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## Original Article

# Effects of different surface-treatment methods on the bond strengths of resin cements to full-ceramic systems

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**KEYWORDS**bonding strength;  
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surface procedure

**Abstract** *Background/purpose:* Success with resin-bonded all-ceramic restorations is highly dependent on obtaining a reliable bond, which has to integrate all parts of the system into one coherent structure. There are still bonding problems that reduce the clinical success between restorations and cement in applications of full-ceramic systems. The purpose of this study was to determine whether surface-treatment methods can provide a stronger bonding between the cement and the full-ceramic materials by changing the superficial properties of the ceramic materials.

*Materials and methods:* Four different surface processes (sandblasting with Al<sub>2</sub>O<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub> sandblasting and chemical etching with hydrofluoric acid gel, blasting with soda and glass beads, and the Bateman etch retention system) were used as surface-treatment procedures. The first three procedures were applied to samples of IPS Empress and IPS Empress 2 ceramic discs. Sandblasting with Al<sub>2</sub>O<sub>3</sub>, blasting with soda and glass beads, and the Bateman etch retention system were applied to samples of In-Ceram ceramic discs. The discs were cemented to composite bars. Two types of cement were used, Rely X adhesive resin cement and Rely X modified glass ionomer cement and, all the samples were subjected to a shear test to evaluate their bond strengths.

*Results:* With the IPS Empress and the IPS Empress 2 ceramic discs, the best bonding was obtained in the group etched with Al<sub>2</sub>O<sub>3</sub> sandblasting and hydrofluoric acid after cementation using the Rely X adhesive resin cement. The surfacing procedures applied to the In-Ceram materials did not change their bonding strengths.

*Conclusions:* The *in vitro* findings from this study indicate that surface-treatment procedures applied to the IPS Empress and the IPS Empress 2 full-ceramic systems are important when

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cement types are considered. In contrast, cement types and surface-treatment methods had no effect on changing the bond strength of the In-Ceram ceramic system.

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## Introduction

Ceramics are still the most aesthetically pleasing materials available for restorative dentistry. However, due to the metal infrastructure of metal-supported ceramic systems, which were developed to increase the physical properties, esthetic and biological adaptations cannot be fully obtained.<sup>1,2</sup> Full-ceramic systems were developed to achieve satisfactory esthetic and biological adaptations. An increasing number of all-ceramic materials and systems are presently available for clinical use.<sup>3,4</sup> At present, the all-ceramic materials represent preferential visual properties for highly esthetic restorations. As a result of the variety of materials and increase in the number of laboratories, which have these systems, full ceramics are now one of the primary choices of doctors and patients and full-ceramic restorations have gradually become the preferred option. Due to the improvements in their mechanical properties, these materials are used to restore not only single tooth defects but also multiunit defects now.<sup>1–5</sup> Furthermore multiple clinical studies have documented the excellent long-term success of resin-bonded restorations such as porcelain laminate veneers, ceramic inlays and onlays, resin-bonded fixed partial dentures, and all-ceramic crowns.<sup>6–13</sup> Improvements in full ceramics also led to improvements in the cements used to adhere these restorations to the teeth. The natural brittleness of some ceramic materials, specific treatment circumstances, and certain clinical conditions require effective resin bonding of the completed ceramic restoration to the supporting tooth structures for long-term clinical success. A strong and durable resin bond provides high retention, improves marginal adaptation, prevents microleakage, and increases the fracture resistance of the restored tooth and the restoration.<sup>6,8,10</sup> Adhesive bonding techniques and modern all-ceramic systems offer a wide range of highly esthetic treatment options. Bonding to traditional silica-based ceramics is a predictable procedure yielding durable results when certain guidelines are followed.<sup>12</sup>

Today, it is recognized that adhesion of the full-ceramic restorations with the conventional cements reduces the clinical success; furthermore, microleakage, which can appear with the conventional cements, can also cause coloring of the crown.<sup>11</sup> Resin cements have become the dominant cementation materials in recent years since they increase the mechanical resistance of the restoration and prevent microleakage.<sup>14–16</sup>

