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Research Article

Effect of Post Silanization Drying Methods and Duration on The Shear Bond Strength Between The Leucite-reinforced Glass Ceramic and Resin Cement

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Effect of Post Silanization Drying Methods and Duration on The Shear Bond Strength Between The Leucite-reinforced Glass Ceramic and Resin Cement

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Abstract

Statement of the problem: Studies are ongoing to enhance the bonding strength between glass ceramic restorations and resin cements. The effect of various drying methods on bonding strength is still controversial.

Objective: The aim of this study is to determine whether different drying methods and durations affect the bonding strength between leucite reinforced glass ceramics and resin cement.

Materials and Methods: Thirty different leucite reinforced glass ceramic (G-Ceram, Atlas Enta) specimens were divided into 5 groups. All specimens were treated with 5% hydrofluoric acid (HF) (IPS Ceramic Etching Gel, for 60 seconds according to the manufacturer's instructions. After acid etching, specimens were rinsed with water spray for the duration of acid etching and dried with pressurized air. Following the application of primer (Monobond N, Ivoclar Vivadent), specimens were divided into 5 groups based on air-drying methods and durations: Control (C) group (dried with pressurized air for 10 seconds; air drying in ceramic furnace for 30 seconds (CF30); air drying in ceramic furnace for 60 seconds (CF60); air drying with a hair dryer for 30 seconds (HD30); air drying with a hair dryer for 60 seconds (HD60). Ceramic specimens were bonded with dual cure resin cement (Multilink N; Ivoclar Vivadent) using a split Teflon mold with a diameter of 2.5 mm. Force was applied to the specimens with a 1 mm/min speed using a chisel-shaped applicator tip until failure occurred. The results were analyzed using one-way ANOVA followed by Tukey's post hoc test within the 95% confidence interval.

Results: There was a significant difference among the mean bond strength values of the groups ($P < 0.05$). Group CF60 and Group HD60 both exhibited significantly higher mean shear bond strength values compared with the group C ($P < 0.05$). Group CF30 and Group HD30 showed similar bonding values with CF60, HD60 and C groups ($P > 0.05$).

Conclusions: Drying the primer with ceramic furnace or the hair dryer for 60 seconds significantly enhanced the bonding strength between the leucite-reinforced glass ceramic and resin cement.

Keywords: Glass Ceramic, Shear Bond Strength, Silane Coupling Agent, Silanization, Heat Treatment, Primer, Resin Cement

Introduction

Full ceramic restorations stand out in the selection of indirect restorative materials due to their wear resistance, chemical stability, long-term durability, biocompatibility, and superior aesthetics.¹ Glass ceramics are non-metallic inorganic ceramic materials containing a glassy phase.² Adequate surface treatment is one of the key factors affecting the bonding performance and long-term clinical survival of ceramic restorations when bonded with resin cement.³ Various methods are used clinically to establish a reliable and durable bond between glass ceramics and resin cement.^{4,5}

In clinical commercial silanes, there is mostly 3-methacryloxypropyltrimethoxysilane (MPS) as the reactive key component diluted in ethanol and water.³ Silane molecules are chemically bonded to methacrylate-based materials (cement or bonding) via the organofunctional group (methacrylate) and to glass ceramics via the silanol group resulting from silane activation.⁶⁻⁸

During cementation of ceramic restorations, there are two interfaces: tooth-cement and cement-restoration. Cementation success depends on the success of processes at these two interfaces.⁹ Reliable cementation increases retention, improves marginal adaptation, minimizes microleakage, and enhances fracture resistance.¹⁰ Various methods involving mechanical and chemical modification have been proposed to achieve optimum resin-ceramic bond strength.⁷ For silica-based ceramic restorations, a reliable bond between resin cement and ceramic materials can be achieved through silane priming (chemical bonding) combined with hydrofluoric acid etching (mechanical bonding). Clinically, an effective air-drying step is required after the application of silane primer to ceramic restorations to distribute excess primer solution, evaporate ethanol, water-like by-products, and complete the condensation reaction between ceramic and silane coupling agent.¹¹⁻¹³ If a homogeneous silane layer cannot be formed on the ceramic surface, the resin-ceramic bond strength may deteriorate, adversely affecting the clinical performance of glass ceramic restorations. The presence of residual solvents can affect monomer polymerization, hinder the integrity of the bonded hybrid layer, create unwanted gaps at the interface, and reduce bond strength.^{3,7} Several methods for silane drying heat treatments have been proposed. The most common ones include drying with hot air, drying with a hairdryer, rinsing with hot water, preheated furnace or hot air furnace, and combinations of these methods.¹⁴⁻¹⁶ Recommended heating temperatures vary between 38°C and 120°C in studies.^{17,18}

