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## Effect of surface conditioning methods on the bond strength of luting cement to ceramics

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### Abstract

**Objectives.** This study evaluated the effect of three different surface conditioning methods on the bond strength of a Bis-GMA based luting cement to six commercial dental ceramics.

**Methods.** Six disc shaped ceramic specimens (glass ceramics, glass infiltrated alumina, glass infiltrated zirconium dioxide reinforced alumina) were used for each test group yielding a total number of 216 specimens. The specimens in each group were randomly assigned to one of the each following treatment conditions: (1) hydrofluoric acid etching, (2) airborne particle abrasion, (3) tribochemical silica coating. The resin composite luting cement was bonded to the conditioned and silanized ceramics using polyethylene molds. All specimens were tested at dry and thermocycled (6.000, 5–55 °C, 30 s) conditions. The shear bond strength of luting cement to ceramics was measured in a universal testing machine (1 mm/min).

**Results.** In dry conditions, acid etched glass ceramics exhibited significantly higher results (26.4–29.4 MPa) than those of glass infiltrated alumina ceramics (5.3–18.1 MPa) or zirconium dioxide (8.1 MPa) (ANOVA,  $P < 0.001$ ). Silica coating with silanization increased the bond strength significantly for high-alumina ceramics (8.5–21.8 MPa) and glass infiltrated zirconium dioxide ceramic (17.4 MPa) compared to that of airborne particle abrasion (ANOVA,  $P < 0.001$ ). Thermocycling decreased the bond strengths significantly after all of the conditioning methods tested.

**Significance.** Bond strengths of the luting cement tested on the dental ceramics following surface conditioning methods varied in accordance with the ceramic types. Hydrofluoric acid gel was effective mostly on the ceramics having glassy matrix in their structures. Roughening the ceramic surfaces with air particle abrasion provided higher bond strengths for high-alumina ceramics and the values increased more significantly after silica coating/silanization.

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**Keywords:** Surface conditioning; Ceramics; Acid etching; Silica coating; Zirconia ceramics; Alumina ceramics

### 1. Introduction

Numerous attempts have been made to develop ceramic systems that eliminate metal infrastructures and provide optimal distribution of reflected light. Currently clinicians have an increasing range of ceramics capable of delivering high quality aesthetic restorations to choose from for many clinical indications. New ceramic systems involve reinforced ceramic cores through dispersion with leucite [1–6], glass infiltration into sintered alumina (Al<sub>2</sub>O<sub>3</sub>) [7,8], the use of

high-purity alumina [9] or zirconium dioxide (zirconia, ZrO<sub>2</sub>) [10].

To enhance the bond strength of luting cement to the ceramic surface, a number of techniques have been reported which mechanically facilitate resin–ceramic bonding. Etching the inner surface of a restoration with hydrofluoric acid followed by the application of a silane coupling agent is a well-known and recommended method to increase bond strength. Although hydrofluoric acid is efficient in roughening feldspathic ceramic for bonding composite resin [11–16], neither etching with these solutions nor adding silane resulted in an adequate resin bond to some new ceramics [17–19]. Particularly high-alumina [20,21] or zirconia ceramics [22,23] cannot be roughened by

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hydrofluoric acid etching since such ceramics do not contain a silicon dioxide (silica) phase. For this reason, special conditioning systems are indicated for these types of ceramics.

Advances in adhesive dentistry have resulted in the recent introduction of modern surface conditioning methods that require airborne particle abrasion of the surface before bonding in order to achieve high bond strength. One such system is silica coating. In this technique, the surfaces are air abraded with aluminium trioxide particles modified with silica [24–27]. The blasting pressure results in the embedding of these silica coated alumina particles on the ceramic surface, rendering the silica-modified surface chemically more reactive to the resin through silane coupling agents. Silane molecules react with water to form three silanol groups ( $-\text{Si}-\text{OH}$ ) from the corresponding methoxy groups ( $-\text{Si}-\text{O}-\text{CH}_3$ ). The silanol groups then react further to form a siloxane ( $-\text{Si}-\text{O}-\text{Si}-\text{O}-$ ) network with the silica surface. Monomeric ends of the silane molecules react with the methacrylate groups of the adhesive resins by a free radical polymerization process.

