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Laboratory Study of Micro-shear Bond Strength of Two Resin Cements to Leucite Ceramics using Different Ceramic Primers

[1]Abdullah A Al-Harthi, [2]Mohammed H Aljoudi, [3]Mulham N Almaliki, [4]Khalid A El-Banna

ABSTRACT

Aim: To evaluate the effect of Monobond Etch & Prime on the micro-shear bond strength (MSBS) of resin cements to leucite surface and to compare the MSBS of two different resin cements to conditioned leucite surfaces with different primer systems.

Materials and methods: Twenty-one leucite ceramic disks (10 mm diameter, 2 mm thickness) were divided into three groups (n = 7). Group I: 9.6% hydrofluoric (HF) acid and Monobond S (Ivoclar Vivadent, Liechtenstein), then conventional resin cement was applied. Group II: Monobond Etch & Prime (Ivoclar Vivadent, Liechtenstein), then conventional resin cement was applied. Group III: 9.6% HF acid and with Monobond N (Ivoclar Vivadent, Liechtenstein), then adhesive resin cement was applied. The assigned resin cement was applied in each disk through five plastic tubes with an inner diameter of 1.6 and 1.9 mm height, and then light cured. Micro-shear bond strength was determined by pulling out the resin cement using universal testing machine (Instron®, USA).

Results: One-way analysis of variance (ANOVA) and Student's t-test were used to determine statistical difference (α = 0.05) between each two groups. The results showed that group III had the highest MSBS values (7.32 ± 2.47) followed by group II (6.24 ± 2.16), whereas group I had the lowest MSBS values (5.7 ± 2.7). Nevertheless, there was no statistically significant difference between the results of all the groups.

Conclusion: Monobond Etch & Prime has shown comparable results to the most popular combination of HF acid and silane. The combination of HF acid and Monobond N and self-adhesive resin cement has shown the best MSBS results, though not statistically significant.

Clinical significance: The clinicians can use simplified ceramic primer technique (Monobond Etch & Prime) which has comparable MSBS to the most popular combination of HF acid and silane.

Keywords: Bond strength, Ceramic primer, Micro-shear, Resin cement, Self-etch primer, Silane.

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[1,2]Umm Al-Qura University, Makkah, Kingdom of Saudi Arabia

[3]Security Forces Hospital, Makkah, Kingdom of Saudi Arabia

[4]Department of Fixed Prosthodontics, Umm Al-Qura University Makkah, Kingdom of Saudi Arabia

Corresponding Author: Abdullah A Al-Harthi, Umm Al-Qura University, Makkah, Kingdom of Saudi Arabia, Phone: +966595066644, e-mail: Abdullah.alharthi1234@gmail.com

INTRODUCTION

With the growing demand for esthetic treatments, there has been development and improvement in the composition and of ceramic and cementing systems in order to improve mechanical strength of the ceramic material, enhance bonding capacity to both the tooth and the restoration, and boost esthetics.[1,2] Due to these improvements, the indications of all-ceramic restorations have increased to include veneers, inlays, onlays, full coverage crowns, and fixed partial dentures.

Cementation is a crucial step to ensure the retention, marginal seal, and durability of indirect restorations.[3] The cementation gives retention and seal of the space between the tooth and restoration.[4] Resin cements have significantly higher shear bond strength to ceramic restoration when compared with zinc-phosphate cements.[5] Resin cements have high bond strengths to both tooth structure and porcelain.[6] Dual-cured resin cements are preferred because they have extended working time.[7]

Total-etch adhesives combined with conventional resin cements are considered the best luting agents with glass ceramic.[8]

Conventional and self-adhesive resin cements are appropriate for cementation of all-ceramic restorations.

The use of both cements in the same clinical case has been shown to be adequate to achieve satisfactory esthetic and functional results after a 3-year follow-up.[9]

The development of new restorative materials has been based on the concept of microretention (rather than macroretention), which allows better conservation of the dental structure, provided appropriate adhesive procedures are used.[10]

Hydrofluoric acid when used for etching fitting ceramic surface will provide proper texture by dissolving the glass matrix to expose the crystalline phase;[11] 9.6% HF etching followed by silane is the most effective surface treatment.[12]

However, there are some health hazards known to be associated with HF.[13] In addition, the multistep application technique is time-consuming and technique-sensitive, and consequently may compromise bonding effectiveness.[14] Moreover, insoluble silica fluoride salts which are by-products from the etching process may weaken the bond strength of the cement.[15]

Monobond Etch & Prime (Ivoclar Vivadent, Liechtenstein) is recently introduced in the market as a one-step primer replacing the two-step etching and silanation. It is based on ammonium polyflouride and silane methacrylate. Currently, the data on this new ceramic primer are limited, so the comparison between this new primer and the gold standard for surface treatment of ceramic is deemed necessary.