There are different opinions on silane drying time. Different ingredients and types of silane coupling agents have different manufacturer's instructions. Silanes dried under different conditions and at different temperatures have been evaluated in many studies.^{14,19,20} However, to the authors' knowledge, there is no study in the literature that compare both ceramic furnace and hair dryer for different drying durations.

Therefore, the aim of this *in vitro* study, was to investigate the effect of different drying methods and durations following silanization on the shear bond strength between resin cement and leucite reinforced ceramics. The null hypothesis of this study was that the different silane primer drying methods and durations would not improve the shear bonding strength between the leucite-reinforced glass ceramic and the resin cement.

Materials & Methods

The materials used in this study have been shown in Table 1. The leucite containing glass ceramic specimens (G Ceram, Atlas Enta, Gülsa, Turkey) were cut to a thickness of 1.5 mm using a low-speed diamond saw (Isomet, Buehler Ltd., Lake Bluff, IL, USA) and sanded under water by the same researcher using 600, 800, 1000 and 1200 grit silicon carbide abrasive papers, in this order. The final thickness of the specimens was measured as 1.5 ± 0.01 mm by using a digital caliper. Before the bonding process, all the specimens were cleaned in an ultrasonic bath with distilled water. All bonding processes were performed at room temperature (23°C). Before priming the ceramic specimens, 4.5% HF (IPS Ceramic Etching Gel, Ivoclar Vivadent AG, Schaan, Liechtenstein) acid was applied for 60 s according to the manufacturer's instructions. The specimens were rinsed with water spray for the duration of the etching time and dried with pressurized air. The specimens were divided into 5 experimental groups (n=6) (Figure 1).

Control Group (C): After etching, primer was applied with a plastic brush according to the manufacturer's instructions. The primer was left to react for 60 second and then air-dried with a dental unit air spray for 10 seconds.

Ceramic Furnace 30 sec (CF30): After the etching process and primer application with a plastic brush, the drying process was carried out in a dental furnace (Programat 310, Ivoclar Vivadent, Liechtenstein) with a lowest temperature setting of 100°C for 30 seconds.

Ceramic Furnace 60 sec (CF60): After the etching process and primer application with a plastic brush, the drying process was carried out in a dental furnace for 60 seconds at 100°C.

Hair Dryer 30 sec (HD30): After the etching process and primer application with a plastic brush, the drying process was carried out with a hair dryer ($60^{\circ}\text{C} \pm 5$) from a distance of 15 cm for 30 seconds.

Hair Dryer 60 sec (HD60): After the etching process and primer application with a plastic brush, the drying process was carried out with a hair dryer (Sinbo, People Republic of China) ($60^{\circ}\text{C} \pm 5$) from a distance of 15 cm for 60 seconds.

After applying different silane drying treatments to the ceramic specimens, all the specimens were bonded with a light-curable self-cure resin cement (Multilink N, Ivoclar Vivadent, AG, Schaan, Liechtenstein) using a split Teflon apparatus with a diameter of 2.5 mm and a height of 3 mm. The manufacturer's instructions for curing were to light cure (Woodpecker iLED, Guilin Woodpecker Medical Instrument Co., Ltd., Guilin, Guangxi, China) for 20 seconds for each region. The specimens in this study were light cured for a total of 40 seconds. The polymerization light was held at a distance of max. 10 mm. It has a working time of approximately 3 minutes and a curing time of approximately 5 minutes. After the bonding process, all the specimens were kept in distilled water for 24 h before the shear bond strength testing.

