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# Effect of Filler Ratio in Adhesive Systems on the Shear Bond Strength of Resin Composite to Porcelains

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### Clinical Relevance

The results of this study indicate that adhesive systems with a high filler ratio provide the greatest strength for resin repair of low-fusing feldspathic porcelain restorations. In addition, self-etching adhesive systems may not be indicated for such repairs.

## SUMMARY

**This *in vitro* study evaluated the effect of six different adhesive systems on the shear bond strength of resin composite to feldspathic and low-fusing porcelains. Sixty porcelain blocks were prepared for each low-fusing (Matchmaker) and feldspathic (MVK95) porcelain specimen. After surface preparation, the porcelain specimens were divided into six groups (n=10) for different adhesive systems (Adper Prompt L-Pop, QuadrantUni1 Bond, Te-Econom, PQ1, One-StepPlus and Prime&Bond NT). After adhesive application, a universal resin composite (FiltekZ250) was condensed on the specimens. The prepared specimens were then stored in distilled water at 37°C for 24 hours, then all the samples were thermal cycled 1000 times between 5°C and 55°C. Shear testing was performed on a universal test machine using a crosshead speed of 0.5 mm/minute. The statistical analysis of the bond strength data included two-way ANOVA. Then, the means were compared by Tukey HSD test (α=0.05). The lowest bond strength was observed in Adper Prompt L-Pop. No statistically significant difference was observed between One-Step Plus and Prime&Bond NT. The highest bond strength was observed in PQ1. When low-fusing or feldspathic porcelain restorations are repaired with resin composite, self-etching adhesive systems may not be indicated. If maximum bond strength is the goal in porcelain resin bonding, adhesive systems that have a high filler ratio should be used.**

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DOI: 10.2341/08-88

## INTRODUCTION

New esthetic restorative materials are constantly being introduced into the market, while porcelain remains a material of choice for most clinicians. Dental porcelain materials exhibit many desirable material properties, including biocompatibility, esthetics, diminished plaque accumulation, low thermal conductivity, abrasion resistance and color stability.[1]

Manufacturers employ glass modifiers to produce dental porcelains with different firing temperatures. Dental porcelains are classified according to their firing temperatures. A typical classification is high fusing, medium fusing, low fusing and ultra-low fusing.[2] The demand for highly esthetic restorations has generated the development of more advanced porcelain systems that can be used for both all-ceramic and porcelain-fused-to-metal restorations. The newest porcelains made of low-fusing porcelain provide many greater advantages over traditional porcelains, including increased opalescence, the ability to polish chairside and less abrasive wear on opposing teeth and materials.[3-4]

Clinically, failures often begin as porcelain fractures, which may result from inappropriate coping design, poor abutment preparation, technical errors, contamination, physical trauma or premature occlusion.[5-6] Porcelain fracture is a serious and costly problem for both the patient and dentist. Instead of removing the restoration, the esthetic repair option is a more practical solution and enables continued use of the existing restoration if it is in an acceptable condition.

The clinical success of porcelain repair systems is almost entirely dependent on the integrity of the bond between the porcelain and resin composite. The bond is achieved either by chemical or mechanical methods. Several studies have focused on mechanical retention, chemical agents and a combination of the two.[7-10]

Modern bonding agents contain three major ingredients (etchant, primer and adhesive) that may be packaged separately or in combination. Bonding systems with different components are currently available and have characteristics, such as total-etch, multiple-bottle (fourth-generation); total-etch, single-bottle (fifth-generation); self-etching primer/adhesive (sixth-generation, Type I); self-etching adhesive (sixth-generation, Type II) and no-mix, self-etching adhesive (seventh-generation). Although most bonding agents are unfilled, some products contain inorganic fillers ranging from 0.5% to 40% by weight. Filler particles include microfillers, also called nanofillers, and sub-micron glass.[11]

Numerous bonding systems for intraoral porcelain repair have been suggested, many of which are considered interim but are still preferable to replacement, as it is important to salvage an extensive fixed restoration even for a few years, if possible. If only a small part is missing, resin composite should be the repair material of choice, because of its esthetic appearance and ease of manipulation.[7,12] Recent applications of adhesive dentistry have simplified the bonding and luting procedures. The bonding performance of time saving and simplified materials has been evaluated in comparison with multistep products.[13-15]

With the introduction of new resin cements and adhesive systems, these circumstances may be confusing to clinicians in terms of which product to use. The current *in vitro* study evaluated the effect of six different adhesive systems on the shear bond strengths of resin composite to feldspathic and low-fusing porcelains.