When a ceramic exhibits very similar surface compositions and chemical states of silicon and oxygen, then it is reasonable to hypothesize that the siloxane bond will be achieved as these represent the binding sites for the coupling agent to the ceramic surface. Since silane coupling agents do not bond well to alumina, the bond strengths of resin composite to the ceramic will be affected [19]. However, when alumina or zirconia ceramics are glass infiltrated, they are melted together at high temperatures to form a ceramic matrix. The chemical components of the ceramics (traces such as  $\text{Li}_2\text{O}$ ,  $\text{Na}_2\text{O}$ ,  $\text{K}_2\text{O}$ ,  $\text{CaO}$ ,  $\text{MgO}$ ) are then bound to each other by strong covalent bonds with hydroxyl groups at the surface of the ceramic material [28]. When the surface is acid etched and rinsed, this would generate more hydroxyl groups on the surface and also enhance micro-mechanical retention. Furthermore, the methoxy groups of silane would react with water to form silanol groups that in turn will react with the surface hydroxyl groups to form a siloxane network. It was hypothesized in this study that amphoteric alumina in the ceramic matrix could form strong enough chemical adhesion bonds, covalent bridges, through its surface hydroxyl groups with hydrolysed silanol groups of the silane:  $-\text{Al}-\text{O}-\text{Si}-$ .

The microstructure, morphology and mechanical properties of the intermediate region adjacent to the silane-modified surface of the substrate and to the matrix are also

important considerations. If contact is supplied between a polymer and the uncross-linked siloxane/nonreacted silanol bridges, the bonding can take several forms including copolymer formation and interpenetrating polymer networks via methacrylate groups [29,30]. Increased cross-linking of the siloxane structure in the interphase region by adhesive monomers can give higher bond strength and superior resistance to moisture. One other function of adhesive silane monomer is to achieve better wetting of the substrate surface. Although intermediate resin is not necessarily needed with flow viscosity, some products clearly benefit using them [30].

Although comparative studies exist, showing the advantages of various types of surface conditioning methods on various ceramics [31–39], there has been no consensus in the literature regarding the best surface conditioning method for optimum bond strength depending on the luting cements or ceramics used. Therefore, the objectives of this study were to evaluate the effect of current surface conditioning methods on the bond strength of a resin composite luting cement bonded to ceramic surfaces and to identify the optimum method to be used for conditioning the ceramics prior to cementation.

## 2. Materials and methods

Thirty-six experimental groups ( $n = 6$ ) of six types of ceramic materials, namely Finesse (FIN), In-Ceram (INC-AL), Celay (INC-ZR), IPS Empress 2 (EMPII), Procera<sup>®</sup> AllCeram (PRO) and Experimental alumina (EAL) were obtained from the manufacturers. The specimens were in disc forms with 10 mm in diameter and 2 mm in thickness. Three surface conditioning techniques were assessed for the ceramic materials at both dry and thermocycled storing conditions. Tables 1 and 2 summarize the characteristics of surface conditioning methods and ceramic types with codes and manufacturing company names. Before initiating the bonding procedure, the specimens were embedded in acrylic resin blocks ensuring that one surface of the disc remained uncovered for bonding procedures. The exposed surface of each specimen was ground finished to 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 ethylacetate and air-dried. Subsequently,

Table 1  
Characteristics of surface conditioning methods assessed

Conditioning principle		Manufacturer
Hydrofluoric acid	(9.5%, 90 s) (5%, 20 s)	Ultradent Porcelain Etch, South Jordan, USA IPS Empress Ceramic Etch, Vivadent, Schaan, Liechtenstein
Air particle abrasion	(110 $\mu\text{m}$ alumina, 380 kPa, 10 mm, 13 s)	Korox, Bego, Bremen, Germany
Tribochemical silica coating	Rocatec Pre, Rocatec Plus (280 kPa, 10 mm, 13 s), Silane (5 min)	3M ESPE AG, Seefeld, Germany

