Original Article

Effect of different adhesive systems and silane application on shear bond strength of cement to indirect restorations

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Abstract

Objective: This study evaluated and compared the influence of various adhesive systems and silane application on the adhesion of resin-based luting cement to lithium disilicate, indirect composite resin, and zirconia restorations.

Methods: Lithium disilicate (n=50), indirect composite resin (n=50), and zirconia (n=50) blocks were divided into five groups (n=10), according to the adhesive protocol applied as follows: 1. Optibond XTR, 2. silane + Optibond All in One, 3. One Coat 7 Universal, 4) Adper Scotchbond Multi-Purpose, and 5) Silane + Single Bond Universal. The blocks were bonded to a resin-based luting cement after surface treatments (sandblasting and acid application). The bonded specimens were incubated in 37° C water for 24 hours and thermocycled for 5,000 cycles. The shear bond strength (SBS) was evaluated by a universal testing machine. The adhesion protocols for each type of restoration were compared by one-way ANOVA and Dunnett T3 test.

Results: There were significant differences in the bond strength of cement to the indirect restoration between various adhesive protocols ($P<0.05$). In all types of indirect restorations, the highest SBS values were observed in group 5, which was silanized and bonded with Single Bond Universal. The bond strength of group 5 in lithium disilicate, indirect composite resin, and zirconia groups were 26.1 ± 4.9 MPa, 20.5 ± 5.7 MPa, and 15.4 ± 4.7 MPa, respectively.

Conclusions: It appears that the best adhesive protocol for bonding cement to lithium disilicate, indirect composite resin, and zirconia restorations is the use of silane and a universal adhesive containing silane. (J Dent Mater Tech 2023;12(2): 104-110)

Keywords: Bond strength, Composite resin, Lithium disilicate, Silane, Zirconia

Introduction

The composition of restorative materials greatly influences the clinical effectiveness of indirect restorations (1). The chemical composition of the adhesive system and the pretreatment of the internal surface of the restoration influence the wetting capacity and bond durability (2, 3). Consequently, manufacturers have introduced a variety of primers or adhesives to enhance the bond between resin cement and indirect restorative materials (1, 4).

As a coupling agent, silane is a bifunctional molecule capable of bonding with silicon dioxide (SiO_2) and the organic matrix of resin composite cement (5, 6). Silane is applied for surface pretreatment to facilitate the bonding of resin-based luting cement to substrate surfaces (7). Universal adhesives have become increasingly popular among clinicians due to their fewer and simpler application steps, with a shorter application time (8). They can be utilized in the etch-and-rinse or self-etching modes (8). Some universal adhesives contain silane in their formulation, which may be effective in enhancing bond strength (9). However, there is limited evidence about the effect of Universal bonding systems on bond strength to indirect restorations. Furthermore, it is not well clear whether a separate silane application can improve the adhesion of bonding systems to glass ceramics, resin composites, and zirconia.

Self-adhesive cement containing 10-methacryloyloxydecyl dihydrogen phosphate (MDP) has been developed for zirconia restorations to enhance bonding. It has been demonstrated that shear bond strength (SBS) and compressive strength to zirconia are improved with MDP-containing self-adhesive resin cement (10).

The present study aimed to investigate the effects of various adhesive systems and silane application on the

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bond strength of resin-based luting cement to lithium disilicate, zirconia, and indirect composite resin restorations. The surface topography of the specimens was also assessed after SBS testing.

Materials and Methods

Sample preparation

The materials used in this study are listed in Table 1. Three groups of materials, including zirconia, lithium disilicate, and composite resin, were prepared as follows. Planar-shaped zirconia specimens (n=50), each with a surface area of 21 mm × 21 mm and a thickness of 10 mm, were procured from sintered disc-shaped zirconia blocks (AvaDent, Italy). Composite blocks (n=50), matching the size of zirconia and lithium disilicate materials, were constructed from an indirect composite resin system (Tescera ATL II, Bisco Inc, Illinois, USA). A silicone impression mold (Coltene®, Whaledent, Altstätten, Switzerland) was utilized for each composite block, intending to replicate the zirconia blocks. Following the manufacturer's instructions, the specimens underwent light-curing paired with heat-curing via the reinforced microfill composite lightbox and heat box. After final polymerization, the surface of the composite resin blocks was polished using 50 μm aluminum oxide discs (Super-Snap Rainbow Kit, Shofu Corp., Tokyo, Japan), then rinsed with water.

