Evaluation of 24-Hour Shear Bond Strength of Resin Composite to Porcelain According to Surface Treatment

Ahmet Umut Güler, DDS, PhD^a/Fikret Yilmaz, DDS, PhD^b/Çagri Ural, DDS^c/Eda Güler, DDS^d

Purpose: The purpose of the present in vitro study was to examine shear bond strengths of resin composite to porcelain according to surface treatment. Materials and Methods: One hundred eight feldspathic porcelain blocks were prepared. Specimens were divided into nine surface treatment groups: sandblasting with 50- or 110-µm Al₂O₂, acid etching with hydrofluoric acid, applying silane agent, and combinations of these treatments. Microhybrid resin composite was condensed and light cured for 40 seconds on the porcelain specimens, which were then stored in distilled water at 37°C for 24 hours before mechanical testing. Results: The bond strengths were significantly different according to one-way ANOVA. The lowest bond strength was observed in the silane group (4.09 MPa); the highest bond strengths were observed with acid etching + silane (11.97 MPa) and sandblasting with 50- μ m Al₂O₃ + acid etching + silane (12.34 MPa) (no significant difference between groups). Conclusion: Acid etching with 9.6% hydrofluoric acid gel or sandblasting with 50- or 110-µm Al₂O₂ particles alone did not provide adequate bond strength. Silane agent was effective in increasing the shear bond strength of resin composite to porcelain after sandblasting or acid etching. Porcelain treatment with a combination of 50- μ m Al₂O₃ air abrasion, 9.6% hydrofluoric acid, and silane agent provided higher bond strengths than treatment with any of these procedures alone. Int J Prosthodont 2005;18:156-160.

Because of their high esthetic qualities and mechanical stability in the oral environment, porcelain-fused-to-metal restorations are commonly used in daily dental practice.¹ Clinically, failures often begin as porcelain fractures that may be caused by inappropriate coping design, poor abutment preparation, technical errors, contamination, physical trauma, or premature occlusion.^{2,3}

Clinical studies show failure rates of up to 9% for ceramic veneers.⁴ Factors such as impact, fatigue, occlusal forces, and incompatibility between metal and porcelain physical properties may result in porcelain fracture, frequently of a cohesive nature.^{2,5,6} Because of the inherently brittle nature of ceramic restorative materials, failure of metal-ceramic restorations under intraoral conditions is not uncommon.⁷ The majority (65%) of failures are observed in the anterior region (60% in labial, 27% in buccal, 5% in incisal, and 8% in occlusal regions). These fractures are mainly in the maxilla (75%), predominantly at the labial surface.⁸

It is necessary to assess the possible causes of fracture so that the most suitable treatment can be recommended. Depending on the extent of the area to be restored, cost, and time available, treatment may range from making a new prosthesis, faceting, or overcasting to resin composite repair.⁶ Replacement of a failed restoration is not necessarily the most practical solution, for obvious economic reasons and because of the

^aAssistant Professor, Department of Prosthodontics, Faculty of Dentistry, Ondokuz Mayis University, Samsun, Turkey.

^bAssistant Professor, Department of Restorative Dentistry, Faculty of Dentistry, Ondokuz Mayis University, Samsun, Turkey.

^cResearch Assistant, Department of Prosthodontics, Faculty of Dentistry, Ondokuz Mayis University, Samsun, Turkey.

^dResearch Assistant, Department of Restorative Dentistry, Faculty of Dentistry, Ondokuz Mayis University, Samsun, Turkey.

Correspondence to: Dr Ahmet Umut Güler, Ondokuz Mayis Üniversitesi, Dis Hekimligi Fakültesi, Protetik Dis Tedavisi Anabilim Dali, Kurupelit, Samsun 55139, Turkey. Fax: + 90 362 4576032. e-mail: auguler@omu.edu.tr

complex nature of the restoration. The availability of a sound esthetic repair option would be the more practical solution in these situations.⁹ The clinical success of the porcelain repair systems is almost entirely dependent on the integrity of the bond between porcelain and resin composite. The bond is achieved by either chemical or mechanical methods.

