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Microtensile bond strength testing of resin cements

Zeynep Duymus Yesil *

Department of Prosthodontics, School of Dentistry, Atatürk University, Erzurum, Turkey

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ABSTRACT

To investigate the microtensile bond strength (μTBS) and failure mode of resin cements bonded to ceramic blocks following various surface treatments.

Seventy-two Ceramco II (Ceramco Inc., Burlington, NJ) ceramic discs 10 mm in diameter and 4 mm thick were prepared. The ceramic specimens received 8 different surface conditions treatments before the application of resin cement. These surface treatments were sanding with 600-grit silicon carbide paper, microetching with aluminum oxide, sanding followed by silane application, microetching followed by silane application, hydrofluoric acid etching, hydrofluoric acid etching followed by silane application, application of adhesive resin, and combination of the previous two treatments (HF+S+Adh). Seventy-two extracted molars were ground flat at 90° to the long axis of the tooth until a sufficient circular area of dentin was exposed (at least 5 mm in diameter). Three resin cements were applied to these surfaces. After 24 h storage at 37 °C, the non-trimming version of μTBS test was used to produce 1 mm^2 microbars. The microbars were subjected to a tensile load using a modified testing device.

Data were analyzed with 2-way analysis of variance. The interaction between the substrate surface treatment and cement type is significant ($p < 0.001$).

The results of this in vitro study suggest that when the tested ceramic restoration is cemented with a resin cement system, the ceramic should be etched with hydrofluoric acid, silane and adhesive should be applied prior to cementation. The results also suggest that an auto- or light-polymerizing cement should be considered instead of a dual-polymerizing cement.

1. Introduction

Interest in all-ceramic dental restorations has increased in recent years. Ceramic inlays, onlays, veneers, and complete-coverage crowns have gained popularity. These restorations offer superior esthetics compared with metal–ceramic restorations [1,2]. However, ceramic restorations are very brittle and in most situations need to be bonded to tooth structure with resin or composite cements [3,4]. Most modern resin cement kits contain both an adhesive (dentin bonding agent [DBA]) for bonding to the tooth structure and a dual-polymerizing cement (composite) for bonding to the restoration. When bonding ceramic to tooth structure, 2 different interfaces need to be considered: the dentin/adhesive interface and the ceramic/cement interface [5]. The bond strength at both of these interfaces should be optimized, because the lower one will determine the final bond strength of the cemented restoration [6].

Besides the advantages of the indirect ceramic and composite restoration, bonding to the tooth structure is still a challenging matter, due to the fact that the indirect restorative procedure will increase the interfaces for bonding. One interface is at the tooth structure and the other at the fitting surface of the restoration. In order to establish a strong and durable bond, which is necessary for the biomechanical aspect of the tooth-restoration system, appropriate treatment of the respective surfaces is crucial. Various investigations have shown that using adhesive cements increases the fracture resistance of ceramic restorations [7–9].

With contemporary adhesive cements and the new generation of bonding systems, achieving a strong and durable bond to both the tooth structure and the indirect restoration could be feasible [10].

Bond strength measurements are among the methods used to evaluate the effectiveness of adhesive systems, hence predicting their performance in the oral environment. Shear and tensile strength tests are the most widely used. However, various investigations have reported that the mode of failure, occurring after shear bond testing is often cohesive within the substrate rather than adhesive at the interface [11–13]. Cohesive failures are rarely seen clinically with bonded restorations. Testing the bond strength by tensile loading produces more adhesive failures which may favor the evaluation of the true bond strength [14]. However, the results from this test are greatly influenced by specimen

* Tel.: +90 442 2311781; fax: +90 442 2360945.
E-mail addresses: zyesilz@hotmail.com, duymus.1@osu.edu

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geometry and the occurrence of non-uniform stress distributions during load applications [15].

The introduction of the microtensile bond strength test (μTBS test) by Sano et al. [16] has shifted the failure pattern further to occur at the adhesive interface. The small bonded interface of specimens used in this test of approximately 1 mm^2, results in a more uniform stress distribution during loading. Accordingly, higher bond strength values with fewer cohesive fractures can be obtained [17,18].

The purpose of the current study was to investigate the effect of different surface treatments of porcelain blocks on the bond strength to resin cements. In addition, the failure mode was evaluated.