In this study, bonding to leucite was investigated using two different resin cements and three different primer systems.

MATERIALS AND METHODS

Materials used in this study are shown in Table 1.

Sample Classification

A total of 21 disks were made and randomly divided into three equal groups (n = 7) according to surface treatment and cement used.

Wax Pattern Preparation

Twenty-one wax patterns were made from dipping wax (BEGO, Germany), as shown in Figure 1. For standardization of wax dimensions, a special metal ring was customized with 10 mm diameter and 2 mm height, as shown in Figure 1. Then, wax was poured in the ring until the ring was filled completely with the wax.

Spruing and Investing Procedure

A 10 mm length of sprue wax (BEGO, Germany) with 2.5 mm thickness was attached to the side of each wax pattern. Phosphate-bounded investment materials (VITA PM, Germany) were used to invest six wax patterns with their sprue in one investment ring according to the manufacturer's instructions.

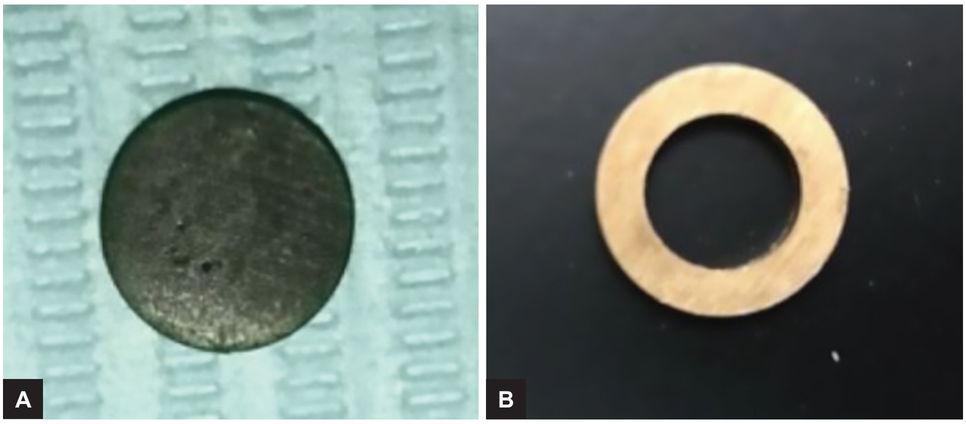
Wax Elimination

After the investment materials set, the ring was placed in Midtherm furnace (BEGO, Germany) according to the manufacturer's instructions to eliminate the wax from the ring.

Table 1: Materials used in this study

Material	*Category*	*Composition*
PM9 (VITA, Germany)	Leucite ceramic	Based on the proven fine-structure ceramic VITA VM 9 and is used for pressing to yttrium-stabilized ZrO_2 frameworks in the CTE range of 10.4–10.6 x 10^{-6} such as VITA In-Ceram YZ and other zirconium oxide substructure materials for natural looking high strength
Pulpdent, USA	Ceramic etchant	9.6% hydrofluoric acid
Monobond S (Ivoclar Vivadent, Liechtenstein)	(Silane) ceramic primer	Alcohol solution of silane methacrylate.
Monobond etch and prime (Ivoclar Vivadent, Liechtenstein)	Self-etching ceramic primer.	Ammonium polyfluride, silane system based on trimethoxypropyl methacrylate, solvents (alcohols and water) and food colorant (fast green).
Monobond N (Ivoclar Vivadent, Liechtenstein)	Universal primer	Alcohol solution of silane methacrylate, phosphoric acid methacrylate, and sulfide methacrylate
Tetric® N-Bond (Ivoclar Vivadent, Liechtenstein)	Total etch adhesive	Contains phosphoric acid acrylate, HEMA, bis-GMA, urethane dimethacrylate, ethanol, film-forming agent, catalysts, and stabilizers
Variolink® N (Ivoclar Vivadent, Liechtenstein)	Conventional resin cement	The monomer is composed of bis-GMA, urethane dimethacrylate, and triethylene glycol dimethacrylate. The inorganic fillers are barium glass, ytterbium trifluoride, Ba-Al-fluorosilicate glass, and spheroid mixed oxide. Additional contents: Initiators, stabilizers, and pigments. The particle size is 0.04–3.0 μm. The mean particle size is 0.7 μm
seT (SDI, Australia)	Self-adhesive resin cement	