For the shear bond strength test, the chisel-shaped application tip was placed parallel to the resin bonding interface, and force was applied to the specimens at a loading rate of 1 mm/min until failure occurred using an universal testing machine (Marestek, Mares Eng., İstanbul, Turkey). After the bonding test, the specimens were evaluated with a stereomicroscope with x10 magnification. The failure types were classified as adhesive (no resin cement remnants over the ceramic specimen), cohesive (the complete coverage of the bonding area by the resin cement), and mixed (combination of both adhesive and cohesive failures).

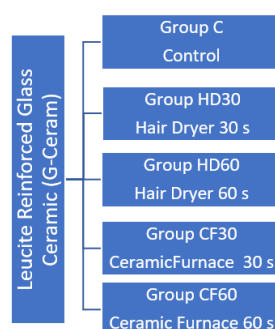


Figure 1. Experimental groups.

Table 1. The materials used in the study, their contents and manufacturers.

Material	Type	Chemical Composition	Manufacturer
N Multilink	Light-curable Self-cure resin cement	Monomer matrix: dimethacrylate and HEMA, Inorganic fillers: barium glass, yttrium trifluoride, spheroid mixed oxide	Ivoclar Vivadent, Schaan, Liechtenstein
bond N Mono-	Ceramic primer coupling agent	Alcohol solution of silane methacrylate, phosphoric acid methacrylate and sulphide methacrylate	Ivoclar Vivadent, Schaan, Liechtenstein
IPS Ceramic Etching Gel	4.5% hydrofluoric acid	<5% hydrofluoric acid	Ivoclar Vivadent, Schaan, Liechtenstein
G-Ceram	Leucite-reinforced glass ceramic	SiO ₂ 56-58%, Al ₂ O ₃ 18-25%, Na ₂ O 8-12%, K ₂ O 8-14%, CaO 0,2-1%, TiO ₂ 0,1-0,2%	Atlas-Enta Dental A.Ş., İzmir, Turkey

Statistical Analysis were performed using SPSS software (Version 26; IBM Corp., Armonk, NY, USA). One-way analysis of variance (ANOVA) was used to analyze the effects of primer drying methods. Post hoc Tukey's HSD test was applied to determine pairwise differences between groups. The confidence interval for all statistical analyses was set to 95%.

Results

A significant difference was found between the mean bond strength values of the groups according to the one-way ANOVA test ($F=5.848$, $df=4$, $P=.002$). Table 2 presenting the mean and standard deviations of the shear bond strength values of the test groups. Figure 2 shows the mean shear bond strength values and 95% confidence intervals of the test groups. The group CF60 and group HD60 both exhibited significantly higher mean shear bond strength values compared with the group C ($P<0.05$). Group CF30 and Group HD30 showed similar bonding values with the groups CF60 and HD60 ($P>0.05$). The control group exhibited the lowest bond strength value; however, there was no significant difference among the groups C, HD30 and CF30 ($P>0.05$).

Table 2. Mean shear bond strength values of the experimental groups (MPa).

Group	Mean Shear Bond Strength \pm Standard Deviations	Min.	Max.
Control	12.217 \pm 3.656 ^A	6.88	17.18
Hair Dryer 30 seconds	16.255 \pm 3.490 ^{AB}	10.22	20.04
Ceramic Furnace 30 seconds	17.190 \pm 4.992 ^{AB}	10.07	22.92
Hair Dryer 60 seconds	19.286 \pm 2.390 ^B	16.23	22.15
Ceramic Furnace 60 seconds	23.017 \pm 4.978 ^B	15.26	28.86

*Different superscript letters indicate a significant difference between the groups according to Tukey's HSD test (P<.05).

