals were observed in the interface between the composite resin and the lithium disilicate glass ceramic, indicating the presence of adhesive. Figure 4C shows representative CLSM images after 24 hours of soaking and 5000 or 10,000 runs of thermocycling. For the CUB group, the middle area of the adhesive layer significantly thinned after 10,000 cycles of aging compared with that after 24 hours of water storage, possibly implying a weak zone for interfacial fracture. For the S-CUB group, the adhesive layer showed only a few small cracks, cleavages, and voids after aging.

DISCUSSION

In this study, the SBS of all universal adhesives in the silane-pretreated mode was higher than that in the non–silane-pretreated mode regardless of the occurrence of immediate or durable bonding. Furthermore, the durable marginal sealing performance of these universal adhesives in the silane-pretreated mode was better than that in the nonpretreated mode. Thus, the first null hypothesis was rejected. These results suggested that additional silane pretreatment was necessary to improve the durable bonding effectiveness of universal adhesives to lithium disilicate glass ceramics. The SBS of these adhesives after aging was lower and their marginal sealing capability was less stable than those in the immediate condition regardless of the non–silane-pretreated or silane-pretreated mode, except that the SBS of S-ABU after aging was similar to that after 24 hours of water storage. Accordingly, the second null hypothesis was also rejected.

In the oral environment, chemical, thermal, and mechanical factors including saliva, temperature change, biting force, and other habits[7,28] may

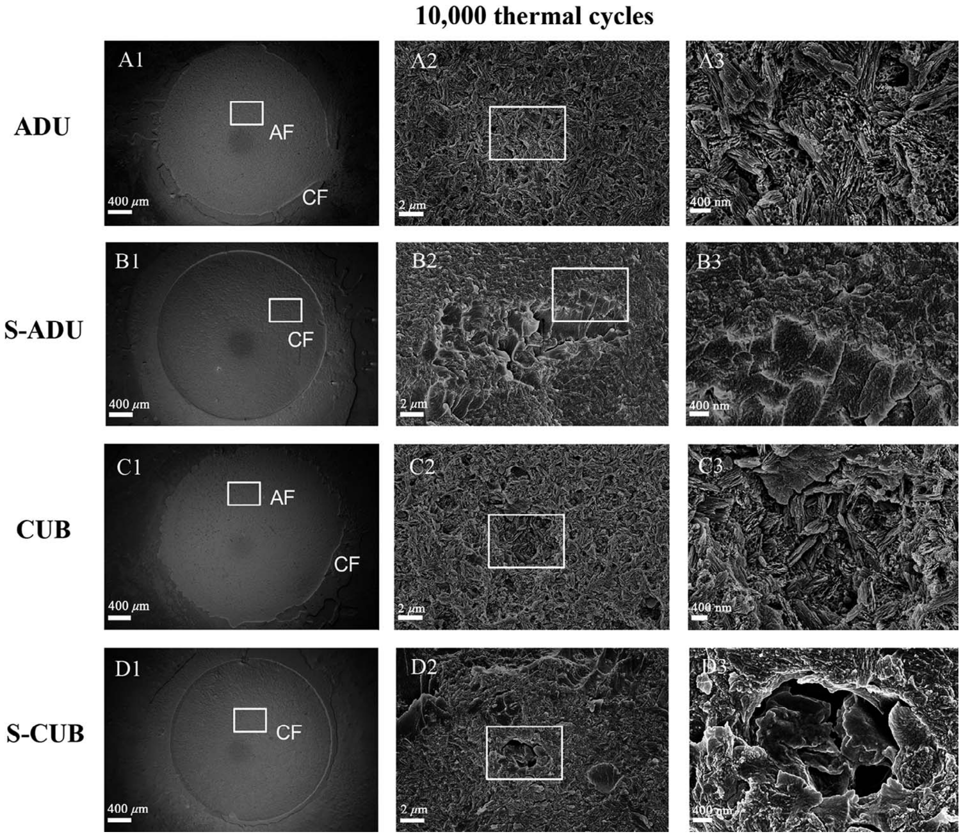

Figure 3. *Representative FESEM images of fracture surface after aging. (A1-A3): ADU group. (B1-B3): S-ADU group. (C1-C3): CUB group. (D1-D3): S-CUB group. Adhesive failure dominated in the ADU and CUB groups. Cohesive failure dominated in the S-ADU and S-CUB groups. AF, adhesive failure; CF, cohesive failure.*

influence ceramic–resin bonding stability.[29] We adopted 10,000 thermal cycles in this study to imitate 1-year clinical physiological aging[16] and to evaluate bond degradation. Our results showed that the SBS of the universal adhesives in the silane-pretreated or non–silane-pretreated mode declined after aging, indicating that thermal cycling aging negatively affected the bonding performance of universal adhesives on lithium disilicate glass ceramic. This deterioration can be explained as follows: (1) thermal cycling could produce hoop stress[30] as a result of the different thermal expansion rates of substrates in bonded interfaces[17] and consequently induce volumetric changes in adhesive layers; thus, the local ingress of water is further enhanced, thereby decreasing the physical/mechanical properties by weakening the frictional forces between polymer chains,[31] (2) the effects of aging may be exacerbated by the chemical hydrolysis of the interfacial components,[32,33] including hydrolytically susceptible groups within methacrylate adhesives, such as hydroxyl, carboxyl, ester, and urethane,[34] thus, adversely affecting durable bonding performance because of these vulnerable areas in the vicinity of the ceramic–resin interface.