HEMA: 2-Hydroxylethyl methacrylate; MA: Glycidyl methacrylate



Figs 1A and B: Wax pattern and customized metal ring with 10 mm diameter

Fig. 2: Disks were removed from the ring and cut the sprue

Heat Pressing

After the wax elimination, the ring was placed in Vacumat 6000 M (VITA, Germany). Two heat-pressed leucite ceramic ingots (VITA PM9, Germany) were pressed in each ring according to the manufacturer's instructions.

Disk Preparation

The disks were removed from the ring and the sprue was cut as in Figure 2. Then, 110 T sandblasting of the surfaces was made using Protempomatic (BEGO, Germany). The samples were divided into three equal groups (n = 7).[5]

Bonding Procedures

Group I: The bonding surfaces were etched with 9.6% HF acid (Pulpdent, USA) for 20 seconds, then rinsed, and dried for 10 seconds, then coated with Monobond S (Ivoclar Vivadent, Liechtenstein) using a microbrush and left to react for 60 seconds, and then dried. Then, Tetric® N-Bond (Ivoclar Vivadent, Liechtenstein) was applied by microbrush and dried for 10 seconds, and then light cured for 20 seconds. Then, conventional resin cement Variolink® N (Ivoclar Vivadent, Liechtenstein) was mixed and applied according to the manufacturer's instructions (Table 2).

Table 2: Groups tested according to surface treatment

Group	*Surface treatment and cement*
I	Hydrofluoric acid + silane ceramic primer + universal adhesive + conventional resin cement
II	Self-etching ceramic primer + universal adhesive + conventional resin cement
III	Hydrofluoric acid + universal primer + self-adhesive resin cement

Group II: Monobond Etch & Prime (Ivoclar Vivadent, Liechtenstein) was applied on the bonding surfaces and agitated by microbrush for 20 seconds and left to react for another 40 seconds, then rinsed and dried for 10 seconds. Then, Tetric® N-Bond (Ivoclar Vivadent, Liechtenstein) was applied by microbrush and dried for 10 seconds, and then light cured for 20 seconds. Then, conventional resin cement Variolink® N (Ivoclar Vivadent, Liechtenstein) was mixed and applied according to the manufacturer's instructions.

Group III: The bonding surfaces were etched with 9.6% HF acid (Pulpdent, USA) for 20 seconds, rinsed and dried for 10 seconds, then coated with Monobond N (Ivoclar Vivadent, Liechtenstein) by microbrush and left to react for 60 seconds, and then dried. Then self-adhesive resin cement seT (SDI, Australia) was mixed and applied according to the manufacturer's instructions.

For standardization of bonding surface area, plastic tubing with an inner diameter of 1.6 and 1.9 mm height was used to place the resin cements on the ceramic bonding surfaces. Five tubes were distributed on the bonding surface of each disk as shown in Figure 3. The assigned resin cement was mixed and applied through the tubes to the ceramic bonding area, and then light cured according to the manufacturer's instructions, as shown in Figure 4.[16]

Micro-shear Bond Strength Testing

The MSBS was determined by pulling out the resin cement using universal testing machine 5940 single

Fig. 3: Plastic tubing with an inner diameter of 1.6 mm and height of 1.9 mm

Fig. 4: The assigned resin cement was mixed and applied through the tubes to the ceramic