The mechanical retention provided by the surface treatments is of paramount importance for proper adhesion.<sup>12</sup> Previous studies investigated the bonding problems experienced with the full-ceramic restorations and also attempted to eliminate those problems by strengthening the bond by changing the surface properties of the ceramic materials.<sup>12,16–23</sup> Although comparative studies were

conducted that showed the advantages of various types of surface-conditioning methods on various ceramics, there is no consensus in the literature regarding the best surface-conditioning method to produce optimal bond strengths depending on the luting cements and the ceramics used.<sup>23,24</sup> Therefore, the objectives of this study were to examine the effects of current surface-conditioning methods on the bond strength of resin-based luting cements bonded to ceramic surfaces and to identify the optimal method that can be used to condition ceramics before cementation. We also attempted to find an ideal surface procedure and bond for each system by changing the surface properties of the IPS Empress, the IPS Empress 2, and the In-Ceram, which are the most common full-ceramic materials presently used in dentistry.

## Materials and methods

In this study, 180 full-ceramic samples were prepared in accordance with the instructions of the manufacturers. Then samples were divided into three groups for each ceramic system and different surface-treatment procedures were applied to each of the 20 test samples of each group. The procedures applied to the three groups of the IPS Empress, leucite glass ceramic (Ivoclar, Schaan, Liechtenstein), and the IPS Empress 2, lithium-disilicate glass ceramic (Ivoclar, Schaan, Liechtenstein), full-ceramic systems were sandblasting, 50- $\mu\text{m}$   $\text{Al}_2\text{O}_3$  (Korox, Bego, Bremen, Germany), blasting (Perlablast, 50  $\mu\text{m}$ ) (Bego Perlablast, Bego, Bremen, Germany) + glass beads, and sandblasting (50- $\mu\text{m}$   $\text{Al}_2\text{O}_3$ ) + acid etching with 9.6% hydrofluoric (HF) acid (Porcelain Etch Gel; Pulpdent, Watertown, Massachusetts, USA).

These procedures were found to be insufficient to achieve a surface difference in the In-Ceram (a glass-infiltrated aluminum-oxide ceramic) (Vita Zahnfabrik, DZ880, Bad Säckingen, Germany) samples because of their mechanical and chemical resistances. Therefore the procedures applied to the In-Ceram samples were sandblasting (50- $\mu\text{m}$   $\text{Al}_2\text{O}_3$ ), blasting (Perlablast 50  $\mu\text{m}$ ) + glass beads, and Bateman etch retention system (BERS).

The first 20 samples of all the groups that were prepared for each ceramic system were blasted in a media-blasting device (Comblabor CL-FSG 3, Heraeus Kulzer, Hanau, Germany) at a pressure of 2.5 bar using 50- $\mu\text{m}$   $\text{Al}_2\text{O}_3$  for 14 seconds. The second 20 samples of all the groups were subjected to blasting with soda and glass beads. For this purpose, the Perlablast blasting procedure was applied at a pressure of 2.5 bar using 50- $\mu\text{m}$  soda and glass beads for 14 seconds. Then 9.6% HF acid gel was applied to the last 20 samples of the IPS Empress and the IPS Empress 2 ceramic systems for 90 seconds. The acid was then washed off the samples to clean them.

The surfacing procedure, which is explained in this section, was only applied to the last 20 In-Ceram samples. The suspension used in the study was prepared from a solution of 2 g Aerosil 380 (Degussa, Frankfurt, Germany), 0.09 g Beigostat phosphate diester antiflocculant, and more than 0.1 g of silicon resin in 40 g ethanol. The particle size of Aerosil 380 was 7 nm. The prepared suspension was applied to the samples, which were then kept at 960°C in a furnace for 30 minutes following evaporation of the ethanol.<sup>15,22</sup>

Next, all of the samples were ultrasonographically cleaned in distilled water and air-dried. A sterilization procedure was applied using a pressure steam engine (Bego, Triton, Germany) at a pressure of 3 bar and 133°C for 10 seconds.