However, the extent of SBS decline for adhesives in the non–silane-pretreated mode was significantly higher than that in the silane-pretreated mode, indicating that additional silane may be an efficient material to improve the long-term durability of adhesive interface bonding. This finding corroborated the results obtained by Kim and others,[13] who studied the bonding performance of universal adhesives on leucite-reinforced ceramics. A previous work also reported that optimal bonds are achieved by applying silane to etched lithium disilicate glass ceramics before Scotchbond Universal is used.[35]

Notably, the higher SBS in the silane-pretreated mode than in the non–silane-pretreated mode was possibly related to the predominant cohesive failure. The present study demonstrated that cohesive failure was dominate for all of the universal adhesives in the silane-pretreated mode after 10,000 cycles of thermal aging. Cohesive failure was recognized to increase bond strength values because a fracture propagates through the bulk of a bonded material.[29] Consistent with our findings, previous research also indicated that the bond between glass ceramic and composite resin is stronger if there is cohesive failure within composite

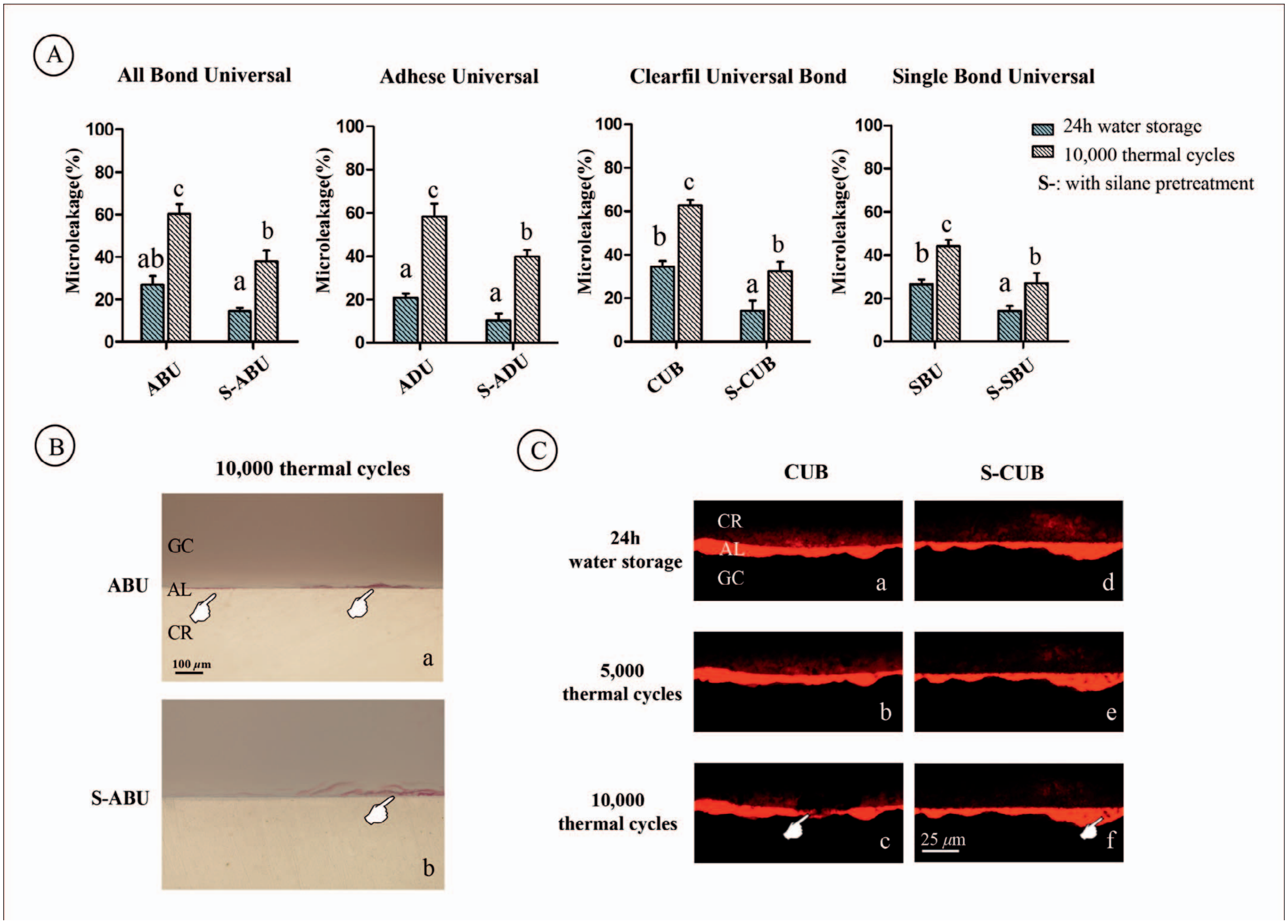

Figure 4. *Effectiveness of bonding strategy and aging on the marginal sealing ability of lithium disilicate glass ceramic with four universal adhesives. (A): Microleakage percentage was analyzed separately for each adhesive using two-factor ANOVA with the main effects. For each adhesive, columns labeled with the same lowercase letters are not significantly different in pairwise comparisons ($p > 0.05$). (B): Representative LM images after aging. a, ABU group. b, S-ABU group. The sign indicates the area with existing microleakage. (C): Representative CLSM images after aging. The sign indicates cleavages and voids existing in the adhesive layer. AL, adhesive layer; CR, composite resin; GC, lithium disilicate glass ceramic.*