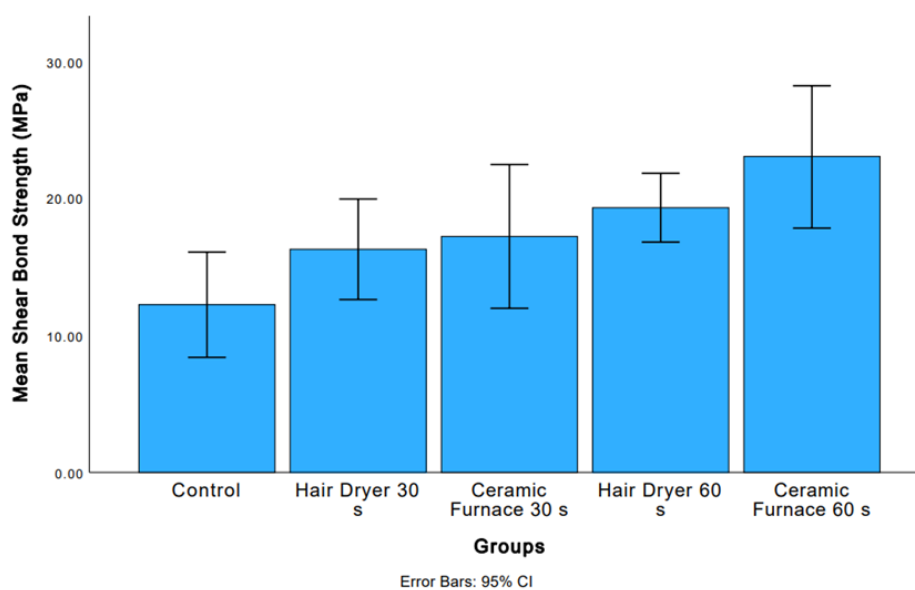


Figure 2. Mean shear bond strength (MPa) and the 95% confidence intervals of the test groups.

The failure types of ratios of the specimens were 50% cohesive failure, 25% mixed failure and 25% adhesive failure were observed in the control group, while 83% cohesive failure and 16% mixed failure were observed in the DF60 Group. The failure types of all groups are shown in Figure 3.

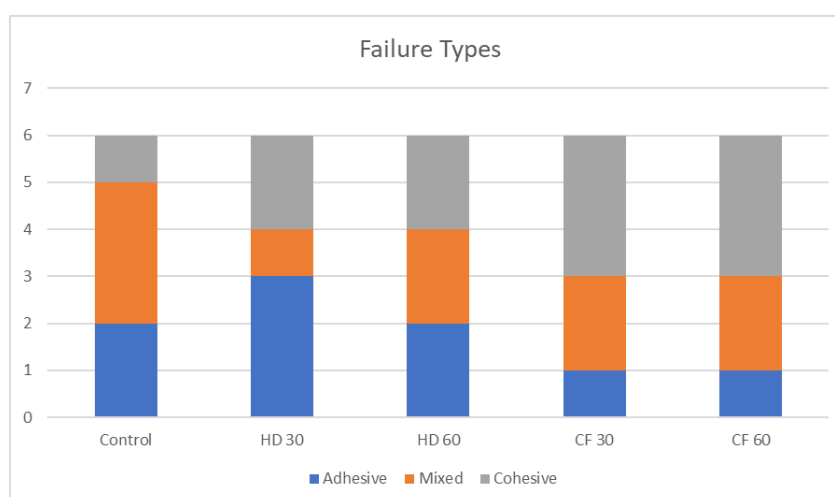


Figure 3. The failure types ratios of the specimens.

Discussion

The effect of primer drying methods and durations on the shear bond strength between the leucite-reinforced glass ceramic and resin cement was investigated in this study. Based on the findings, significant differences were found among the test groups. Therefore, the null hypothesis of this study was rejected.