After the surface procedures were conducted on the IPS Empress, the IPS Empress 2, and the In-Ceram samples, composite bars of A1 color (Z-250 3M Filtek, 3M Dental, St. Paul, Minnesota, USA) were prepared for the cementation procedure using 3-mm-diameter transparent pipettes. These bars were fixed to the ceramic samples using two types of cement. For this reason, test samples prepared for each group were divided two groups. Ten samples were luted with the composite resin Rely X adhesive resin cement (ARC) (3M Dental), and the other ten samples were luted with the resin-modified glass ionomer cement Rely X modified glass ionomer cement (3M Dental). After cementation, all the samples were kept at room temperature in water for 24 hour. A thermocycle procedure was applied using 5°C and 55°C bath solutions. The cycle duration was 10 minutes in each bath solution. Each sample underwent a total of 500 thermal cycles.

At the end of the procedures, a shear bond test was applied using a Lloyd Universal Test Device (Lloyd Instruments, LRX, Fareham Hants, UK) at the Prosthodontics Laboratories of the Faculty of Dentistry, Ankara University. The prepared samples were placed into the test device using special acrylic molds. The knife-edge-shaped apparatus used in our study was placed between the joint of the ceramic disc and the composite material. After the system was turned on with a force of 0.5 mm/min, the value at which the ceramic disc and the composite material ruptured was recorded (Fig. 1).

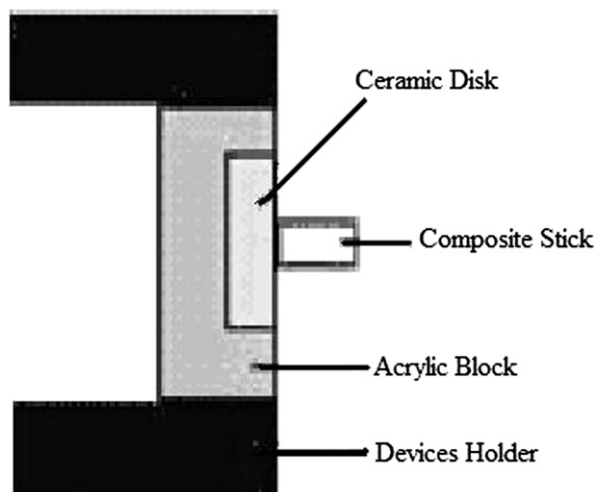


Figure 1 Scheme of the test sample.

The Kruskal-Wallis statistical test was used to evaluate any performance variation in the bond achieved using the surface procedures applied to the ceramics. Values were interpreted with a multiple-comparison test. A Mann-Whitney U-test was used to compare luting cements applied to the ceramics with the sample surface procedures.

## Results

Provided that the cement type was the same, when the same surface procedures applied to the ceramics were compared with each other using the Rely X ARC composite resin cement, the tests indicated that the IPS Empress and the IPS Empress 2 samples showed statistically significant differences among all the three groups of ceramic systems (sandblasted with 50- $\mu\text{m}$   $\text{Al}_2\text{O}_3$ , blasted with Perlalablast + glass beads, and sandblasted with 50- $\mu\text{m}$   $\text{Al}_2\text{O}_3$  + acid etched with 9.6% HF acid). There was no statistical difference between the In-Ceram groups (Table 1).

As a result of the  $\text{Al}_2\text{O}_3$  and the glass beads + soda blasting procedures, the Rely X ARC composite resin cement achieved higher bonding values with the IPS Empress and the IPS Empress 2 samples than with the In-Ceram samples, but the results were not statistically significant. Because the etching procedure with 9.6% HF acid and the sandblasting with 50- $\mu\text{m}$   $\text{Al}_2\text{O}_3$  were not applied to the In-Ceram samples, a comparison was made only between the samples of IPS Empress and IPS Empress 2, and no difference was found. However, because the BERS method was applied only to the In-Ceram samples, those results were not included in the evaluation at this stage. No difference was found in the test results of the three ceramic systems in which the Rely X modified glass ionomer cement was used (Table 2).