Table 2  
Types of ceramics with codes, and manufacturing company names

Trade name	Abbreviation	Ceramic Type	Manufacturer
Finesse	FIN	Leucite reinforced	Ceramco, Burlington, NJ, USA
In-Ceram	INC-AL	Glass-infiltrated alumina (70%)	Vita Zahnfabrik, Bad Sackingen, Germany
Zirkonia Blank for Celay	INC-ZR	Glass-infiltrated zirconia	Vita Zahnfabrik, Bad Sackingen, Germany
IPS Empress 2	EMPII	Lithium disilicate	Ivoclar, Schaan, Liechtenstein
Procera AllCeram	PRO	High alumina (99.9%)	Nobel Biocare AB, Göteborg, Sweden
Experimental alumina	EAL	High alumina (99.7%)	Technical University, Tampere, Finland

the specimens were randomly assigned to one of the following three conditioning methods.

### 2.1. Surface conditioning methods

In hydrofluoric acid-etched groups, the ceramic substrates were etched with 9.5% hydrofluoric acid gel for 90 s except EMPII for which etching was performed for 20 s with 5% hydrofluoric acid gel according to the manufacturer's strict regulations. The ceramic surfaces were etched in the laboratory under ventilation, wearing acid-resistant gloves and protective glasses. The etching gel was rinsed in a polyethylene cup and the diluted solution was neutralized using the neutralizing powder (calcium carbonate,  $\text{CaCO}_3$  and sodium carbonate,  $\text{Na}_2\text{CO}_3$ ) for 5 min and washed thoroughly for 20 s as recommended by the manufacturers of FIN and EMPII. The etched substrates were washed and rinsed thoroughly to remove the residual acid after etching, air-dried and coated with a 3-methacryloxypropyltrimethoxy silane coupling agent (Monobond S, Vivadent, Schaan, Liechtenstein). Silane was allowed to remain in contact for 60 s. The surface was then dried with air.

As an alternative conditioning method to the etching, airborne particle abrasion was performed using 110  $\mu\text{m}$  grain sized aluminium trioxide powder at a pressure of 380 kPa from a distance of approx. 10 mm, for 13 s. Following air particle abrasion, silane coupling agent (ESPE-Sil, 3M ESPE AG, Seefeld, Germany) was applied and waited for its evaporation for 5 min.

The third conditioning method was tribochemical silica coating in which the specimens were first conditioned by air-abrasion with 110  $\mu\text{m}$  grain sized aluminium dioxide particles at a pressure of 280 kPa with Rocatec Pre abrasive in a Rocatec Delta device (3M ESPE). Then the specimens were air-abraded with Rocatec Plus abrasive, which was 110  $\mu\text{m}$  grain sized aluminium dioxide modified with salysilic acid, at 280 kPa from a distance of 10 mm for 13 s. The surfaces were coated with silane coupling agent (ESPE-Sil) and allowed to dry for 5 min.

### 2.2. Bonding procedure

Throughout the experiments, the bonding procedures were carried out in accordance with the manufacturers' instructions. All materials were mixed and applied in

a standardized way by the same operator. In the acid etched groups, adhesive resin (Heliobond, Vivadent) was applied as a thin layer, excess resin was removed with air and it was light polymerized (Elipar, 3M ESPE) for 20 s. Light-intensity was 800  $\text{mW}/\text{cm}^2$ . The low-viscous resin cement (Variolink<sup>®</sup> II, Vivadent) was then bonded to the conditioned ceramic specimens using translucent polyethylene molds with inner diameter of 3.6 mm and height of 5 mm. The low-viscous resin was packed against the substrate with a composite-filling instrument. The resins were light polymerized for 40 s. Polyethylene molds were gently removed from the test specimens. While dry samples were kept in a dessicator at room temperature for 24 h prior to testing, the other groups were subjected to thermocycling (custom procedure made by NIOM-Scandinavian Institute for Dental Materials, Haslum, Norway) for 6,000 cycles between 5 and 55 °C in deionised grade 3 water. The dwelling time at each temperature was 30 s. The transfer time from one bath to the other was 2 s.

