Intra-oral adhesive systems for ceramic repairs: a comparison

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The aim of this investigation was to compare the bond strength of restorative composite resin to dental ceramic conditioned with primers and adhesives of various commercial repair kits. Three intra-oral ceramic repair systems—Silistor (Heraeus Kulzer), Cimara (Voco), Ceramic Repair (Vivadent)—were used on all-ceramic (IPS Empress 2, Ivoclar-Vivadent) substrate. Shear bond strength of restorative composite resin to substrate was tested after thermocycling and without thermocycling ($n = 10$). Substrate surfaces of the specimen after loading were examined microscopically (SEM). The highest bond strengths in both water-stored (7.0 ± 5.7 MPa) and thermocycled conditions (2.5 ± 1.8 MPa) were obtained with the Vivadent repair system, while the lowest values were observed with the Cimara system (0.6 ± 1.4 MPa and 0.0 ± 0.0 MPa, respectively). Shear bond strengths appeared to be significantly affected by thermocycling (ANOVA, $P < 0.05$). It is concluded that there are significant differences in the bond strengths of resin composites and ceramic substrate. The roughened surface does not necessarily provide a better bond strength; the bond strength of composite decreases with storage in water and after thermocycling. Bond strength values were generally low for all of the tested materials. □ *All-ceramics; intra-oral repair; shear bond strength*

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Increased esthetic demands in dentistry have led to greater use of all-ceramic restorations. Relatively good long-term success rates have been documented in studies of porcelain laminate veneers, ceramic inlays and onlays, resin-bonded fixed partial dentures and all-ceramic crowns (1–10). Although the strength of ceramics has improved with the introduction of reinforced ceramic core materials of different compositions, ceramic restorations can still fracture due to their brittle nature. Intra-oral repair options for ceramics may provide a practical approach for repairing minor defects by composite resin rather than removal of whole restorations.

While the literature is replete with reports on intra-oral repair of porcelain-fused-to-metal (PFM) restorations (11–14), there are only a few studies about all-ceramic repairs (15). The difference in repair of PFM restoration compared to all-ceramic restoration lies in the exposed core or framework material. It has recently been suggested that the composite resin adheres well to the exposed metal alloy with the use of silica coating systems (13, 14). Silica coating may also provide improved adhesion of composite resin to all-ceramics such as lithium disilicates (16). On the other hand, a novel intra-oral repair system could provide an alternative to the silica-coating system. The aim of this study was therefore to evaluate the influence of three commercial repair kits for all-ceramics on the adhesion of restorative composite resin to lithium disilicate all-ceramic substrate.

Material and methods

The materials used in this investigation (Table 1) were prepared and handled in accordance with the instructions of the manufacturers. Sixty lithium-disilicate reinforced glass-infiltrated ceramic disks (10 mm diameter and 2 mm thick) were fabricated and embedded in acrylic resin blocks, care being taken to ensure that one surface of the disk remained uncovered for bonding procedures. All specimens were wet-ground with 500 grit silicone carbide abrasive, followed by 1200 grit silicone carbide abrasive (Struers RotoPol 11, Struers A/S, Rodovre, Denmark) and cleaned for 10 min in an ultrasonic bath (Quantrex 90 WT, L&R Manufacturing, Inc., Kearny, NJ, USA) containing ethyl acetate, and then air-dried. Twenty specimens were randomly assigned to one of three groups for ceramic repair.

Group 1. Ceramic Repair Kit (Ivoclar-Vivadent)—Substrate surfaces were etched for 15 s with 37% phosphoric acid etching agent (Total Etch, Ivoclar-Vivadent) for surface roughening. After water rinse and air-drying, Monobond-S silane coupling agent was applied and air-dried after 60 s. A thin layer of adhesive resin Heliobond was applied; excess resin was removed with air and light-cured (Optilux 501, Kerr, Orange, CA; light intensity 1,000 mW/cm^2) for 20 s. Using a composite filling instrument, the hybrid restorative composite (referred to as repair material) Tetric Ceram was applied in layers (max. 2 mm) to the conditioned ceramic substrate using translucent polyethylene molds (inner diameter 3.6 mm and height 5 mm). Each layer of resin was light-cured for 40 s.