The surfacing procedures applied to each ceramic type were compared in terms of cement types. The results of the Mann-Whitney U-test indicated that in all the complete-ceramic types used in the study and with the applied surfacing procedures, the Rely X ARC composite resin cement produced the strongest bond (Fig. 2).

## Discussion

A requirement for the clinical success of ceramic restorations over time is appropriate adhesion between the ceramic and the tooth substances.<sup>11,12,23</sup> Bonding of a ceramic to the tooth substance is based on adhesion of the luting cement and its bonding resin to the ceramic substrate together with the adhesion of the luting cement to the enamel and the dentin. Bond strengths are influenced by several factors, one of which is the type of luting cement.<sup>1-15</sup>

A strong resin bond relies on micromechanical interlocking and chemical bonding to the ceramic surface, which requires roughening and cleaning for an adequate surface activation.<sup>11</sup> Common treatment options are grinding, abrasion with diamond rotary instruments, airborne particle abrasion with aluminum oxide, acid etching, and combinations of any of these methods. Acid etching with the solutions of HF acid or ammonium bifluoride can achieve proper surface texture and roughness.<sup>11-14</sup>

**Table 1** Shear bond test results applied to all the test samples after cementation using the Rely X ARC.

	Surface treatment	n	$\bar{X}$	SD	Mean rank
IPS Empress	Sandblasting (50- $\mu\text{m}$ $\text{Al}_2\text{O}_3$ )	10	24.1948	6.3259	17.40*
	Blasting (Perlablast 50 $\mu\text{m}$ ) + glass beads	10	21.0234	4.5559	9.90*
	Sandblasting (50- $\mu\text{m}$ $\text{Al}_2\text{O}_3$ ) + acid etch (HF)	10	28.9786	4.9729	19.20*
IPS Empress 2	Sandblasting (50- $\mu\text{m}$ $\text{Al}_2\text{O}_3$ )	10	28.1307	3.7986	15.60**
	Blasting (Perlablast 50 $\mu\text{m}$ ) + glass beads	10	23.9273	4.8867	9.80**
	Sandblasting (50- $\mu\text{m}$ $\text{Al}_2\text{O}_3$ ) + acid etch (HF)	10	31.8899	4.2475	22.10**
In-Ceram	Sandblasting (50- $\mu\text{m}$ $\text{Al}_2\text{O}_3$ )	10	18.6846	3.2054	19.70
	Blasting (Perlablast 50 $\mu\text{m}$ ) + glass beads	10	15.9997	1.3845	10.30
	BERS	10	17.7850	2.9919	16.50

The differences are statistically significant that placed at the two ends of vertical lines.

\*A P value <0.05 and \*\*P < 0.01.

ARC = adhesive resin cement; BERS = Bateman etch retention system; HF = hydrofluoric; SD = standard deviation.

Because the concept of etching porcelain surfaces was introduced and adhesive cementation of full-ceramic restorations was reported, several authors demonstrated that the concentrations and etching periods must be adjusted to each specific type of ceramic to optimize the bond strength.<sup>11,14,23</sup>

The HF acid selectively dissolves glassy or crystalline components of the ceramic and produces an irregular porous surface that increases the surface area and facilitates the penetration of resin into micro-retentions on the etched ceramic surface.<sup>11</sup> In this study, acid etching demonstrated better results with glass ceramics (IPS Empress and IPS Empress 2), although it was not used to improve the bond strength of luting cements to high-alumina ceramics (In-Ceram). The BERS that relies on incorporation of plastic chips on the surface of a specimen was used for those samples. This procedure was introduced first by Saudon M and Asmussen E. in 1994.<sup>6</sup> The plastic chips are subsequently burnt-out to leave pits on the fitting surface of the ceramic restoration.<sup>15–22</sup> In this study, the effect of these surface pits on the bond strength of test specimens was compared to etched and the sandblasted samples of high-alumina ceramics.