Specimens were mounted in a jig (Bencor Multi-T shear assembly, Danville Engineering Inc., San Ramon, CA, USA) of the universal testing machine (Llyod LRX, Lloyd Instruments Ltd., Fareham, UK) and the shear force was applied to the adhesive interface until fracture occurred. The specimens were loaded at a crosshead speed of 1.0 mm/min and the stress-strain curve was analysed with Nexygen 2.0 software (Llyod LRX, Lloyd Instruments Ltd, Fareham, UK).

Statistical analysis was performed using SAS System for Windows, release 8.02/2001 (Cary, NC, USA). The means of each group were analysed by two-way analysis of variance (ANOVA), with shear bond strength as the dependent variable, the surface conditioning methods and the ceramic types as the independent factors. *P* values less than 0.05 are considered to be statistically significant in all tests. Multiple comparisons were made by Tukey's adjustment test. Furthermore, one-way ANOVA was used to determine the significant differences between dry and thermocycled conditions.

## 3. Results

The results of the shear bond strength test for hydrofluoric acid etching, airborne particle abrasion and

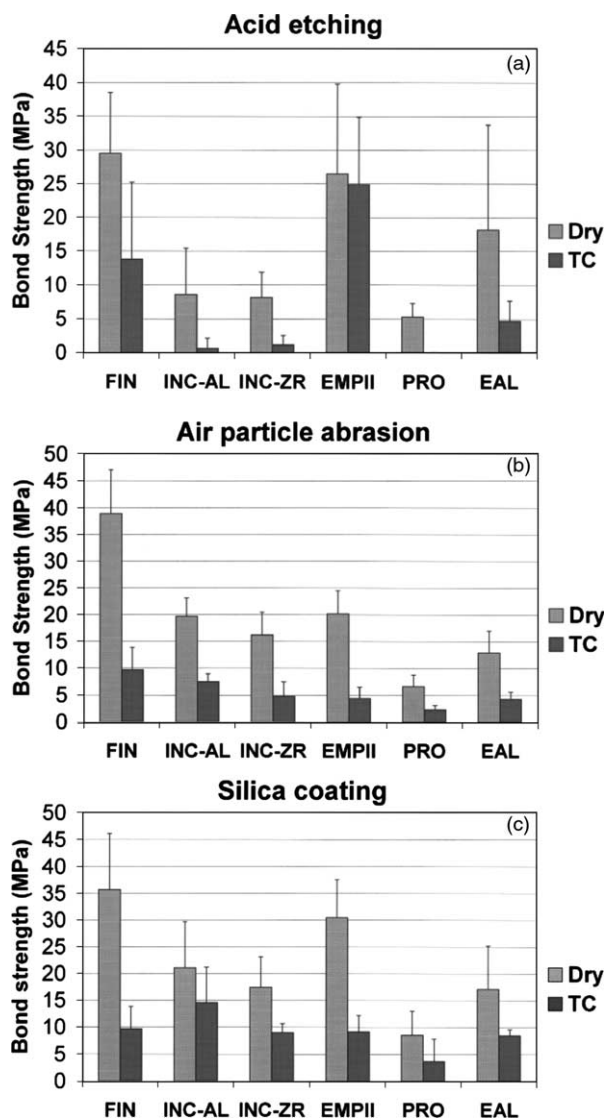


Fig. 1. Shear bond strengths after (a) hydrofluoric acid etching; (b) airborne particle abrasion and (c) tribochemical silica coating at dry and thermocycled conditions. Vertical lines represent the standard deviations. For abbreviations, see Table 2.

tribochemical silica coating are shown in Fig. 1(a)–(c). While ANOVA showed significant influence of the ceramic type on the shear bond strength values ( $P < 0.0001$ ), less difference was found for surface conditioning methods (Tables 3 and 4).