2. Materials and methods

Seventy-two Ceramco II (Ceramco lnc., Burlington, NJ) ceramic discs 10 mm in diameter and 4 mm thick were prepared. Ceramco II is a low-fusing feldspathic ceramic. By weight, it is 60–70% silica, 10–20% alumina, 5–15% soda, 7–15% potassia, and 1–7% calcia, and it contains traces of magnesia, tin oxide, and zirconia. The ceramic was condensed in a cylindrical metal mold, then expressed from the mold and fired according to manufacturer's directions. The surfaces to bond to were wet ground on a polishing machine (Buehler Ecomet V, Buehler Ltd., Lake Bluff, IL, USA) using 600 grit SiC paper. The specimens were then ultrasonically cleaned for 5 min in distilled water and air-dried.

Before application of the three cements to each of the 72 samples of Ceramco II, the surfaces to bond to were treated in one of the following ways:

Group 1 (Control): No surface treatment.

Group 2 (ME): Microetching was performed with an air-abrader (Miniblaster; Belle de St. Claire, Encino, CA) at a pressure of 80 psi with 50-μm particles. The tip of the microetcher was kept 1 mm away from the surface of each specimen and applied for 3 s. All specimens were rinsed under running tap water to remove the debris.

Group 3 (S): A silane solution (provided by the manufacturer of each cement) was applied on the ceramic specimens with a tip applicator (Kerr) and allowed to air-dry. The manufacturers' recommendations were followed for application of each silane solution.

Group 4 (ME+S): Ceramic specimens were microetched as described for Group 2. Silane solution then was applied as described for Group 3.

Group 5 (HF): Ceramic specimens were etched with 8% hydrofluoric acid for 5 min. They were rinsed under running tap water for 5 s and dried with oil-free compressed air from an air-syringe for 10 s.

Group 6 (HF+S): Porcelain specimens were etched with hydrofluoric acid as described for Group 5. Vinyl silane then was applied as described for Group 3.

Group 7: A thin layer of adhesive (Optibond solo plus; Kerr Corp., Orange, CA, USA) was applied with a brush and then light cured for 20 s with a Translux CL (Kulzer and Co. GmbH, Wehrheim, Germany).

Group 8: Porcelain specimens were etched with hydrofluoric acid as described for Group 5. Adhesive was applied as described for Group 7.

Each of the 8 main groups was divided into 3 sub-groups ($n = 3$), 1 for each of the 3 cements tested.

Seventy-two extracted molars stored in saline solution were used for bond strength testing. Each molar was ground flat at 90° to the long axis of the tooth until a sufficient circular area of dentin was exposed (at least 5 mm in diameter). The tooth was mounted in the same mounting plates as used for ceramic specimen preparation, and the dentin surface was sanded flush with the holder with 400-grit followed by 600-grit wet silicone carbide paper. These specimens were divided into 8 groups ($n = 9$). These 8 groups ($n = 9$) were divided into 3 groups ($n = 3$). The three resin cements tested are listed in Table 1. Cement were mixed and applied according to the manufacturer's directions.

Table 1
Resin cements used in this study

Cement	Manufacturer	Polymerization method
Panavia 21	Kuraray Co Ltd., Osaka, Japan	Auto polymerized
Nexus	Kerr	Light polymerized
Nexus	Kerr Corp., Orange, CA	Dual polymerized

The built-up specimens were stored in distilled water at 37 °C for 24 h. Using a low-speed cutting saw (Buehler Isomet 1000 Low Speed Saw, Buehler Ltd., Lake Bluff, IL, USA), each specimen was cut into slabs of 1 mm thickness, starting at the cement side, through the ceramic block perpendicular to the bonded interface. The cutting advanced until 1 mm remained in order to keep the slabs fixed in position. The block of slabs was then rotated 90° and again cut perpendicular to the bonded interface to gain $1 \pm 0.1\ mm^2$ rectangular microbars. During this second cutting procedure, premature debonding of the cement from the substrate occurred in a few cases, but for the ceramic controls in all cases. The surviving microbars per built-up specimen were counted after cutting. Ten microbars were selected randomly and their cross-sectional areas were measured with a digital caliper (Mitutoyo Corp., Japan) before testing. The other remaining microbars were left without testing. The microbars were glued to a testing device by means of a light-curing adhesive (Clearfil SE Bond, Kuraray Co., Japan).

2.1. *Testing device*

The device to test the μTBS was specially designed to facilitate accurate alignment of the microbar with the applied force during testing. The device is composed of two stainless steel articulating members, which are attached to each other at one end by a 0.35 mm thick brass sheet. This attachment permits hinge movement of the two parts and ensures application of a pure tensile force to the microbar specimen, which is glued to the free ends of the device. By a using a Hounsfield testing machine (Hounsfield Test Equipment Company, HTE 37 Fullerton Road, Croydon, England) at a cross head speed of 1 mm/min, a force is applied to the lower member via a steel ball, which loosely fits in an outlet in the upper member.