Group 2. Cimara Kit (Voco)—Substrate surfaces were conditioned with Cimara grinding burs and cleaned with special brushes enclosed with the kit. A thin layer of Cimara silane coupling agent was applied and left to dry

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Table 1. Materials used in the study (compositions from the information provided by the manufacturer)

Brand	Type	Manufacturer
IPS Empress 2	Ceramic*	Ivoclar-Vivadent, Schaan, Liechtenstein
Ceramic Repair	Intraoral Ceramic Repair Kit†	Ivoclar-Vivadent, Schaan, Liechtenstein
Cimara	Intraoral Ceramic Repair Kit‡	Voco, Cuxhaven, Germany
Silistor	Intraoral Ceramic Repair Kit§	Heraeus Kulzer, Hanau, Germany

* Lithium disilicate infiltrated with glass.
† Contains phosphoric acid (37%) etching gel (Total Etch), silane (Monobond-S: 3-Methacryloxypropyl-trimethoxysilane), adhesive resin (Heliobond: Bisphenol-A-glycidyldimethacrylate [Bis-GMA] and triethylene glycol dimethacrylate), and composite resin (Tetric Ceram: Hybrid composite).
‡ Contains Cimara grinding burs, silane (Cimara silane coupling: methylmethacrylate), adhesive resin (Cimara Opaquer liquid: Bis-GMA and urethanedimethacrylate [UDMA]), and composite resin (Arabesk Top: Hybrid composite).
§ Contains grinding burs (K 1), silane (Silicer: isopropylalcohol, silane), adhesive resin (Silibond: methylmethacrylate), and composite resin (Charisma: Hybrid composite).

for 2 min, when a thin layer of Cimara Opaquer liquid was applied and light-cured (Optilux 501) for 20 s. Using a composite filling instrument, restorative composite Arabesk Top was applied in layers to the specimens using translucent polyethylene molds. Each layer of the resin was light-cured for 40 s.

Group 3. Silistor kit (Heraeus Kulzer)—Specimen surfaces were conditioned with K1 grinding burs and cleaned with brushes enclosed with the kit. A thin layer of Silicer silane coupling agent was applied and left to dry for 2 min, followed by a thin layer of adhesive resin Silibond, which was light-cured for 40 s (Optilux 501). Using a composite filling instrument, restorative composite Charisma was applied in layers on the substrate. Each layer of the resin was light-cured for 80 s.

Roughness of substrate surfaces after being mechanically treated (Groups 2, 3) or etched (Group 1) was measured with a profile meter (Mitutoyo Surftest-301, Mitutoyo Corporation, Tokyo, Japan). Average roughness (Ra) values were reported for three measurements of each group. Roughness of the control surface was measured on 1200 grit ground ceramic.

The specimens in each treatment group were divided into two subgroups. Ten specimens from each subgroup were stored in distilled water at 37 ± 1°C for 24 h before shear bond strength of restorative composite resin to the substrate was measured. The other 10 specimens of each subgroup were stored in distilled water at 37 ± 1°C for 24 h and thermocycled for 6,000 cycles in water baths of 5°C and 55°C (dwell time of 30 s in each bath).

The specimens were mounted in the jig (Bencor Multi-T shear assembly, Danville Engineering Inc., San Ramon, CA) of a universal testing machine (Lloyd LRX, Lloyd Instruments Ltd, Fareham, UK). A schematic drawing of the test set-up is shown in Fig. 1. The cross-head speed of continuous loading was 1 mm/min until fracture or debonding occurred; the load deflection curve was recorded with Nexygen 2.0 software (Lloyd LRX, Lloyd Instruments Ltd, Fareham, UK).

Specimens that gave shear bond strength values closest to the mean value of each group were selected for SEM (JSM 5500, Jeol) examination of the substrate surface after loadings. The substrate surfaces were visually analyzed from the SEM photomicrographs.

Statistical analysis was performed using SPSS System for Windows, Release 10.0.5/1999 (SPSS Inc., Chicago, IL). The means of shear bond strengths of each group were analyzed by multivariate analysis of variance (ANOVA), with shear bond strength as the dependent variable and brand of the repair kit and storage condition as independent variables. *P* values less than 0.05 were considered to be statistically significant in all tests. Multiple comparisons were made using Tukey's and Dunnet's T3 post-hoc tests.