Table 1. The product name, composition, and manufacturer of the materials used in this study

Product (Lot No)	Composition	Manufacturer
Adper Scotchbond Multipurpose (N794505)	Bisphenol A glycol dimethacrylate (Bis-GMA), 2-hydroxyethyl methacrylate (HEMA)	3M ESPE, St Paul, MN, USA
Single Bond Universal (613052)	Bisphenol A glycol dimethacrylate (Bis-GMA), 10-methacryloxydecyl dihydrogen Phosphate (MDP), Dimethacrylate resins, 2-hydroxyethyl methacrylate (HEMA), Vitrebond copolymer, Silane, Ethanol, Water	3M Deutschland GmbH, Neuss, Germany
Silane Primer (5442600)	80-85% Ethyl alcohol	Kerr, Orange, CA, USA
Optibond XTR Adhesive	Bisphenol A glycol dimethacrylate (Bis-GMA), 2-hydroxyethyl Methacrylate (HEMA), Tri-functional monomer, Ethanol, Photoinitiator, Barium glass filler, nano-filler. Fluoride-containing filler	Kerr, Orange, CA, USA
Optibond All in One (5918660)	Hexafluoroglutaric anhydrideglycerodimethacrylate, Glycerol phosphate dimethacrylate, Water, Butylhydroxytoluene, Ethanol, 2 Ethylhexyl – 4 dimethylamino dimethacrylate, Silica dioxide (SiO_2), Barium aluminoborosilicate, sodium hexafluorosilicate.	Kerr, Orange, CA, USA
One Coat 7 Universal	2-hydroxyethyl Methacrylate, Methacrylate modified polyacrylic acid, Urethanedimeth acrylate, Glycerol dimethacrylate, Amorph silicic acid, Water (5%), initiators and stabilizers	Coltene Whaledent, Switzerland
Panavia SA Plus Automix (5L0096)	Silanated colloidal silica filler, Silanated barium, Glass filler, Peroxide, dl-Camphor-quinone, Hydrophobic aliphatic dimetacylate/aromatic, Dimetacrylate, Catalysis	Kuraray Europe GmbH Germany
Tescera ATL	Reinforced microfill composite (Body) Urethane dimethacrylate (UDMA) (< 15%), glass filler (< 80%), amorphous silica (< 25%)	Bisco Dental Product Asia Ltd., Seoul, Korea
Hydrofluoric Acid 9.5% (1400000180)		Bisco Inc, Schaumburg, IL,USA

Lithium disilicate blocks (e.max CAD, Ivoclar Vivadent, Amherst, NY, USA) in their bisque form were sectioned into rectangular shapes by a low-speed cutting device (Isomet, Buehler Ltd, Lake Bluff, IL, USA). Afterward, they were sintered as per the manufacturer's instructions. Each ceramic was embedded in an autopolymerizing acrylic resin block (Paladur; Heraeus Kulzer, Armonk, NY) in a silicone impression mold to mimic the size of zirconia blocks. The specimens were then ultrasonically cleaned in distilled water for 10 min, then dried using compressed air.

A tribochemical silica coating was applied to all indirect restoration specimens using an intraoral sandblaster (Miniblaster, Deldent Ltd, Israel) with 30 µm particles (Cojet, 3M ESPE, St. Paul, MN, USA) from a distance of 10 mm for 15 seconds at 0.3 MPa air pressure. After that, ceramic specimens received an application of 9.5% hydrofluoric acid (Porcelain Etchant, Bisco, Schaumburg, IL, USA). A droplet of etchant was distributed evenly across the bonding surface of the ceramic for 60 seconds using a micro brush. The surface was cleaned with water for 20 seconds and dried with compressed air for 15 seconds.