Various methods have been introduced to repair fractured porcelain with resin composite. Mechanical roughening of porcelain surfaces with a coarse diamond has improved repair strength.^{10,11} Sandblasting with aluminum oxide (Al₂O₂) is another method of surface roughening,¹² and porcelain can also be etched with hydrofluoric acid or acidulated phosphate fluoride gel to facilitate micromechanical retention of resin composite.¹³⁻¹⁵ Silane coupling agents possess the general chemical structure x-(CH₂)₃Si-(OR)₃ and have the ability to bond chemically to both organic and inorganic surfaces.¹⁶ Silanes were introduced in dentistry by Bowen and Rodriguez,¹⁷ who developed resin composites by adding silanized filler particles to bisphenol glycidyl methacrylate (bis-GMA) resin. The type of resin composite also affects its bond strength to porcelain. It is assumed that larger particle size resin composites or hybrid-type resins at the porcelain interface result in higher bond strengths than smaller-particle resin composites.18,19

The purpose of the present in vitro study was to examine shear bond strengths of resin composite to porcelain according to surface treatment.

Materials and Methods

The materials used in this study are as follows:

- Vita VMK95 (Vident): feldspathic porcelain
- Korox 50 (Bego): 50-μm Al₂O₃ powder
- Korox 110 (Bego): 110-μm Al₂O₃ powder
- Porcelain Etch Gel (Pulpdent): 9.6% hydrofluoric acid gel
- Silane Bond Enhancer (Pulpdent): silane agent
- Prime&Bond NT (Denstply): adhesive primer
- Arabesk Top (Voco): microhybrid resin composite

One hundred eight feldspathic porcelain blocks (Vita VMK95), approximately 7 mm \times 7 mm \times 3 mm, were prepared. All porcelain samples were glazed according to the manufacturers' suggestions. Eighteen blocks (height 20 mm, diameter 30 mm) were prepared with self-curing acrylic resin (Vertex, Dentimex, Zeist) for the purpose of holding the porcelain samples, which had a cohesive resistance to the test machine. On each side of the acrylic resin blocks, three sockets were prepared. The sockets had a depth of 3 mm and a diameter of 10 mm. The sockets were prepared at equal distance to the center of the acrylic resin block and to one another. The porcelain specimens were embedded into the sockets with self-curing acrylic resin. For the purpose of surface standardization, the specimens were gradually ground wet with 320-, 400-, and 600-grit silicon carbide paper for 10 seconds each on a 300-rpm grinding machine (Buehler Metaserv, Buehler). Thus, each of the nine study groups was composed of two acrylic resin blocks, each containing six porcelain specimens. Surface treatment procedures evaluated are as follows:

- Group S₅₀: Specimens were air abraded with 50-µm Al₂O₃ powder (Korox 50) at 60 psi for 5 seconds through a nozzle distance of 10 mm. After sandblasting, the specimens were cleaned with compressed air to remove the remaining powder.
- Group S₁₁₀: Specimens were air abraded with 110μm Al₂O₃ powder (Korox 110) at 60 psi for 5 seconds through a nozzle distance of 10 mm. After sandblasting, the specimens were cleaned with compressed air to remove the remaining powder.
- Group A: In this group, porcelain surfaces were acid etched with 9.6% hydrofluoric acid gel for 2 minutes, rinsed with water for 20 seconds, and dried with oil-free air spray for 20 seconds.
- Group Si: A thin layer of silane agent was applied on the porcelain surfaces with a brush, retained for 2 minutes, then blown gently with air for 1 minute when applicable.
- Group S₅₀Si: Specimens were sandblasted exactly as in group S₅₀. When applicable, a thin layer of silane was applied on the porcelain surfaces as in group Si.
- Group S₁₁₀Si: Specimens were sandblasted as in group S₁₁₀. When applicable, a thin layer of silane was applied on the porcelain surfaces as in group Si.
- Group ASi: Porcelain surfaces were etched as in group A. When applicable, a thin layer of silane was applied on the porcelain surfaces for 2 minutes and allowed to air dry for 1 minute.
- Group $S_{50}ASi$: Specimens were sandblasted exactly as in the 50-µm Al_2O_3 sandblasting group (group S_{50}), then acid etched with 9.6% hydrofluoric acid gel for 2 minutes, rinsed with water for 20 seconds, and dried with oil-free air spray for 20 seconds. When applicable, a thin layer of silane was applied on the porcelain surfaces for 2 minutes and allowed to air dry for 1 minute.
- Group S₁₁₀ASi: Specimens in this group were sandblasted exactly as in the 110-µm Al₂O₃ sandblasting group, then acid etched with 9.6% hydrofluoric acid gel for 2 minutes, rinsed with water for 20 seconds, and dried with oil-free air spray for 20 seconds.