## METHODS AND MATERIALS

Sixty porcelain blocks, approximately 7 x 7 x 2 mm, were prepared for low-fusing porcelain (Matchmaker, Davis Ltd, Letchworth, England) and feldspathic porcelain (MVK95, Vita, Germany). The porcelain specimens were glazed according to the manufacturers' suggestion.

Sixty acrylic blocks (height of 20 mm and diameter of 30 mm) were prepared with self-polymerized acrylic resin (Vertex, Dentimex, Zeist, Holland) for the purpose of holding the porcelain specimens that had a cohesive resistance to the test machine. On one side of each acrylic block, a socket was prepared, having a depth of 3 mm and a diameter of 10 mm. The sockets were prepared in the center of the acrylic blocks and the porcelain specimens were embedded into the sockets with self-polymerized acrylic resin. For purposes of surface standardization, the specimens were gradually sanded with 400, 600 and 1000 grit wet silicon carbide paper for 10 seconds each on a 300 rpm sand machine (Buehler Metaserv, Buehler, Germany).

The specimens were air-abraded with 50 µm $Al_2O_3$ powder (Korox 50, Bego, Germany) at 60 psi for five seconds through a nozzle distance of 10 mm. After sandblasting, the specimens were cleaned with compressed air to remove the remaining powder. All the porcelain surfaces were acid-etched with 9.6% hydrofluoric acid gel (Porcelain Etch Gel, Pulpdent Corporation, Watertown, MA, USA) for two minutes, rinsed with water for 20 seconds and dried with oil-free air spray for 20 seconds. A thin layer of silane agent (Silane Bond Enhancer, Pulpdent) was applied onto the porcelain surfaces of all the groups with a brush, retained for two minutes, then blown gently with oil-free air spray for one minute, when applicable. After the surface preparation, the specimens were randomly divided into six groups (n=10) for different adhesive systems. The adhesive systems used in this study are shown in Table 1.

For each of the six different adhesive systems, a thin layer of bonding agent was applied to the prepared porcelain surfaces with an applicator tip (Applicator Tips Longred, Heraeus Kulzer, Hanau, Germany) according to the manufacturer's suggestions. The

Table 1: *Adhesive Systems Used in This Study*

| Product | Material Type | Composition | Manufacturer | LOT # |
|---|---|---|---|---|
| Adper Prompt L-Pop | Self-etch adhesive | Liquid 1: Metacrylated phosphoric esters, Bisphenyl glysidyl methacrilate<br>Liquid 2: Water, 2-hydroxyethyl metacrylate, polyalkenoic acid | 3M ESPE AG Seefeld, Germany | 222274 |
| Quadrant Uni 1 Bond | 1-bottle light-curing adhesive | Ethanol (56%), 2-hydroxyethyl metacrylate (10-25%), Poly (methacrylic-oligo-acrylic acid) (5-10%), 4-methacryloxyethyl trimellitic anhydride (0-5%) | Cavex Holland, Haarlem, The Netherlands | 010068 |
| Te-Econom | Bonding agent | Phosphonic acid acrylate (<11%), hydroxyethylmethacrylate (<20%), dimethacrylates (<53%), alcohol (<20%) | Ivoclar Vivadent Polska | G21880 |
| One-Step Plus | Filled universal dental adhesive | Bisphenyldimethacrylate (15-40%), hydroxyethyl metacrylate (15-40%), acetone (40-70%), dental glass (1-10%) | BISCO Inc Schaumburg, IL, USA | 0500011052 |
| Prime & Bond NT | Bonding agent | Urethanedimethacrylate resin (<20%), dipentaerythritolpentaacrylatephosphate (<10%), polimerizable dimethacrylate resin (<10%), polimerizable trimethacrylate resin (<10%), acetone (<70%), nanofillers-amorphous silicon dioxide (1-10%) | Dentsply Caulk Milford, DE, USA | 0509002448 |
| PQ 1 | Highest filled single component adhesive | Metacrylic acid (6%), 2-hydroxyethyl metacrylate (10.2%), ethanol (8.5%), filler (40%) | Ultradent Products Inc South Jordan, UT, USA | K087 |