After undergoing surface treatments, the zirconia, lithium disilicate ceramic, and composite resin specimens were categorized into five subgroups (n=10) based on the adhesion protocol used for surface treatments:

1) Optibond XTR
2) Silane + Optibond All in One
3) One Coat 7 Universal
4) Adper Scotchbond Multi-Purpose
5) Silane + Single Bond Universal

Plexiglass tubes with an inner diameter of 3.2 mm and a height of 3 mm were subsequently affixed to the specimens and filled with resin cement (Panavia SA Plus Automix, Kuraray Europe GmbH, Germany). The resin-based luting cement was then light-cured from two opposing sides for 40 seconds (BlueLEX LD-105, Monitex Ind. Co., Taipei, Taiwan). The specimens were subsequently incubated in distilled water at 37°C for 24 hours, then thermocycled for 5,000 cycles between 5°C and 55°C water baths, with each cycle lasting 15 seconds.

Shear bond strength (SBS) test

The plastic tube was removed for the shear bond test, and the specimens were fixed onto a steel fixture in a universal testing machine (Instron 5565, Canton, MA, USA). A sharpened stylus applied a shear load to the side of the cement cylinder until failure, moving at a crosshead speed of 1 mm/min. The shear bond strength (SBS) values were calculated in MPa by dividing the failure load (N) by the bonding area (mm2).

Failure mode analysis

In the failure mode analysis, two specimens from each group were randomly selected, sputter-coated with gold, and then observed under a scanning electron microscope (SEM; Zeiss Evo LS 10, Germany) at magnifications ranging from 1000 to 5000 to visualize the type of failure following various treatments. The failure mode was classified as one of three types: adhesive failure at the ceramic/cement interface, cohesive failure, or a combination of both.

Statistical Analysis

The data were analyzed using SPSS version 22 (IBM; Armonk, NY, USA). The data were normally distributed according to the Kolmogorov-Smirnov test (P>0.05). The adhesion protocols for each restorative material were compared by one-way analysis of variance (ANOVA), followed by the post hoc Dunnett T3 test for multiple comparisons. A significance level of 0.05 was established.

Results

Table 2 presents the SBS values of resin cement to the zirconia samples prepared by different adhesive protocols. The highest SBS was observed after using silane + Single Bond Universal (group 5). ANOVA showed a significant difference in SBS between the different groups in the zirconia substrate (P=0.03; Table 2). Further analysis with post hoc Dunnett T3 test revealed that bonding with Optibond XTR (12.18±2.90 MPa), Adper Scotchbond Multi-Purpose (12.27±7.08 MPa), and silane + Single Bond Universal (15.38±4.67 MPa) provided comparable bond strength (P>0.05; Table 2), which were significantly higher than that of the other zirconia groups (P<0.05; Table 2).

The SBS values of resin cement to the lithium disilicate substrate after different adhesive protocols are presented in Table 3. ANOVA showed a significant difference in SBS values between the groups in the lithium disilicate samples (P=0.001; Table 3). According to pairwise comparisons, SBS was significantly greater in samples treated by silane + Single Bond Universal (26.08±4.86 MPa), as compared to the other groups (P<0.05; Table 3). ANOVA displayed a significant difference in SBS of resin cement to indirect composite resin substrate among

Table 2. Comparison of shear bond strength (MPa) values of resin cement to zirconia samples prepared by different adhesive protocols

Adhesive	Mean± SD	Minimum	Maximum
Optibond XTR	12.18 ± 2.90[c]	8.49	17.45
Silane + Optibond All in One	6.39 ± 2.15[a]	2.52	10.45
One Coat 7 Universal	8.67 ± 2.97[b]	5.18	15.98
Adper Scotchbond Multi-Purpose	12.27 ± 7.08[c]	7.04	31.85
Silane + Single Bond Universal	15.38 ± 4.67[c]	7.96	22.51
P-value	0.03		

*Different lowercase letters represent a significant difference between the groups at P<0.05., SD: Standard deviation

Table 3. Comparison of shear bond strength (MPa) values of resin cement to lithium disilicate samples prepared by different adhesive protocols

Adhesive	Mean± SD	Minimum	Maximum
Optibond XTR	10.69 ± 3.93[a]	5.65	16.77
Silane + Optibond All in One	12.90 ± 3.41[a]	8.77	17.87
One Coat 7 Universal	10.51 ± 3.42[a]	4.85	15.18
Adper Scotchbond Multi-Purpose	11.00 ± 3.76[a]	4.91	16.42
Silane + Single Bond Universal	26.08 ± 4.86[b]	19.61	35.33
P-value	0.001		