| Group* | Mean (standard deviation) | Range | Difference between groups [†] |
|----------------------|------------------------------|-------------|---|
| S ₅₀ | 5.37 (0.88) | 4.29-6.33 | b |
| S ₁₁₀ | 6.88 (0.25) | 6.51-7.25 | С |
| A | 8.41 (1.26) | 6.77-10.15 | d |
| Si | 4.09 (0.75) | 3.06-5.04 | а |
| S ₅₀ Si | 10.55 (0.63) | 9.94-11.37 | е |
| S ₁₁₀ Si | 7.96 (0.93) | 6.90-9.57 | d |
| ASi | 11.97 (0.47) | 11.28-12.53 | f |
| S ₅₀ ASi | 12.34 (0.62) | 11.12-12.82 | f |
| S ₁₁₀ ASi | 9.87 (1.09) | 7.95-10.97 | е |

| Table 1 Shear Bond Strengths (MP) |
|-----------------------------------|
|-----------------------------------|

*n = 12 specimens per group; see Materials and Methods section for explanation of groups. [†]According to Tukey's HSD test (P < .050).

When applicable, a thin layer of silane was applied on the porcelain surfaces as in the silane group.

A thin layer of adhesive primer (Prime&Bond NT) was applied to the treated porcelain surfaces and polymerized for 20 seconds with a curing light (XL 3000, 3M). After a 2-mm-wide Teflon (DuPont) mold containing a 5-mm-diameter hole was placed onto the surface-treated specimens, microhybrid resin composite (Arabesk Top) was condensed in the hole and light cured for 40 seconds according to the manufacturers' suggestions.

The specimens were then stored in distilled water at 37°C for 24 hours before mechanical testing. Shear testing of all groups was performed on a Lloyd LRX test machine (Lloyd Instruments) using a cross-head speed of 0.5 mm/min (Fig 1). The shear debonding forces were recorded in N and converted into MPa. The statistical analysis of the bond strength data included one-way analysis of variance (ANOVA) and Tukey's honestly significant difference (HSD) test at a 5% level of significance.

Results

The mean shear bond strengths of the nine groups are shown in Table 1. The bond strengths were shown to be significantly different by one-way ANOVA (F =65.538, P = .000). The lowest bond strength was observed in group Si (4.09 MPa).

Although group S_{50} demonstrated higher bond strength (5.37 MPa) compared to group Si, the former group had lower bond strength compared with all other groups. Group S_{110} showed higher bond strength (6.88 MPa) than group S₅₀. Although no statistically significant difference was observed between group $S_{110}Si$ (7.96 MPa) and group A (8.41 MPa), these groups demonstrated average values of bond strength for all groups. Also, there was no statistical significance of the



Specimen in testing apparatus. Fia 1

difference in bond strength between group $S_{110}ASi$ (9.87 MPa) and group S_{50} Si (10.55 MPa). These groups demonstrated the highest bond strength values compared to the above-mentioned groups. The highest bond strength was observed in group ASi (11.97 MPa) and group $S_{50}ASi$ (12.34 MPa), which demonstrated no significant differences between groups. The differences between all groups are listed in Table 1.

Discussion

Intraoral repair of fractured porcelain restorations with resin composite presents a substantial challenge for clinicians. Newer generation multipurpose adhesive systems involve several treatment steps and agents for porcelain repair with resin composite. In this study, the optimum bond strength of resin composite bonded to porcelain was evaluated by applying a combination of different surface treatment methods. Several studies focus on mechanical retention, chemical agents, and the combination of these two methods.^{5,20-22} Because of the insufficient bonding characteristics of the chemical

agents, physical alteration of the porcelain surface must be used together with these agents to promote adhesion. The methods included in the present study were combinations of sandblasting with different particle size Al_2O_3 powder, etching with 9.6% hydrofluoric acid gel, and silane treatment.