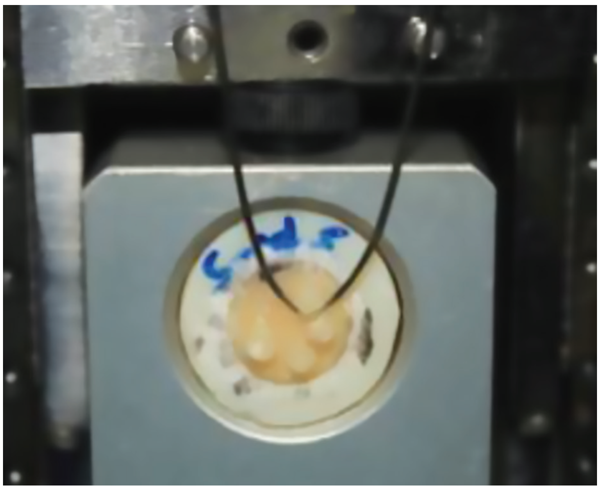
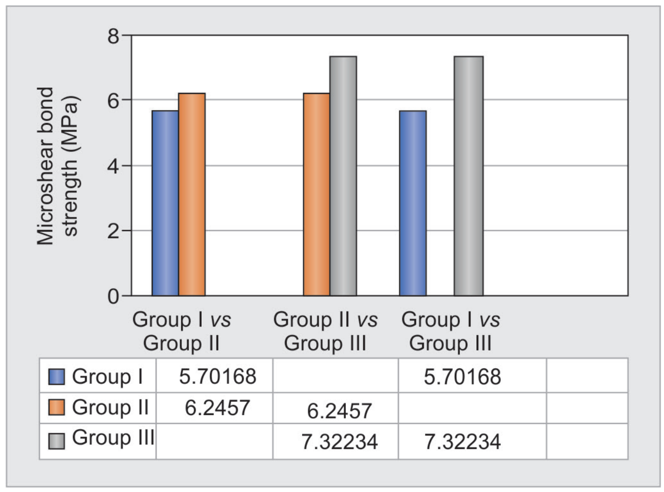


Fig. 5: Pulling out the resin cement using universal testing machine (Instron®, USA)



Graph 1: Bar chart showing MSBS results

column (Instron®, USA) at a crosshead speed 0.5 mm/min, and shear force was applied through a stainless steel orthodontic wire 0.7 mm in diameter, which was positioned as close as possible to ceramic–resin interface as shown in Figure 5.[16] The obtained load values (N) were converted into the megapascals by dividing the failure load (N) by the bonding area (mm^2).[17]

Table 3: Means, standard deviations, and significance among three groups

	Groups I vs II	*Groups I vs III*	*Groups II vs III*
Mean (MPa)	5.70168	5.70168	6.24570
	6.24570	7.32234	7.32234
Standard deviation	2.705190	2.705190	2.168692
	2.168692	2.477314	2.477314
Significance	0.382	0.950	0.274

RESULTS

Data were analyzed using Kolmogorov–Smirnov test to assess normal distribution. One-way ANOVA and Student's t-test were used to determine statistical difference ($\alpha = 0.05$) between each two groups, as shown in Graph 1 and Table 3.

- The results showed that group III had the highest MSBS values (7.32 ± 2.47) followed by group II (6.24 ± 2.16), whereas group I had the lowest MSBS values (5.7 ± 2.7). Nevertheless, there was no statistically significant difference between the results of all the groups.
- There was no significant difference in MSBS when HF acid and silane were compared with Monobond Etch & Prime.
- No significant differences in MSBS were found when Monobond Etch & Prime and conventional resin cement were compared with HF acid and Monobond N and self-adhesive resin cement.

- No significant differences in MSBS were found when HF acid and silane were compared with HF acid and Monobond N and self-adhesive resin cement.

DISCUSSION

When a ceramic material is bonded to tooth, two different interfaces should be taken into consideration: Tooth/cement interface and ceramic/cement interface. The bond strength at both interfaces should be optimized because the weakest one will determine the final bond strength of the cemented restoration. If the adhesive seal in the above interfaces fails, it results in microleakage, jeopardizing the clinical performance and longevity of the restoration.

The strength and durability of the bond between ceramic and resin cement depends on multiple factors including the type of treatment selected, which is in turn governed by the microstructure of the ceramic material. Hence, for bonding to ceramic, mechanical union is achieved typically by sandblasting with aluminum oxide particles and conditioning with HF acid which exposes the crystals at the surface of the ceramic structure, creating areas of microretention. Silane promotes additional chemical bonding by facilitating the contact with the ceramic due to bi-functional molecules, besides providing bond between silica in the ceramic and the organic matrix of the resin cement by way of siloxane bonds.[17,18]

In this study, the previously described surface treatment technique was used in the first group as a control group. In the second group, another surface treatment was performed using Monobond Etch & Prime. Both groups were combined with conventional resin cement to evaluate if combining the two steps of etching and silanation into one step has affected bond strength to the glass ceramic.