Results

The results of the shear bond strength test and of the statistical analysis are presented in Tables 2 and 3, respectively. The highest shear bond strengths in both the water-stored (7.0 MPa) and the thermocycled

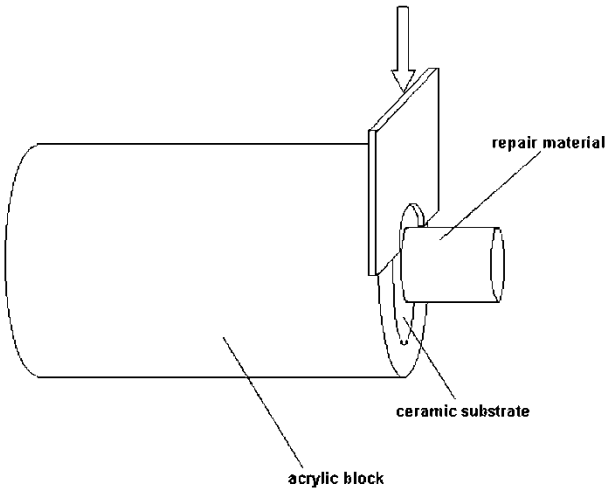

Fig. 1. Schematic drawing of the test set-up to determine the shear bond strengths of restorative composite resin to ceramic substrate. Dimensions in millimeters, with the arrow showing direction of shear force.

Table 2. The means and standard deviations of shear bond strengths (MPa) of repair composite resin to the ceramic substrate with three repair systems

Brand	Storage	Mean	*s*	*n*
Vivadent	Water-stored	7.0	5.7	10
	Thermocycled	2.5	1.8	10
Voco	Water-stored	0.6	1.4	10
	Thermocycled	0.0	0.0	10
Kulzer	Water-stored	3.5	2.6	10
	Thermocycled	0.2	0.9	10

s = standard deviation.

(2.5 MPa) subgroups were obtained with the Vivadent repair system, the lowest values (0.6 MPa and 0.0 Mpa, respectively) with the Cimara repair kit. Shear bond strength was significantly affected by thermocycling ($P < 0.05$) (Table 3). Mean roughness of substrate surfaces after being ground with burs of Silistor was 1.1 μm; Cimara 1.3 μm or etched (Ceramic Repair kit) was 0.2 μm, where mean roughness of the control surface was 0.05 μm (Table 4).

The SEM photomicrographs of the ceramic substrate are shown in Fig. 2. The micrographs showed that the debondings were adhesional, i.e. few if any remnants of restorative composite resin were left on the surface of the substrate.

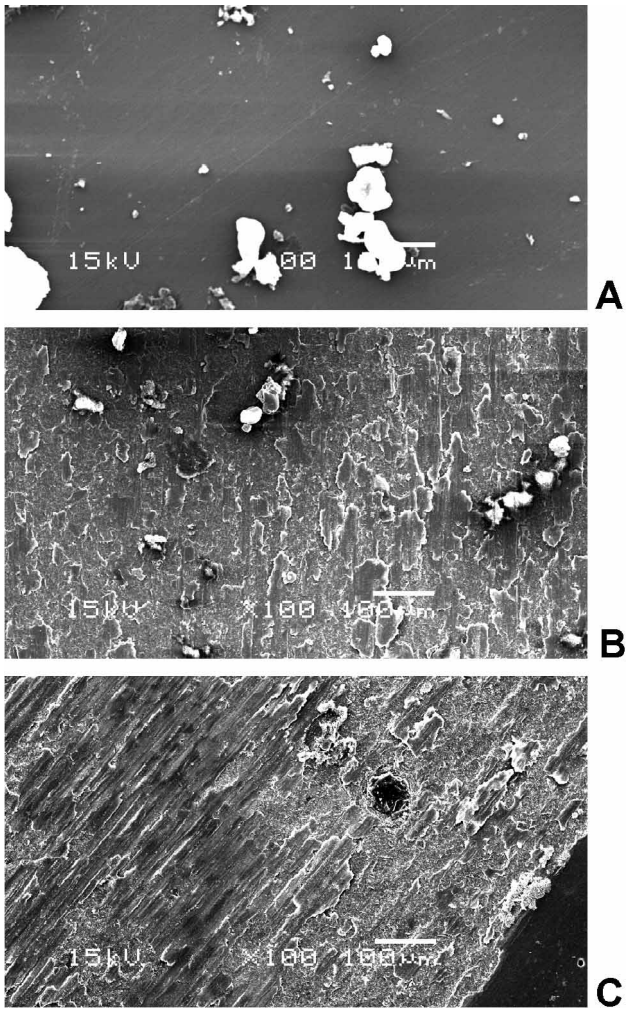

Fig. 2. The SEM photomicrographs of the fracture surface of the ceramic after testing (original magnification ×100). A. Ceramic surface etched by phosphoric acid. B. Ceramic surface treated by Voco special burs. C. Ceramic surface treated by K1 burs.