Many methods of measuring the in vitro bond strength afforded by porcelain repair systems have been described. These include torsion,¹⁴ flexural,²³ tensile,¹⁵ and shear bond strength tests.^{2,6,24,25} One of the most commonly employed is the shear bond strength test,¹⁵ which was preferred in the present study. The increase in porcelain surface roughness by means of diamond burs and disks contributes to the repair's adhesive resistance, and the use of acids improves adhesion by creating microretentions, producing cohesive failures if used with silane agents.²⁶ Sandblasting with 50- μ m Al₂O₃ is a better method for preparing the surface than bur-performed retention.¹⁶ For this reason, surface treatment with burs was not included in the present study. Hydrofluoric acid attacks the glass phase of ceramics, partially dissolving it and creating microporous retention by exposing areas of crystals that make up the crystalline phase of the materials.²⁷ Microporosity increases the surface area and makes micromechanical interlocking of resin possible. Although various acid solutions can be used for this purpose, hydrofluoric acid has been shown to be the most effective.14,28

The present study showed that sandblasting alone is not sufficient to improve the bond of the resin composite-porcelain interface. The observed results demonstrated similarity to other studies.7,29 Our results indicate that although sandblasting with 110-µm Al₂O₃ produced higher bond strength values than 50- μm Ål₂O₃, sandblasting with 50- or 110- μm Ål₂O₃ particles cannot provide retentive surfaces as satisfactorily as etching with acid. Wolf et al¹⁶ conclude that sandblasting with Al₂O₂ or roughening by burs may achieve satisfactory bond strength in many cases. However, they found that when more durable and higher bond strength is desired, hydrofluoric acid etching is the most significant step in the surface treatment because of the deep acid penetration. The highest bond strengths in 24-hour test findings were in group S₅₀ASi and group ASi. However, etching with hydrofluoric acid is more effective if applied together with sandblasting and silane pretreatment. The silane coupling agents achieved a chemical link between the resin composite and porcelain; moreover, they promoted wetting of the porcelain surface so that it enhanced the flow of the low-viscosity resin composites.^{30,31} Silane coupling agents can also improve the bond of resin composite to porcelain by approximately $25\%.^{26,32}$ In the present study, the silane agent groups

 $(S_{50}Si, S_{110}Si, AS)$ showed higher bond strengths compared with groups without silane (S_{50}, S_{110}, A) . Taking this into account, to achieve a durable and stable bond, silane treatment must be performed after mechanical alteration such as hydrofluoric acid etching or sandblasting. The use of hydrofluoric acid etching was more practical than use of sandblasting prior to silane treatment. However, the hazards of hydrofluoric acid are well-recognized, so care had to be taken during application.²⁵

Conclusions

Within the limits of this study, the following conclusions were drawn:

- 1. Acid etching with 9.6% hydrofluoric acid gel or sandblasting with 50- or $110-\mu m Al_2O_3$ particles did not provide adequate bond strength.
- Although silane agent use alone cannot provide adequate bond strength, silane agent was effective in increasing the shear bond strength of resin composite to porcelain after sandblasting or acid etching.
- Porcelain treatment with a combination of 50-µm Al₂O₃ air abrasion, 9.6% hydrofluoric acid, and silane agent provided higher bond strengths than treatment with any of these procedures used alone.

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Literature Abstract

Retention force and fatigue strength of overdenture attachment systems

The aim of this article was to compare the retention force and fatigue resistance of balland-socket and bar-and-clip overdenture attachments made by two different commercial manufacturers. Twenty samples were used in all, with 4 groups of 5 samples each. A universal testing machine was used to test for fatigue and tensile strength. Insertion and removal of the attachments was performed along the implant's long axis. Fatigue testing was done with the specimens immersed in artificial saliva with 5,500 cycles of insertion and removal of components. Assuming the average number of insertion and removal of dentures to be 3 times a day, this represented 5 years of prosthesis usage. Tensile strength was measured using software with a load cell of 1 kN and speed of 1 mm/min⁻¹. Maximum retention force values were an average of five measurements taken at each interval. Analysis of variance with Tukey's test at 95% confidence level was used to determine the statistical significance. Results show that 5 years of insertion and removal did not decrease retention; nor did it cause a fracture of the attachment systems. Retention force of the bar-clip and the O-ring systems varied significantly between manufacturers. The authors recommended that selection of these attachments for patient use should be based on factors other than retention.

Botega DM, Mesquita MF, Henriques GEP, Vaz LG. *J Oral Rehabil* 2004;31:884–889. References: 14. Reprints: Dr Marcelo Ferraz Mesquita, Avenida Limeira, 901, Piracicaba, Sao Paulo 13414-903, Brazil. e-mail: mesquita@fop.unicamp.br—*Esquivel-Upshaw, San Antonio, TX*

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