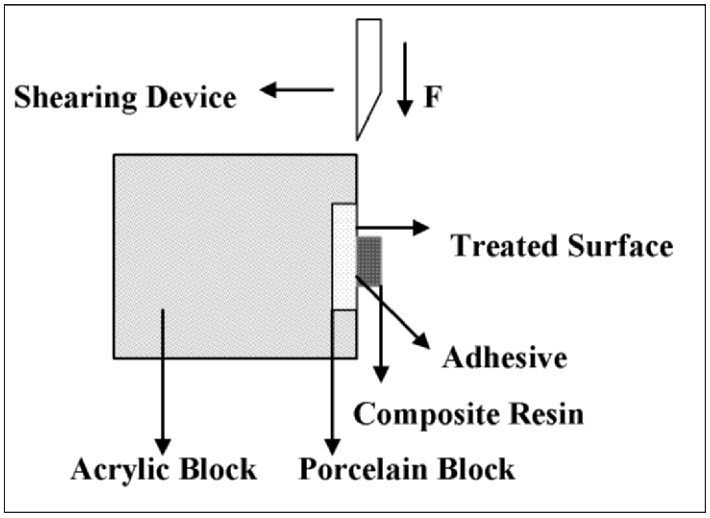


Figure 1. *Schematic drawing of tested specimens in testing apparatus.*

adhesives were light-cured with a Hilux Ledmax 550 light source (Benlioglu Dental, Ankara, Turkey). A 3-mm thick Teflon mold containing a 5 mm diameter cylindrical opening was placed onto the surface-treated specimens, and a universal resin composite (Filtek Z250, 3M ESPE, Seefeld, Germany) was condensed in the opening of the Teflon mold and light-polymerized for 20 seconds according to the manufacturer's suggestions.

Before mechanical testing, the prepared specimens were stored in distilled water at 37°C for 24 hours, then all the samples were thermal cycled 1000 times between 5°C and 55°C. The dwell time was 30 seconds in each bath, with a transfer time of five seconds. Shear testing of all groups was performed on a universal test machine (Lloyd LRX, Lloyd Instruments, Fareham, Hampshire, England) using a crosshead speed of 0.5 mm/minute (Figure 1). The shear debond forces were recorded in Newton and converted into MPa. A two-way analysis of variance (ANOVA) was performed on the bond strength data using statistical software (SPSS for Windows, Version 12.0.1, SPSS, Chicago, IL, USA). Then, the means were compared using the Tukey HSD test ($\alpha$=0.05).

The porcelain sides of the fractured interfaces were examined with a stereomicroscope (Nikon SMZ 1500, Nikon, Tokyo, Japan) to determine the fracture mode that occurred during the shear bond strength testing. From that analysis, three types of failure were defined: adhesive failure was considered to occur at the porcelain/adhesive system interface; cohesive failure occurred in the composite material or porcelain, with no damage to the interface and mixed failure was defined as involving both the interface and the material.

## RESULTS

According to the ANOVA, although the adhesive systems were statistically significant ($p$<.0001), the porcelain materials and interaction (porcelain and adhesive system) were not statistically significant ($p$>0.05) (Table 2).

For all porcelain groups, the lowest bond strength was observed in Adper Prompt L-Pop. Although, Quadrant

Table 2: *Two-way ANOVA for Porcelains and Adhesive Systems*

| Variable (Source) | *df* | Sum of Squares | Mean Squares | F-value | Probability* |
|---|---|---|---|---|---|
| Porcelain | 1 | 0.014 | 0.014 | 0.028 | 0.866 |
| Adhesive System | 5 | 946.819 | 189.364 | 376.485 | 0.001 |
| Interaction | 5 | 0.415 | 0.083 | 0.165 | 0.975 |
| Error | 108 | 54.322 | 0.503 | | |

**Significantly different at p<.05.*

Table 3: *Mean Shear Bond Strengths (MPa), Standard Deviation Values for Study Groups and Differences Between Groups According to the Tukey HSD Test*

| Groups | Matchmaker | VMK 95 |
|---|---|---|
| Adper Prompt L-Pop | 2.97 (0.61) | 2.90 (0.54) |
| Quadrant Uni 1 Bond | 6.27 (0.68) | 6.36 (0.76) |
| Te-Econom | 8.44 (0.61) | 8.41 (0.59) |
| One-Step Plus | 9.61 (0.63) | 9.69 (0.72) |
| Prime & Bond NT | 9.78 (0.82) | 9.63 (0.83) |
| PQ 1 | 11.52 (0.86) | 11.72 (0.77) |