*Different lowercase letters represent a significant difference between the groups at P<0.05., SD: Standard deviation

Table 4. Comparison of shear bond strength (MPa) values of resin cement to indirect composite resin samples prepared by different adhesive protocols

Adhesive	Mean± SD	Minimum	Maximum
Optibond XTR	15.84±4.17[a]	10.98	24.57
Silane + Optibond All in One	16.90±4.17[a]	10.31	23.35
One Coat 7 Universal	14.03±1.94[a]	11.58	18.64
Adper Scotchbond Multi-Purpose	13.41±6.60[a]	5.84	24.47
Silane + Single Bond Universal	20.55±5.70[b]	9.91	31.00
P-value	0.02		

*Different lowercase letters represent a significant difference between the groups at P<0.05., SD: Standard deviation

different adhesive protocols (P=0.02; Table 4). The result of multiple comparisons by Dunnett T3 revealed that the application of silane with Single Bond Universal (group 5) led to a significantly higher bond strength (20.55±5.70 MPa), as compared to the other adhesive groups (P<0.05; Table 4).

The failure mode analysis revealed a cohesive type of failure in groups with higher bond strength (Figures 1 and 2), whereas, in samples with lower bond strength, adhesive failure was more frequently observed.

Discussion

The present study scrutinized the influence of various adhesive protocols and silane application on the bond strength of resin cement to zirconia, lithium disilicate, and indirect resin composite substrates. An array of adhesive systems and methodologies have been devised to amplify bond strengths and simplify the bonding procedure (11). Enhancing the bond strength of indirect ceramic restorations involves improving mechanical interlocking by sandblasting or chemical enhancement methods such as silane application and using different adhesive monomers (12)

Establishing a desirable mechanical bonding via sandblasting can be challenging due to variables like air blasting pressure, application time, particle size, the sectional form of blasted particles, and the material's surface properties (7, 13, 14). Therefore, incorporating an additional silanization step can strengthen chemical bonding to the exposed hydroxyl groups and enhance surface wettability, thereby increasing bond strength (15). Furthermore, silane impregnation in universal adhesive simplifies the protocols and reduces bonding step failures (16).

In the present study, the application of additional silane alongside the silane-containing universal adhesive system (group 5) substantially increased the bond strength of resin cement to all types of indirect restorations. In lithium disilicate and indirect composite

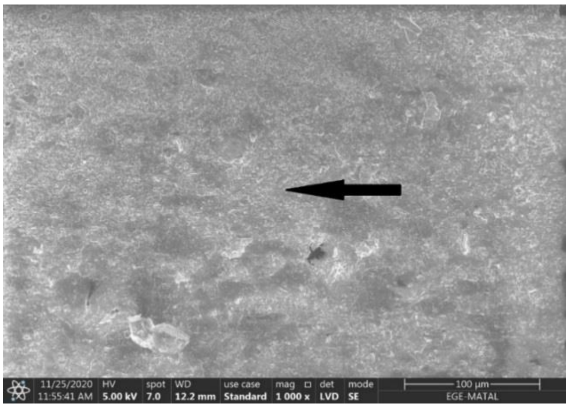

Figure 1. Lithium disilicate group with universal adhesive. Resin cement is widely noted as indicated with an arrow.