Acid etchants used for silica-based dental ceramics do not sufficiently roughen the surface of aluminum-oxide

ceramics. Airborne particle abrasion with  $\text{Al}_2\text{O}_3$  is effective and practical for creating an activated and roughened surface on aluminum-oxide ceramics.<sup>23</sup> The number, the size, and the distribution of leucite crystals influence the formation of micro-porosities created by acid etching. Leucite crystals grow during the cooling phase of the ceramic-firing process. Some low-fusing ceramics and glass ceramics contain only minimal amounts of leucite crystals, which may inhibit the formation of highly retentive micro-porosities with acid etching.<sup>11</sup> For the leucite-reinforced feldspathic porcelain, the IPS Empress and the solutions of 9.6% HF acid applied for 90 seconds were the most successful in the present study. The lithium-disilicate glass ceramic, the IPS Empress 2, has a high crystalline content and exhibited significantly higher bond strengths than the IPS Empress independent of the surface conditioning. This result seems consistent with the opinion that the ceramic microstructure also significantly influences the fracture resistance of the composite-ceramic adhesion zone.

Changes occur in the surface topography after sandblasting procedures.<sup>4,5,7</sup> This technique was included in the present study as it is a commonly employed procedure in prosthodontic laboratories, and dental offices have miniaturized devices, which facilitate its use. The particle size, procedure duration, and pressure and distance used in the

**Table 2** Shear bond test results applied to all the test samples after cementation using the Rely X RMGIC.

	Surface treatment	n	$\bar{X}$	SD	Mean rank
IPS Empress	Sandblasting (50- $\mu\text{m}$ $\text{Al}_2\text{O}_3$ )	10	11.5761	3.3028	17.80
	Blasting (Perlablast 50 $\mu\text{m}$ ) + glass beads	10	10.6911	2.0520	12.90
	Sand blasting (50- $\mu\text{m}$ $\text{Al}_2\text{O}_3$ ) + acid etch	10	11.5656	3.1561	15.80
IPS Empress 2	Sandblasting (50- $\mu\text{m}$ $\text{Al}_2\text{O}_3$ )	10	11.9441	2.9474	13.30
	Blasting (Perlablast 50 $\mu\text{m}$ ) + glass beads	10	10.9681	2.0059	15.90
	Sandblasting (50- $\mu\text{m}$ $\text{Al}_2\text{O}_3$ ) + acid etch (HF)	10	12.2202	2.5551	17.30
In-Ceram	Sandblasting (50- $\mu\text{m}$ $\text{Al}_2\text{O}_3$ )	10	10.2785	1.9391	16.70
	Blasting (Perlablast 50 $\mu\text{m}$ ) + glass beads	10	10.1580	2.1844	15.60
	BERS	10	9.8917	1.6157	14.40

BERS = Bateman Etch Retention System; HF = hydrofluoric; RMGIC = modified glass ionomer cement; SD = standard deviation.



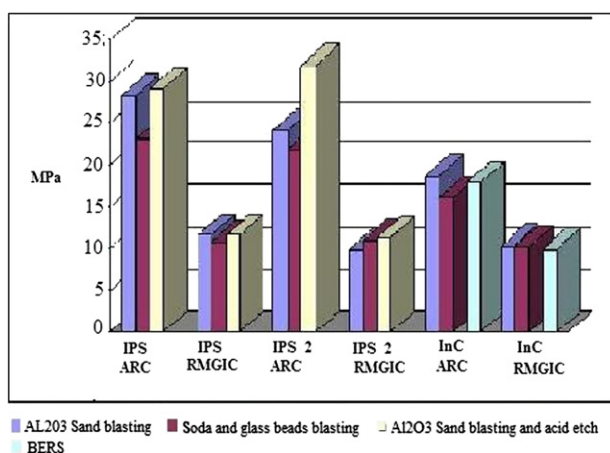


Figure 2 Shear tests results.