In the third group, HF acid etching and Monobond N were used in combination with self-adhesive resin cement not only because the primer has silane methacrylates which enhances bonding to the ceramic surface but also because both the primer and the cement contain phosphoric methacrylates, which ensure better bonding between the two materials.

A total-etch adhesive (Tetric N Bond) was standardized across the groups as a bonding agent instead of universal adhesives which is the latest generation of dental adhesives. This comes from the fact that universal adhesives are primarily self-etching adhesives composed of functional and hydrophilic monomers which may contain silane.[19] Silane may be unstable when combined with 10-methacryloyloxydecyl dihydrogen phosphate (MDP) and bisphenol A-glycidyl methacrylate resins in a one-bottle solution.[20] Under acidic conditions, such as in the presence of MDP and water, a self-condensation reaction might occur between the silanol groups.[19] Therefore, the complex adhesive composition may negatively affect the reaction of silane to the leucite ceramic surface. In addition, the high viscosity of the adhesive solution may reduce the penetrative effects of the agent on the surface irregularities formed after HF acid etching.

One of the recommended resin cements for bonding glass ceramics is the conventional resin cement due to the prior application of an adhesive system, promoting the formation of a hybrid layer, enabling better bonding to the dentin substrate.

The MSBS was used in this study as shear stresses are believed to be major stresses involved in *in vivo* bonding failure of restorative materials.[21] Several authors have used this test to evaluate the bond strength of resin cements to all-ceramic materials.[15,22,23]

In this study, both hypotheses that neither the type of the resin cement nor the type of primer used in this study has significant effect on the MSBS to leucite ceramic have been proven.

There was no statistically significant difference between groups I and II. These results are in accordance with Roman-Rodríguez et al[24] and Alrahlah et al[25] who found statistically nonsignificant differences between the conventional HF-silane technique and the one-step Monobond Etch & Prime technique. This suggests effective microretention induced by the ammonium polyflouride on the surface of the glass ceramics.

The interesting fact was that despite rinsing of the primer, the trimethoxy propyl methacrylate seemed to have reacted well with the ceramic surface and was not washed away by the water rinsing. It was not affected by the by-products of the etching process as well.

There was no statistically significant difference between Monobond N/Self-adhesive resin cement (group III) and the adhesive resin cement/HF/silane and the adhesive resin cement/Monobond Etch & Prime (groups I and II). This was in accordance with Rigolin et al[26] who found no statistical difference between self-adhesive and conventional dual curing resin cements.

However, several studies found that dual polymerized conventional resin cements had better bonding efficacy of leucite ceramic than the self-adhesive resin cements. However, in these studies, the bonding of the two types of cement was tested at both ceramic and dentin interfaces with cement, and the failure for the self-adhesive was mostly adhesive at the cement/dentin interface. In this study, we only tested the ceramic/cement interface, so this may be the cause for different results.[12,27,28]

In the light of this study, it seems that the one-step ceramic primer can be used successfully with the guarantee of excellent bond strength, comparable to the gold standard, and the potential of precluding the hazards of HF acid in addition to saving the time of the step omitted.

Furthermore, since both types of cements have comparable MSBS to leucite ceramic, we can recommend that conventional resin cements manipulating total-etch technique can be used for bonding of low retention restoration as ceramic veneers or where retention of other all-ceramic restoration is compromised, whereas self-adhesive resin cements can be useful in cases where there is enough retention of the restoration (crowns, inlays, onlays). In such cases, this will eliminate the need of tooth etching and bonding, thus simplifying the procedure and reducing the working time.

Further research may be performed to test the effect of using the one-step ceramic primer with the self-adhesive resin cement.

CONCLUSION

Within the limitations of this study, we can conclude the following:

- The newly introduced Monobond Etch & Prime has shown comparable results to the most popular combination of HF acid and silane. This can be very promising, as the new material reduces the time required for preparing the restorations and prevent the dental staff from the hazards of HF acid.
- The combination of HF acid and Monobond N and self-adhesive resin cement has shown the best MSBS results, though not statistically significant.
- Further *in vitro* and *in vivo* studies are required to evaluate the different bonding systems used when cemented to dental substrates.

CLINICAL SIGNIFICANCE

The clinicians can use simplified ceramic primer technique (Monobond Etch & Prime) as a one-step primer replacing the two-step etching and silanation. It has comparable MSBS to the most popular combination of HF acid and silane.

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