**Vertical and horizontal lines connect groups that are not significantly different at p<.05.*

Table 4: *Failure Modes of the Specimens After the Shear Bond Test*

| Groups | | Mode of Failure | | |
|---|---|---|---|---|
| | | Adhesive | Cohesive | Mixed |
| Adper Prompt L-Pop | Matchmaker | 10 (100%) | - | - |
| | VMK95 | 10 (100%) | - | - |
| Quadrant Uni 1 Bond | Matchmaker | 8 (80%) | - | 2 (20%) |
| | VMK95 | 7 (70%) | - | 3 (30%) |
| Te-Econom | Matchmaker | 1 (10%) | 3 (30%) | 6 (60%) |
| | VMK95 | 2 (20%) | 3 (30%) | 5 (50%) |
| One-Step Plus | Matchmaker | - | 5 (50%) | 5 (50%) |
| | VMK95 | - | 4 (40%) | 6 (60%) |
| Prime & Bond NT | Matchmaker | - | 6 (60%) | 4 (40%) |
| | VMK95 | - | 5 (50%) | 5 (50%) |
| PQ 1 | Matchmaker | - | 10 (100%) | - |
| | VMK95 | - | 10 (100%) | - |

Uni 1 Bond demonstrated a higher mean bond strength when compared to Adper Prompt L-Pop, these three adhesives had lower bond strengths when compared with all the other adhesives. Te-Econom showed a higher mean bond strength value than Quadrant Uni 1 Bond. No statistically significant difference was observed between One-Step Plus and Prime&Bond NT, and these groups demonstrated higher bond strength values when compared to the above mentioned groups. The highest bond strength in this study was observed with PQ 1. The differences between groups are listed in Table 3.

Table 4 gives the mode of failure observed in each study group. It is clear from this data that the mode of failure was predominantly cohesive within the porcelain itself for the PQ1 group in both porcelain types. For low-fusing porcelain in the Prime&Bond NT adhesive group, six cohesive failures within porcelain and four mixed failures were observed. Five cohesive failures within porcelain and five mixed failures were observed in the One Step Plus group. In the Adper Prompt L-Pop group, adhesive failure was determined in all specimens. For feldspathic porcelain, five cohesive failures within porcelain and five mixed failures were observed in the Prime&Bond NT adhesive group. In the One Step Plus group, four cohesive failures within porcelain and six mixed failures were observed. In the Adper Prompt L-Pop group, adhesive failure was observed in all specimens.

## DISCUSSION

Various methods have been introduced to repair fractured porcelain with resin composite. In the last decade, several trends have developed in the formulation and marketing of current bonding agents in an attempt to simplify their application and reduce the amount of time required for the procedure.[16] However, the effects of different adhesive systems on bond strength between resin composite and porcelain have not been completely clarified. In this study, the optimal shear bond strength of composite bonded to low-fusing and feldspathic porcelains was evaluated for six different adhesive systems.

Many methods of measuring the *in vitro* bond strength afforded by porcelain repair systems have been described. These include torsion,[17] flexural,[18] tensile[19] and shear bond strength tests.[5,9-10,19-20] One of the most commonly employed test is the shear bond strength test.[19] In the current study, the shear bond test was used to evaluate the effect of different adhesive systems on the bond strength of resin composite to porcelains.

The type of resin composite also affects its bond strength to porcelain. It was assumed that large particle size composites or hybrid-type resins at the porcelain interface resulted in higher bond strengths than small-sized composites.[21-22] Hybrid-type universal resin composite (Filtek Z250) was preferred in the current study.

Güler and others[9] evaluated the 24-hour shear bond strength of resin composite to porcelain treated with sandblasting with 50 µm or 110 µm $Al_2O_3$ etching with hydrofluoric acid, applying silane agents and combinations of these treatments. They reported that a higher bond strength was achieved with a combination of 50 µm $Al_2O_3$ air abrasion, 9.6% hydrofluoric acid and silane agent compared to treatment with any of these procedures alone. For this reason, porcelain specimens were treated with 50 µm $Al_2O_3$ air abrasion, 9.6% hydrofluoric acid and silane agent.