To determine the mode of failure, all specimens were observed immediately after fracturing under a stereomicroscope.

Statistical analysis was carried out using SPSS statistical software package 11.5 (SPSS Inc., Chicago, IL, USA). Analyses of variance were performed with the bond strength as the dependant variable.

3. Results

The means and standard deviations of the microtensile bond strength of all tests are compiled in Table 2.

When analyzing the effect of using different surface treatments for the ceramic substrate on the bond strength within the three types of cements, it was found that only the HF+S+adhesive treatment with Panavia 21 (29.3 MPa) cement gave a significantly higher bond strength ($p < 0.001$). It was found that the silane

Table 2
Means (MPa) and standard deviations of the μTBS test of three cements bonded to ceramic after various surface treatments (n = number of microbars tested)

Cement	Treatment	Mean (SD)	Lost microbars
Nexus (dual polymerized)	Control	9.0 (3.1)	30
	ME	12.3 (4.2)	30
	S	10.2 (5.2)	30
	ME+S	12.4 (6.0)	30
	HF	13.6 (5.7)	30
	HF+S	15.7 (6.9)	30
	Adhesive	13.7 (3.1)	30
	HF+S+adhesive	17.8 (7.1)	30
Nexus (light polymerized)	Control	9.0 (3.1)	30
	ME	14.5 (4.8)	30
	S	11.8 (6.3)	30
	ME+S	15.9 (6.9)	30
	HF	16.8 (6.0)	30
	HF+S	17.5 (7.3)	30
	Adhesive	15.4 (3.3)	30
	HF+S+adhesive	19.1 (7.6)	30
	Control	9.1 (3.1)	30
	ME	17.9 (6.9)	30
Panavia 21	S	12.7 (4.8)	30
	ME+S	21.6 (7.0)	30
	HF	19.7 (5.1)	30
	HF+S	26.1 (7.8)	30
	Adhesive	18.3 (3.8)	30
	HF+S+adhesive	29.3 (8.2)	30

ME: microetching, HF: hydrofluoric acid (8%), S: silane primer.

Table 3
Analysis of variance

Source of variation	F value	p Value
Treatment	9.514	0.000
Cement	7.220	0.000
Treatment × cement	21.024	0.000

Table 4
Shear bond strength (MPa) of resin cement (mean ± SD), and multi-comparison (Duncan) test results

Cement	Polymerization	Bond strength
Nexus	Dual polymerized	13.08 ± 5.1a, b
Nexus	Light polymerized	15.00 ± 5.0a, b
Panavia 21	Autopolymerized	19.33 ± 7.1a

a, b: different from each other ($p<0.01$).

treatment with Nexus (dual polymerized) (10.20 MPa) cement gave a significantly low bond strength ($p<0.001$).

The results of the ANOVA are presented in Table 3 and show that the interaction between the substrate surface treatment and cement type is significant ($p<0.001$).

As a result of multiple-comparison test, it was statistically determined that, Nexus (dual polymerized) and Nexus (light polymerized) showed similar bond strength values, however Panavia 21 showed different bonding strength values than the others (Table 4). The results suggest that an auto- or light-polymerizing cement should be considered instead of a dual-polymerizing cement.

Ceramic substrate showed 60% adhesive failures, 12% cohesive, and 28% mixed failures (adhesive and cohesive).

4. Discussion

The shear test, which is frequently used to determine bond strengths, often produces cohesive bulk fracture of the substrate away from the bonding interface. This way of fracturing gives only limited information about the true bond strength [13]. The frequent upredictability of the mode of failure is caused by surface flaws, internal material flaws in the substrate material, the adhesive layer, or the bonded composite and flaws in the interface [14]. In addition, the results of the shear test, and the tensile bond test as well, are greatly influenced by the non-uniform distribution patterns of the applied stress, with stress concentrations at certain sites on the specimens [19]. On the other hand, with the μTBS, the small dimensions and small interfacial bonding zone of the samples reduce to a great extent the number of these defects and result in a more uniform distribution of the applied stresses [18].