In previous studies, the bonding of silane and bonded ceramic restorations or ceramic posts with dentin, resin cement, composite resin after heat drying has been extensively investigated.^{16,21} The primary factor influencing the extent to which resin cement strengthens the bonding of glass-ceramics is the adhesion of silane to the ceramic surface.²² Various surface treatments can be applied to glass ceramics, including hydrofluoric acid etching, silica coating, aluminum oxide particle abrasion, and laser irradiation.^{23,24} Particle abrasion can increase surface roughness and wettability of ceramic surfaces; however, it can also create microcracks in the structure, leading to new fractures in the long term.²⁵ Furthermore, due to the adverse working conditions of alumina particles, the intraoral method is limited to repair treatments. Additionally, hydrofluoric acid etching of silica-based ceramics has been reported to produce insoluble silica-fluoride salts as residual products on the surface. If not removed, these by-products can interfere with resin cement bonding.²⁶ The chemical bond between ceramic and composite resin cement is primarily achieved through silane coupling agents. Silane coupling agents respond to the increase in surface energy of ceramics and the wettability of cement by forming a siloxane bond, facilitating adhesion between the inorganic phase of ceramics and the organic phase of

the bonding agent applied to the ceramic surface, resulting in microscopic interactions between the two components.³

After drying silane, an interface with three different structures is usually observed instead of a monolayer silane. The outer layer consists of small oligomers that are adsorbed on glass and can be removed using an organic solvent or water at room temperature. The second layer consisted of similar oligomers that could be hydrolyzed with hot water. Cross-linking is more frequent and uniform in the region closest to the glass surface, forming a regular three-dimensional network that is more stable. This last layer is necessary to improve the bonding. Removing the outermost layer of the silane film can enhance bonding, leaving only a more stable layer chemically adsorbed to the ceramic surface.¹¹ In order to investigate the effect of air application at different times and temperatures on the removal of solvents and alcohols in this crosslink, both temperature and time were examined as variables in this study.

In a previous study evaluating the effect of heat treatment of different ceramic silanes on the bond strength of resin cement to glass ceramic, no significant difference in bond strength was observed between specimens dried in an furnace at 100°C for 1 minute and those left to dry at room temperature.²⁷ As a solution to this problem, it has been suggested that allowing liquid flow with a hot air gun or hair dryer can support alcohol and solvent evaporation. In this study, the significant difference between the HD 60 group and the control group, which were dried with a hair dryer for a sufficient time, supports this suggestion.

In a study investigating the effect of silane application and silane heat treatment on the micro-tensile bond strength between composite resin and lithium disilicate ceramic, air-dried and 100°C ceramic furnace-dried specimens were used. It was concluded that silane application and heat treatment showed a significant improvement in the bond strength of lithium disilicate ceramic bonded to the composite.²⁸ The results of this study showed that amongst the groups, the CF60 group had significantly higher shear bond strength values therefore supporting the conclusion drawn by Abduljabbar et al.²⁸

In another study examining the effect of silane activation temperature and activation method on the tensile bond strength between a veneer porcelain and composite resin, no significant difference was found between 38°C (control), 50°C 100°C hair dryer, and hot air drying, whereas the 120°C air drying group showed significantly higher bond strength. The researcher concluded that a definite direct correlation between drying temperature and bond strength could

not be established.¹⁷ In the present study, no significant difference was found between the control and 30 s treatment groups. Unlike the study by Yanakiev et al¹⁷, the difference between the HD 30 and HD 60 groups may have been due to insufficient time in the HD 30 group.

Silanization provides a layered configuration when applied to ceramic surfaces. It has been shown that thermal treatment at 100°C joins the layered surfaces, eliminates the intermediate phase, and increases the bonding strength of the resin cement and composite to ceramics.^{28,29} In this study, the groups that were placed in the ceramic furnace for 60 s and the 60 s hair-dried group had significantly higher mean bond strength values than the control group. The groups dried for 30 s (HD30 and CF30) were not significantly different from the other groups in this study. Although different drying methods were used, the shear bond values increased with the drying temperature. In addition, in groups with the same drying method, an increase in bonding values was observed as the drying time increased.

Studies by Yavuz and Eraslan³⁰ and Yavuz et al²⁴ found that silanization after typical hydrofluoric acid etching significantly increased the bonding strength between resin cement and glass ceramics. In a study by Cotes et al³¹, the bonding strengths between resin cement and feldspathic porcelain were compared for pre-hydrolyzed silane with thermal treatment and silane application after hydrofluoric acid treatment, concluding that thermal treatment of silane alone could not surpass silane application after hydrofluoric acid treatment.