in debonded specimens, and this strong bond is attained by applying silane.[36]

Ensuring stable adhesion and tight sealing is key to evaluating the durability of adhesive systems. The mean SBS of the mechanical analysis cannot be considered the sole indicator of bond quality. Thus, an interfacial morphological evaluation may provide additional information. In this work, basic fuchsin stains were observed in the adhesive layer by using light microscopy to reveal the microleakage areas at the interface. The microleakage percentages of all of the adhesives were significantly higher after aging than those with immediate bonding regardless of the silane application mode. However, the percentages of the groups in the silane-pretreated mode (eg, S-ABU) were significantly lower than those in the non–silane-pretreated ones (eg, ABU) after aging. Similarly, the microleakage results validated that the additional silane enhanced the durability and stability of the adhesive interface.

Although aging inevitably occurred, based on the above SBS and microleakage results after aging in the silane-pretreated mode, our findings provided direct evidence that the additional silane was conducive for maintaining long-term ceramic–resin stability. Two reasons may contribute to this effect. On one hand, although the adhesive interface behaves as a permeable membrane,[37] the silanols of silane coupling agents form a direct siloxane bridge with the hydroxyls of the ceramic surface after silane pretreatment is applied. Thus, a cross-linked siloxane polymolecular layer is produced, thereby forming an interpenetrating polymer network with the composite resin.[38] In brief, a hydrophobic and branched three-dimensional siloxane film is produced through silanization.[39] Therefore, using a separate silane before applying an adhesive may increase the hydrophobicity of a layer and likely inhibit water uptake. Previous studies also found that degradation occurs less frequently when a more hydrophobic adhesive coating is used.[40,41] Consequently, silane pretreatment could help decrease the possibility of hydrolytic degradation of ceramic–resin bonding and increase the durable bonding quality of the adhesive interface.

On the other hand, additional silane pretreatment may increase the thickness and uniformity of the whole adhesive bonding layer because silane can form an additional layer of chemical bonds between adhesive resin and glass ceramic. In particular, methacrylate groups within adhesive resin can copolymerize with silane molecules,[36] and silanol groups produced by the corresponding methoxy groups can react with the glass ceramic surface.[25] Thus, CLSM images were observed to validate the morphological changes between the non–silane-pretreated and silane-pretreated modes after aging. In a previous study,[42] CLSM revealed that fluid moved at the junction of the adhesive resin and a hybrid layer during the flexure of a tooth and a restoration. In our study, CLSM images demonstrated that the rhodamine dye diffused through the adhesive interface. The gaps among composite resins, adhesives, and glass ceramics may be responsible for the unstable bonding. After the S-CUB was subjected to 10,000 cycles of thermal aging, the adhesive layer showed some small cracks, cleavages, and voids. By comparison, greater dimensional alterations were observed in CUB. The alterations at the interfaces were considered flaws that might expand and contract. Water would infiltrate into the flaw zones, thereby impairing the mechanical properties of the adhesive layer and accelerating deterioration. This phenomenon suggested that the relative integrity of the ceramic–resin adhesive layer created by applying additional silane was essential to bond durability.

Another notable observation was that the silane-containing universal adhesives (CUB and SBU) did not perform better than the silane-free ones (ABU and ADU) as we had expected. After aging occurred, the SBS of the silane-containing universal adhesives in the non–silane-pretreated mode significantly decreased and their microleakage percentage increased. This result shed light on the fact that the silane incorporated in universal adhesives was incapable of slowing down the aging of the ceramic–resin bonding interface, possibly because of the finite quantities and proportions of silane in universal adhesives, along with numerous other components, such as bisphenol A diglycidylmethacrylate, camphorquinone, 10-methacryloyloxydecyl dihydrogen phosphate, and solvent.[34] The efficiency and chemical stability of silane existing in universal adhesives should be further examined.

CONCLUSIONS

Four universal adhesives (two silane-containing and two silane-free) in the silane-pretreated mode exhibited an improvement in bond strengths and a reduction in microleakage percentages compared with those in the non–silane-pretreated mode after 10,000 cycles of thermal aging. Despite the enhanced ease of use of universal adhesive, the simplified bonding approach decreased the bonding quality after aging. Therefore, treatment with additional silane prior to the application of the universal adhesives (for either silane-containing or silane-free) should be clinically promoted to improve the effectiveness and longevity of ceramic–resin bonding.

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Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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