procedure are important factors in the performance of the cement bond.<sup>18–21</sup> It was reported that a sandblasting procedure with very high pressure or with very large particles does not increase the bond strength; nevertheless, it causes incompatibility in the restoration as a result of wear.<sup>16,19–21</sup> Kern and Thompson<sup>15</sup> reported that at the end of sandblasting procedures, material loss in the IPS Empress ceramics was 36 times higher than that in the In-Ceram. Shopr et al<sup>16</sup> used an electron microscope to analyze the IPS Empress 2 ceramics to which a 50- and 100- $\mu\text{m}$   $\text{Al}_2\text{O}_3$  were applied. They conducted a shear bond test and reported that surfaces on which 100- $\mu\text{m}$   $\text{Al}_2\text{O}_3$  was applied did not possess sufficient retentive properties, whereas surfaces prepared using 50- $\mu\text{m}$   $\text{Al}_2\text{O}_3$  were more highly etched. They also found at the end of the test that there was a significant increase in the strength of the cement bond. Other researchers reported that chemical and mechanical etching methods can be used together.<sup>13–15</sup>

In the present study, no statistical difference was found between the bonding resistance of the IPS Empress and the IPS Empress 2 samples conditioned with 50- $\mu\text{m}$   $\text{Al}_2\text{O}_3$  and the samples to which 50- $\mu\text{m}$   $\text{Al}_2\text{O}_3$  and the HF acid was applied. These two sample groups showed higher bonding resistance than the groups in which 50- $\mu\text{m}$  soda and glass beads were used as the blasting material, and differences were statistically significant when the Rely X ARC composite resin cement was used (Table 1). For the IPS Empress 2 ceramic group in which 50- $\mu\text{m}$   $\text{Al}_2\text{O}_3$  and HF acid were used together, the most successful statistical result was obtained. This is in accordance with the results of previous studies.<sup>9,10,14,21,24</sup> Micro-pitting of the surface was created by sandblasting, whereas the second phase to remove the glass matrix and lithium orthophosphate of the surface was performed using HF acid, and the results were more closely comparable to sandblasting alone. The findings of this study revealed the importance of HF acid-induced micromechanical retention for resin cement-ceramic bonding. The differences obtained in bond strengths can be explained by the various surface morphologies obtained.

The statistical results of the shear bond test indicate that a higher bonding value was achieved using sandblasting with acid etching in the IPS Empress 2 ceramic group after cementation with the Rely X ARC (Table 2). In the IPS

Empress ceramic group, there was no difference between the sandblasted samples and those subjected to combined sandblasting and acid etching; this is thought to be a result of the acid application period on surfaces etched with sand. As a result, the silica phase of the feldspathic ceramic was dissolved, retentive areas disappeared, and accordingly higher bonding values could not be obtained. There was a predominance of cohesive failure of the porcelain under all experimental conditions.<sup>11</sup> This result is in accordance with those of several studies that evaluated the bond strength at ceramic/cement interfaces after acid etching and/or sandblasting.<sup>7–12</sup> This can be explained by the effects of the different treatments on the porcelain fracture strength, because of modification of its superficial energy, which is an important factor in fracture spreading.<sup>11,14</sup> The present study did not find an ideal surface-treatment method that could be applied to all types of ceramics, because many factors affect the bond strengths of the resin luting cements applied to the ceramics.

## Conclusion

The present study demonstrated that shear bond strengths of the resin composite luting cements tested on the dental ceramics after the surface-conditioning techniques varied in accordance with the type of ceramic. The *in vitro* findings from this study indicated that the surface-treatment procedures applied to the IPS Empress and the IPS Empress 2 full-ceramic systems are important when considering the cement types, although the cement types and surface-treatment methods were ineffective in changing the bonding strength of the In-Ceram ceramic system.

The findings confirmed that the use of HF acid with sandblasting is one of the available methods that can be chosen for bonding the resin-based luting cement to the ceramics with a glassy matrix in their structures.

Within the limits of this study, the BERS method, which was previously recommended for In-Ceram restorations, did not have a noticeable influence on the use of cements in the present study. Conditioning the ceramic surfaces with sandblasting before cementation provided higher bond strengths for high-alumina ceramics.

Further studies are necessary so that the clinicians can understand the characteristics of the ceramics and the surface-treatment methods in accordance with which the cements should be chosen.

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