Several researchers have studied different ceramic surface treatments used to optimize bond strength at the ceramic/cement or ceramic/composite interface [20–24]. Mechanical roughening with coarse diamond, airborne particle abrasion with aluminum oxide and etching using different types of acids are all among the methods used to enhance micromechnical retention [25]. HF is commonly used to etch porcelain for indirect restorations [26,27]. As alternatives to avoid the hazards HF, acidulated phosphate fluoride [28] or phosphoric acid were also investigated. However, their effectiveness on the enhancement of the bond strength is still doubtful [29]. The benefit of HF is that it creates surface pits for micromechanical attachment by preferential dissolution of the glass phase from the ceramic matrix [30]. Treatment of the etched surface with silane improves the wettability and forms a covalent bond with both the porcelain and resin cement [31].

The results of the current study support the importance of HF etching and silanization for treating ceramic surfaces, as no surface treatment or applying only an adhesive to the surface resulted in debonding of all the samples during the cutting procedures (pretesting failures).

It was found that only the HF+S+adhesive treatment gave significantly higher bond strength. The present study shows strong evidence that the HF+S+adhesive treatment of substrates for ceramic use is the most consistent type of treatment.

The significantly higher bond strength, which was found after adding an adhesive to the HF+S treated surface of the composite could be related to the effect of the adhesive of infiltrating and repairing the damaged resin matrix as well as increasing the wettability of the treated surface. A comparable positive effect on the bond strength of adhesive application on composite surfaces after aluminum oxide air abrasion was reported by Latta et al. [32]. Like HF, air abrasion may lead to surface defects, which can be repaired in a similar way.

The results of this study and others published previously [6,8–12] suggest that the most efficiently etched ceramic surface in combination with a silane treatment usually provides the highest bond strengths. The different results from study to study probably are due to the use of different porcelains, different hydrofluoric acid concentrations and etching times, and different microetching pressures and particles.

It follows that the application of silane after ceramic surfaces have been etched is a crucial step. Specimens that were sanded or microetched but had no silane treatment were associated with low bond strengths.

Kupiec et al. [22] reported that best bonds were obtained immediately after bonding and at 3 months when silane was used. Kamada et al. [23] found that a silane coupling agent with or without phosphoric acid etching improved the shear bond strength between ceramic and each of the 4 luting agents that they studied; they also found that, when a silane coupling agent was used, there was no difference in the bond strengths of specimens subjected to water storage alone or to 20,000 thermal cycles. Madani et al. [24] found that when Panavia 21 resin cement was used with Clearfil silane (Kuraray Co. Ltd., Osaka, Japan), air particle abraded In-Ceram ceramic specimens were associated with higher mean shear bond strength values than specimens etched with 9.5% hydrofluoric acid.

Frankenberger et al. [33] compared the dentin bond strength and marginal adaptation of direct resins and ceramic inlays. They found that prepolymerization of the bonding adhesive increased dentin bond strengths in all situations.

Although several different surface treatments and resin cements have been studied, it is not clear whether mechanical roughening (with the use of air particle abrasion, diamond burs, or hydrofluoric acid etching), chemical bonding (with silane), or some combination of the 2 is the most effective surface treatment for bonding ceramic restorations with resin cements. Moreover, with the introduction of several new resin cements, there is confusion among clinicians about which product and technique to use.

This study it was found that only the HF+S+adhesive treatment with Panavia 21 cement gave a significantly higher bond strength. The results suggest that an auto- or light-polymerizing cement should be considered instead of a dual-polymerizing cement.

It is evident that there is no consensus on the type of adhesives to be used with the resin cements.

The small differences in bond strength values between the three brands of resin cements bonded to the preprocessed composite are most likely due to the differences in composition, filler type and filler concentration of the cements.

The modes of failures evaluated in this study after μTBS testing the resin cements bonded to ceramics showed that the majority of the fractures were through the adhesive interface. This is in agreement with Della Bona et al. [34], who found that most of the failures obtained from μTBS testing of composite bonded to hot-pressed ceramic materials occurred within the adhesion zone. A finite element analysis study for rectangular specimens carried out by Phrukkanon et al. [35] revealed that stresses are concentrated at the corners and the central area between the corners of the adhesive interface. This explains the predominant mixed type of failure in our study, where remnants of ceramic or cement tend to remain attached to the corners of the bonded interfaces.

5. Conclusions

Within the limitations of this study and in regard to all three cements tested, bond strengths were highly dependent on surface conditioning. Ceramic restoration is cemented with a resin cement system, the ceramic should be etched with hydrofluoric acid, silane and adhesive should be applied prior to cementation. The results also suggest that an auto- or light-polymerizing cement should be considered instead of a dual-polymerizing cement. It was found that only the HF+S+adhesive treatment with Panavia 21 cement gave significantly higher bond strength.

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