Discussion

This study has demonstrated that three commercial ceramic repair kits produce considerably different results for the adhesion of hybrid composite resin to lithium disilicate substrate. It is known that the ceramic substrate microstructure has a significant influence on fracture resistance of the composite-ceramic adhesion zone (17). Shear bond strengths to phosphoric acid-etched substrate surfaces appeared to be higher than those obtained for surfaces conditioned mechanically with special burs. This finding is interesting because it has been shown, and verified by the SEM photomicrographs and surface roughness measurements of the present study, that phosphoric acid-etching does not provide a micro-retentive porous surface in the way hydrofluoric acid does (15, 17, 18). However, bond strength values in the range 4–7 MPa are low compared to those obtained by hydrofluoric acid-etching or by an air-abrading particle abrasion system (11 to 22 MPa) (16, 19–23).

Application of a silane coupling agent to the ceramic substrate surface should provide a chemical covalent and

Table 3. Statistical analysis of the results of the shear bond strength test by two-way analysis of variance (ANOVA)

Source	F value	*P* value
Corrected model	9.277	<0.001
Intercept	42.637	<0.001
Brand	13.203	<0.001
Storage	15.074	<0.001
Brand*storage	2.454	0.095

Table 4. Mean surface roughness of ceramic substrates before application of silane coupling agent

Brand	Treatment	Mean Ra (μm)
Control	1200 grit ground	0.05
Vivadent	37% phosphoric acid etched	0.2
Voco	K1 bur	1.3
Kulzer	Bur	1.1

hydrogen bond between the ceramic and the composite resin. A few other mechanisms of function of silane coupling agent, such as surface wettability theory, have also been suggested (24). Some silane coupling agents that contain carboxylic acid have been reported to provide clinically adequate bond strengths even without hydrofluoric acid-etching, while other silane coupling agents tested have been successful after phosphoric acid-etching (15, 18, 25). The differences in silane coupling agent of the tested repair kits could be related to essential chemical reactions of silanes in forming bonding between substrate and resin system. There is some evidence that silanes with different chemical composition and concentration in the solvent result in different adhesion. This is related to the hydrolysis of the silane and to the polycondensation of the polysiloxane network on the substrate surfaces. Parameters such as acidity of the substrate surface affect the hydrolysis, and temperature of the environment affects polycondensation. Whether or not polycondensation of the silane differed because of the silane coupling agents of the repair kit needs to be investigated in further studies.

Treatment of the substrate surface by special burs (like K1 burs) very likely enriched the particles of silicone carbide on the substrate surface. Even though surface roughness seemed to be greater with the K1 bur treated surface, the bond strength values were lower than those of the phosphoric acid-etched group. This could have been due to possible deteriorating effects of speed and pressure by the rotating bur on the surface characteristics of the ceramic (18).

The lowest bond strength was obtained with the Cimara kit, and could be attributed to the possible effects of Cimara grinding burs, silane, adhesive or composite resin either individually or in combination. The influence of these factors on bond strength to ceramic surface must be investigated in further studies.

The type of composite resin can also affect the bond strength to ceramic. It has been suggested that larger particle size composites or hybrid type composites at the porcelain interface result in higher bond strengths than small-sized particle composites (26, 27). However, it is unlikely that the hybrid particle size of the restorative composites used in this study differed to the extent that it would have caused such variations in bond strength values.

Thermocycling and water storage in vitro is a common method of testing dental materials to establish their suitability for in vivo use. There is evidence that thermocycling decreases the bond strength by weakening the interface, e.g. due to fatigue based on differences in the thermal expansion coefficient of ceramics and resin composites (14). This was also seen in the shear bond strengths of all three repair systems tested in this study.

It is concluded that there are significant differences in the bond strengths of resin repair systems and ceramic surfaces. The roughened surface does not necessarily provide a better bond strength. Generally, all three intraoral repair kits provided only modest if any improvement in bond strength.

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