The highest shear bond strengths in dry conditions were obtained with glass ceramics (FIN and EMPII) in all surface

conditioning groups varying between 20.1 and 38.8 MPa. The lowest bond strengths were found with PRO in all conditioning methods ranging from 5.3 to 8.5 MPa.

One-way ANOVA showed that shear bond strength was significantly affected by thermocycling ( $P < 0.001$ ). The least reduction in shear bond strength values after thermocycling was with EMPII ceramic following acid etching conditioning. With other ceramic substrates reduction was higher.

#### 4. Discussion

A requirement for the successful function of ceramic restorations over the years is adequate adhesion between ceramic and tooth substance. Bond strengths are influenced by several factors one of which is the luting cement type [40,41]. Bonding of ceramic to tooth substance is based on the adhesion of luting cement and its bonding resin to the ceramic substrate together with the adhesion of luting cement to enamel and dentin.

Hydrofluoric acid selectively dissolves glassy or crystalline components of the ceramic and produces a porous irregular surface that increases the surface area and facilitates the penetration of the resin into the microretentions of the etched ceramic surfaces. In this study, while acid etching demonstrated higher results for glass ceramics (FIN and EMPII), it did not improve the bond strength of the luting cement to high-alumina ceramics or zirconium oxide ceramic. The differences obtained in bond strength can be explained on the basis of varieties in surface morphology. FIN and EMPII are glass ceramics as the first one is a leucite reinforced and the latter a lithium disilicate ceramic. The primary function of leucite is to raise the coefficient of thermal expansion, consequently increasing the hardness and fusion. The FIN ceramic includes 8–10% leucite crystals which are very receptive to hydrofluoric acid etching before bonding with the resin cement.

The great influence of the type of substrates on the bond strength of Bis-GMA resin to ceramics can be clearly seen in the case of high-alumina ceramics. INC-AL, PRO, EAL are loosely sintered high-alumina ceramics. Principally, acid etching will only affect the grain boundaries visible on the surface. Hydrofluoric acid etching did not create sufficient bond strength on INC-AL due to its high alumina content and it was almost ineffective for dissolving

Table 3  
Results of 2-way analysis of variance for dry conditions

Source of variation	df	Sum of squares	Mean square	F-value	P value
Ceramic type (A)	5	7469.397	1493.879	26.078	<0.0001
Surface Conditioning (B)	2	544.882	272.441	4.756	0.0110
A*B	10	985.058	98.506	1.720	0.0891
Error	86	4926.516	57.285		
Total	103	14262.468			

Table 4  
Results of 2-way analysis of variance for thermocycled conditions

Source of variation	df	Sum of squares	Mean square	F-value	P value
Ceramic type (A)	5	1421.607	284.321	10.017	<0.0001
Surface conditioning (B)	2	216.582	108.291	3.815	0.0256
A*B	10	2127.236	212.724	7.495	<0.0001
Error	92	2611.261	28.383		
Total	109	6515.943			

the glassy phase for micromechanical bonding. Our results are in compliance with the earlier report by Sorenson et al. [42] who showed that hydrofluoric acid etching significantly increased the bond strength of most feldspathic ceramics but did not improve the bond strength to the core part of the INC-AL ceramic. Furthermore, a statistically significant difference was observed between PRO and EAL which are also high-alumina ceramics. The reason for this finding could be attributed to the sintering temperatures and processing such ceramics. EAL contained refractory 'ivory' alumina (99.7%) and has been sintered in higher temperatures which could have affected the grain size. Usually the grain size in alumina ceramics is 1–3  $\mu\text{m}$  but since EAL is at an experimental stage at the moment, the manufacturer claimed that the sintering process was not yet precise and therefore it contained grains 20–30  $\mu\text{m}$  in size including some pores. In the case of PRO, resin composite luting cement exhibited poor adhesion to the ceramic substrates.