Previous studies investigated various silane drying methods and durations. Fabianelli et al³² conducted a study examining the bonding strengths between leucite-reinforced glass ceramics subjected to different surface treatments and composite resins using a γ -MPTS-containing silane drying process at 100°C with hot air flow. It was observed that drying with hot air flow at 100 °C significantly increased the micro-tensile bond strength between the ceramics and composite resin. Monticelli et al²⁶ showed that drying at 38°C in warm air increased the bonding strength between ceramics and resin composites and enhanced the effectiveness of silane agents. In a study investigating the effect of different etching and silanization methods on the bond strength between a composite used in fracture repair and fluoroapatite and leucite glass-ceramics, hot air drying of the silanized ceramic at 45 °C was found to be more effective than air drying at room temperature in increasing the bond strength of the composite to the ceramic.¹²

De Carvalho et al¹⁴ investigated the effect of hot air and room temperature dried silane on the bond strength between resin cement and glass ceramic and found no significant difference between them. The ceramic furnace specimens were heated for 120 s, and the hair dryer specimens were heated for 60 s. In this study, a ceramic furnace and hair dryer were used as in the aforementioned study. Unlike deCarvalho et al¹⁴, the reason for the significant difference found in the present study may be the difference and insufficiency of the durations.

Silane was dried for different periods by different researchers. Ramón-Leonardo et al.¹⁹ evaluated the bond strength between lithium disilicate reinforced ceramics and dual cure resin cement. The researchers used a heat gun that was held for 15 s after Monobond N application, which had a positive effect on adhesion. In contrast, De Carvalho et al. used heat treatment for 2 min after primer application to a feldspathic ceramic (VITA VM7) and concluded that it did not contribute to bond strength. Similarly, Silva et al.²⁰ subjected IPS e.max CAD ceramic (Ivoclar Vivadent) to heat treatment (hot air jet) at 50°C for 20 s and found that heat treatment did not affect the results.

In a study investigating the bond strength between leucite-reinforced glass-ceramic and resin cement, silane was dried or rinsed using different methods and temperatures. As in the present study, hot-air drying and porcelain furnaces were used as drying methods. In this study, hot air drying at 50°C provided significantly higher bond strength between ceramic-resin cement than drying in an furnace at 100°C.¹¹ The lack of a significant difference in bond strength between the 30 s drying time and the control group can be explained by the fact that alcohol and solvent residues in the silane content remain on the ceramic surface as a result of insufficient drying of the silane agent, as stated in previous studies.

The removal of water supported the completion of the silane-silica surface condensation reaction and the formation of covalent bonds. Therefore, thermal treatment can strengthen the chemical bonds with ceramics in addition to the silane compound. This process may explain why the micro-tensile bond strength increased when hot silane was used with or without the hydrofluoric acid etching step. Although heat treatment is not suitable for intraoral repair, it can be used in the cementation phase, thus removing the dangerous hydrofluoric acid etching phase from the process.³²

The heat drying of silane appears to facilitate and stabilize the siloxane bond. Combining heat drying and hot water rinsing has been proposed as the most suitable silane application technique.³³

In the present study, the selection of the primer drying method was guided by practical considerations of clinical application and accessibility of materials. By simulating the thermal conditions of the oral cavity during the testing process, it may be possible to obtain more reliable clinical data. Additionally, only one type of primer was employed in the current study, and it is recommended that future research incorporates primers with varying solvent contents and drying temperatures to provide a more comprehensive understanding.

Conclusion

Within the limitations of our study, the following conclusions were drawn.

1- Drying the primer in a ceramic furnace at 100°C for 60 s or at $60 \pm 5^\circ\text{C}$ for 60 s significantly increased the bonding strength between the leucite-reinforced glass ceramic and resin cement.

2- Increasing the primer drying time increases the shear bond strength.

3- Primer drying time can be increased in accordance with the manufacturer's instructions.

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