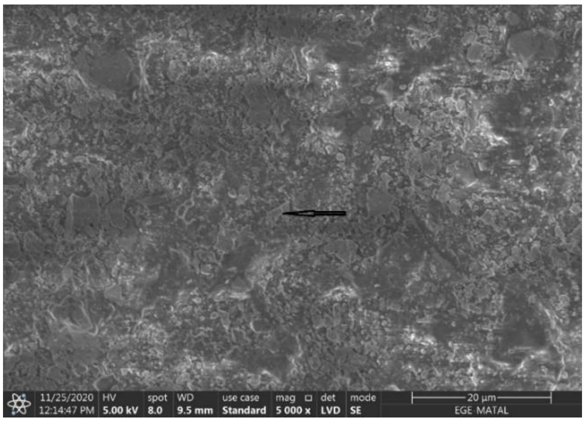

Figure 3. Zirconium group with the universal adhesive

resin samples, bond strength was significantly greater in group 5, as compared to the other groups (26.08±4.86 MPa for lithium disilicate, and 20.55±5.70 MPa for indirect composite resin).In zirconia substrate, the use of silane + Single Bond Universal (15.38±4.67 MPa) provided the highest bond strength, but the difference with Optibond XTR (12.18±2.90 MPa), and Adper Scotchbond Multi-Purpose (12.27±7.08 MPa) was not statistically significant. This outcome can be rationalized by the instability of silane-zirconia conjunctions in acidic pH (2.3–2.7) of most MDP-based universal adhesives. A study by Koko et al. (18) revealed that silane concentrations exceeding a predetermined threshold significantly weakened the bonding performance of MDP on zirconia ceramics. This degradation was attributed to the dilution of MDP content by silane (19).

We preferred to use a separate silane application step before the use of silane-containing Single Bond Universal. Fourier-transform infrared spectroscopy (FTIR) and nuclear magnetic resonance (NMR) have validated the dehydration self-condensation reaction of silanol groups in silanes present in universal adhesives (17). Our findings align with those of Zaghloul et al. (20) and Kalavacharla et al. (21), who reported that both silane-containing universal adhesive and additional silane application substantially improved the SBS values in lithium disilicate and composite resins. Almaskin et al. (22) also noted that isolated silane application significantly strengthened the bond of lithium silicate to the cement compared to silane-containing universal adhesive. This might be attributed to the fact that the adhesive bottle contains stored prehydrolyzed silane with a short shelf-life and reduced efficacy. The silane in the adhesives might be overly simplistic or insufficient to generate an effective siloxane network and yield the same effect as the silane primer. Therefore, additional silane pretreatment can improve the lithium disilicate ceramic bonding with saline-containing adhesives. According to Yao et al. (23), the bond strength of silane-containing universal adhesive did not differ from silane-free adhesives if no separate silane is employed.

There is limited evidence regarding the SBS of indirect resin composite with silane-containing universal adhesive, thus comparing the results of this study with other studies is challenging. In vitro, studies have cited a bond strength of 15-25 MPa for indirect composite resin as the optimal value (24, 25). In our study, most indirect composite resin specimens bonded with adhesive systems with or without silane application exhibited a mean bond strength within the optimal range, which is consistent with previous studies (24, 25). However, some reports argue that extra silane application is less effective than anticipated, as silica permeation is insufficient due to the high density of the substrate (26). Jusue-Esparza et al. (27) reported that silane coupling agents did not influence the adhesion process of the aged resin. Silane interaction with the composite resin's surface depends on silicon availability (28). In our study, air-particle abrasion with SiO_2 (CoJet) was used for all groups to expose silicon by removing the material's surface organic matrix. Despite the lack of consensus in the literature regarding additional silane application to the silane-containing universal adhesive, our study demonstrated that silanization may play a pivotal role in adhesion to restorative materials.

Zirconia ceramic is a unique material resistant to acids and does not contain silica. Silica coating is a strategy to strengthen the bond between the resin and different surfaces, especially zirconia. It benefits from air abrasion to create a tribochemical effect, depositing a molecular layer of SiO_2 on the surface. The surface is then treated with a silane coating to increase its chemical reactivity towards the resin (29, 30, 31). Özdemir et al. (32) showed that the CoJet system amplified the bond of zirconia specimens to resin cement. Moreover, in their study, silane and bonding agent coupled with MDP-based resin cement presented the highest bond strength in zirconia specimens.

Different failure modes were observed in this research. Cohesive failure was more predominant in groups with higher bond strength such as the silane + universal adhesive group in all types of substrates (zirconia, lithium disilicate, and indirect composite resin). However, the groups with lower bond strength primarily exhibited adhesive failures

Future in vivo research is necessary to assess the success and survival rates of different adhesive systems and surface conditioning processes in indirect restorative materials.

Conclusions

The bond strength of cement to zirconia, lithium disilicate, and indirect composite resin restorations is influenced by the adhesive system applied. It appears that the best adhesive protocol for bonding cement to lithium disilicate, indirect composite resin, and zirconia restorations is the use of silane and a universal adhesive containing silane (Single Bond Universal).

Conflict of Interest

No conflict of interest was declared by the authors

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