While some studies found no obvious correlation between different acids [43], the optimal concentration and duration of their application are not well-established, which is reflected in the variety of concentrations of commercially available hydrofluoric acids. Although less concentration and less duration was used, high bond results were obtained for EMPH after acid etching at both dry and thermocycled conditions. In a study by Madani et al. [33], 5 and 9.5% acid gel was compared and bond strength values with 5% hydrofluoric acid was found to be lower but not significantly different. It should also be noted that in this study, all ceramics tested after acid etching showed higher standard deviations compared with other surface conditioning methods. One conceivable explanation for high standard deviations could be that the poorly adherent precipitates that are deposited at the bottom surface of the grooves and channels, created by acid treatment and rinsing, may weaken resin–ceramic bonds and lead to failure [44,45]. Ultrasonic cleaning could be one option but in this experiment, washing and rinsing were performed using an air–water syringe. In clinical applications, however, when etching will be contemplated at the chairside, this finding might have a big impact on the marginal areas of the restorations.

Air-particle abrasion is a prerequisite for achieving sufficient bond strength between the resins and ceramics. Significant improvement was observed in all ceramic

groups after air-particle abrasion followed by silanization except for FIN and EMPH. Although satisfactory bond results were obtained using air-particle abrasion, the material loss from these procedures after employing on different substrates is important [33]. The data showed, however, that aluminum oxide particles were essential for creating micro-mechanical retention on high-alumina ceramics compared to hydrofluoric acid etching.

The tribochemical silica coating followed by silanization, which increased the silica content on the ceramic surface, evidently enhanced the bond between the ceramic surfaces and the luting cement. Since the silica layer is attached well to the ceramic surface, this provides a basis for silanes to enhance the resin bond. Particular increase was observed for INC-AL, EAL and INC-ZR. Similar findings were obtained in previous studies [21,31,33,34].

In this study a Bis-GMA based resin was used as the luting cement. A high and reliable resin bond to alumina and zirconia ceramics was also achieved with airborne particle abrasion and by using a phosphate monomer (MDP) containing resin composite luting cement. Although Wegner et al. [46] reported better long-term results with MDP containing cements than using the tribochemical silica coating procedure, Özcan et al. [21] did not observe significant differences between MDP or Bis-GMA containing resin cements when tribochemical silica coating was employed. The question still needs to be addressed in further studies whether the luting cement alone and/or the combination with the conditioning method play the crucial role in long-term adhesion to the ceramic.

The possible influence of water storage in experimental studies must also be addressed. Different findings compared to others [23,38,39], especially for PRO after air particle abrasion or silica coating may be due to the storage conditions of the specimens. Usually bond strength values decreased after thermocycling [19,47–49], while some others reported no decrease [20]. Such differences might be explained by the differences in experimental set-up, which is important to keep in mind when in vitro studies are extrapolated to a clinical situation. In this study, the specimens were subjected to shear test after 6000 thermocycles. Although it was well above the recommended cycle number according to ISO [50], one limitation of this study could still be the short-term water storage and lower thermal cycling in comparison to other studies that might make it

difficult to predict the long-term durability of the tested bonding methods.

The present study did not find an ideal surface conditioning technique that could be applied to all types of ceramics. Because many factors affect the bond strengths of resin luting cements to ceramics, it is necessary for dentists to understand the characteristics of the ceramics and the surface conditioning methods in accordance with the cements to be chosen.

## 5. Conclusions

1. Bond strengths of the resin composite luting cement tested on the dental ceramics after surface conditioning techniques varied in accordance with the ceramic types.
2. The findings confirmed that the use of hydrofluoric acid appeared to be the method of choice for bonding the Bis-GMA resin composite luting cement to the ceramics having glassy matrix in their structures.
3. Roughening the ceramic surfaces with air particle abrasion prior to cementation provided higher bond strengths for high-alumina ceramics and the values increased more significantly after silica coating/silanization.
4. Thermocycling decreased the bond strength values significantly after all surface conditioning methods tested.

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