Güler and others[10] evaluated the effect of acid etching times with 9.6% hydrofluoric acid gel and self-etching adhesive on the shear bond strength of resin composite to porcelain. According to the results of this study, the highest bond strength value was observed in the group that was acid etched for two minutes. In the current study, the specimens were etched for two minutes. However, in the clinical applications, the hazards of hydrofluoric acid were well recognized, so that care had to be taken during application.

According to the study by Güler and others,[10] Single Bond groups exhibited higher bond strength values than did the self-etching adhesive in porcelain repair. Parallel to this study, the lowest bond strength was observed in the self-etching adhesive system (Adper Prompt L-Pop) for two porcelains tested in the current study. These may be due to the primer in the self-etching adhesive system damaging the microporosity treated by hydrofluoric acid.

Thicker adhesive layers or liners may act as an elastic intermediate layer between the cavity walls and the adjacent composite. That is, they could resist the polymerization shrinkage stress of the resin composite[23] and absorb the shock produced by occlusal loads and thermal cycling.[24] Using unfilled adhesives, thicker layers are not recommended, because these materials have lower mechanical properties and usually provide no radiopacity, which could mislead clinicians to interpret the adhesive radiotransparency as gap formation or recurrent caries at the margin of the restoration.[25] Based on this idea, filled adhesives have been introduced,[26-28] which have included various types of fillers, such as conventional glass, ion leachable glass, silica and nanometer-sized aerosol silica fillers.[29-31]

Filled adhesives were expected to act as an intermediate shock-absorbing elastic layer between resin composite and dentin, thus increasing the bond strength to dentin.[23-24,32] Many studies evaluated comparisons between commercially available filled and unfilled adhesives; however, the advantages of these adhesives as stress buffers remain unpredictable[26] and have not been substantiated with *in vitro* bond tests[33-35] and a clinical trial.[36] According to the results of the current study, for both porcelain materials, Quadrant Uni 1 Bond demonstrated higher bond strength when compared with Adper Prompt L-Pop; this group had lower bond strength when compared with all the other groups. Te-Econom showed higher bond strength values than Quadrant Uni 1 Bond. No statistically significant difference was observed between One-Step Plus and Prime&Bond NT, and these groups demonstrated higher bond strength values when compared to the above mentioned groups. The highest bond strength in this study was observed in PQ 1. These findings showed that the unfilled adhesives (Quadrant Uni 1, Te-Econom) exhibited lower bond strength values than the filled adhesives (PQ1, One-Step Plus, Prime&Bond NT).

In addition, filler type, size, shape, its surface characteristics, their interaction with the resin matrix and various solvents in the adhesives may affect bond strength.[37-39] In the current study, PQ1, which has a 40% filler ratio, demonstrated higher bond strength than One-Step Plus and Prime&Bond NT, both which have a 1%-10% filler ratio.

In the current study, for the PQ1 group, the mode of failure was predominantly cohesive within the porcelain itself, which has a 40% filler ratio. In the Prime&Bond NT group (1%-10% filler), six cohesive failures within the porcelain itself and four mixed failures for the low fusing porcelain, five cohesive failures within the porcelain group itself and mixed failures for feldspathic porcelain were observed. Five cohesive failures within the porcelain group and five mixed failures were observed in the One Step Plus group (1%-10% filler) for low-fusing porcelain. Four cohesive failures within the porcelain itself, and six mixed failures were observed in the One Step Plus group for the feldspathic porcelain group. Eight adhesive and two mixed failures were detected in the Quadrant Uni One Bond group, which does not have filler for low-fusing porcelain. In the Adper Prompt L-Pop group, adhesive failure was determined in all the specimens. This may be due to the stress distribution between the filled and unfilled adhesive that had been expected to be different, since adhesives containing fillers showed higher elastic modules than unfilled resins.[35]

The current study is limited to two porcelain products and six adhesive systems. Nevertheless, these findings allow for a better understanding of the effects of different adhesive systems on the shear bond strength of resin composite to low-fusing and feldspathic porcelains. However, in future studies, the effect of these adhesive systems on the bond strength of composites to different porcelain materials should be done. The effects of filler ratio, type and size of adhesive systems on porcelain repair should also be evaluated.

## CONCLUSIONS

Within the limitations of the current study, the following recommendations can be made: when low-fusing or feldspathic porcelain restorations are repaired with resin composite, self-etching adhesive systems may not be indicated. If maximum bond strength is the goal in porcelain resin bonding, adhesive systems with a high filler ratio should be used.

*(Received 13 June 2008)*

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