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Adhesive Performance of Simplified Protocols for Adhesion to Lithium Disilicate Ceramics

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Abstract

Background: Durable adhesion to glass-ceramic materials such as Lithium disilicate (LD) relies on effective surface conditioning strategies involving etching, salinization, and adhesive application. While conventional multi-step protocols have demonstrated reliable bonding performance, simplified systems have been introduced to reduce clinical complexity. However, their impact on long-term adhesive interface stability and resistance to degradation remains a subject of investigation.

Objective: This study aimed to evaluate the immediate and long-term shear bond strength (SBS) and interfacial durability of resin cement bonded to LD using different conventional and simplified surface treatment protocols.

Methods: Forty LD cylinders (IPS e.max Press, Ivoclar Vivadent, Schaan, Liechtenstein) were randomly assigned to four groups (n = 10): HSil (9.5% hydrofluoric acid etching + separate silane application (Bisco, Schaumburg, IL, USA)), HSBU (hydrofluoric acid + Single Bond Universal adhesive (3M ESPE, St. Paul, USA)), HCFU (hydrofluoric acid + Clearfil Universal Bond adhesive (Kuraray, Tokyo, Japan)), and MBEP (self-etching primer Monobond Etch & Prime (Ivoclar-Vivadent, Schaan, Liechtenstein)). Resin cement (Choice 2, Bisco, Illinois, USA) was applied following surface treatment. Half of the specimens were tested after 24 h of water storage, and the remaining half after 6 months. SBS was measured using a universal testing machine, and failure modes were analyzed. Statistical analysis was performed using two-way analysis of variance ($\alpha = 0.05$).

Results: After 24 h, the HSil group exhibited the highest mean SBS (29.29 ± 4.40 MPa), whereas the MBEP group showed the lowest values (21.39 ± 5.54 MPa). After 6 months, the HSBU group demonstrated the highest SBS (22.87 ± 10.38 MPa), while the HCFU group presented the lowest values (15.89 ± 7.15 MPa). No statistically significant differences were observed among groups at either time point ($p \geq 0.159$), and no significant reduction in bond strength was detected after aging ($p \geq 0.079$). Failure mode analysis revealed a predominance of adhesive failures across all groups.

Conclusions: This study found that simplified surface treatment protocols, including self-etching primers, provide LD bonding performance comparable to conventional methods over 6 months. While conventional approaches initially demonstrated higher bond strength, all techniques exhibited comparable stability after artificial aging, supporting the use of simplified approaches to reduce marginal degradation and improve hygiene-related outcomes.

Keyword

Adhesive force; Cementation; Lithium disilicate; Metals; Monobond Etch and prime; Shear; Universal adhesive.

Introduction

Dental ceramics are widely recognized for their bioinert properties and biocompatibility in the oral environment. The metal-ceramic bond, a well-established material bond, has been utilized in dental crowns and bridges for over 40 years. The increasing demand for natural-looking and aesthetically pleasing smiles has led to a greater use of all-ceramic materials for indirect restorations [1-3]. Among these materials, lithium disilicate (LD) reinforced glass ceramic is one of the most popular [4]. This ceramic system is available in forms that are either pressed (IPS e.max® Press, Ivoclar Vivadent, Schaan, Liechtenstein) or milled (IPS e.max® CAD, Ivoclar Vivadent, Schaan, Liechtenstein) using Computer-Aided Design and Computer-Aided Manufacturing systems [5]. LD is characterized by a silica glass matrix embedded with lithium oxide (Li_2O) crystals, resulting in a material that boasts exceptional flexural strength, chemical stability, and biocompatibility [6].

Achieving predictability in the cementation of ceramic restorations requires excellent mechanical-chemical surface preparation to ensure proper adhesion between the ceramic material and the dentin substrate [7]. This adhesion is facilitated by using adhesive systems that bond both to the tooth and the ceramic surface. The process involves applying hydrofluoric acid (HF) followed by a silane compound. HF acid conditioning of the glass ceramic surface, followed by the application of silane, is often referred to as the "Gold Standard" in this context [8].

The conventional method of inner surface etching of glass ceramics followed by silanization has long been advocated to enhance adhesion, resistance, and durability within the ceramic-cement-tooth structure complex [9]. However, any lapse in correctly executing these steps can jeopardize the long-term success of the restoration. Practitioners seek to streamline procedures without compromising restoration quality, prompting a desire for step reduction [10]. Presently, a universal adhesive system containing silane is commercially available, purportedly eliminating the need for a separate silane application step as per the manufacturer's instructions [8].

Self-etching materials have been introduced for the surface treatment of glass-ceramic materials to simplify the clinical steps by combining the etching and silanization processes [11]. This approach is considered safer than the application of HF, as manipulating HF can expose clinicians and patients to potential risks, such as acid contact and biological damage to soft tissues [12]. Additionally, the self-etching silane primer helps preserve the glass phase of ceramic materials, improving mechanical performance [13]. This preservation enhances the maintenance of adequate adhesion between the restorative materials or dental substrate and the ceramic restorations [14].

Indeed, while the conventional method utilizing HF and a silane coupling agent has proven effective for enhancing bond strength between porcelain restorations and tooth structure, ongoing research seeks to explore updated treatments that may further improve adhesion, particularly with materials like LD. Identifying alternative or supplementary techniques to enhance the bond strength of LD to resin cement remains a key area of interest and investigation in dental materials science. Therefore, the objective of this study was to assess the impact of various surface treatments on the shear bond strength (SBS) between a glass ceramic (LD) and a resin cement. The null hypotheses tested were: (1) there is no difference in the SBS resulting from different surface treatment protocols, and (2) water storage time does not affect the bond strength produced by the different protocols.

Materials and Methods

Experimental design

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In this study, the bond strength of a resin cement LD ceramic was evaluated according to the following factors: surface protocol on four levels: HF + silane, HF + Single Bond Universal, HF + Clearfil Universal Bond, Monobond Etch & Prime (MEP); and aging at two levels: 24 h and 6 months. The sample size was calculated based on a similar study [15] considering a comparative study of 4 independent groups, a minimum detectable difference of 5, a standard deviation of 2, a power of 80% and a type I error of 5%. The materials used and composition are summarized in **(Table 1).**

Name (Manufacturer)	**Composition**
IPS e.max Press (Ivoclar Vivadent)	Lithium Disilicate–Reinforced Pressable Glass-Ceramic.
Porcelain Etchant (Bisco)	9.5% Buffered Hydrofluoric Acid Gel.
Porcelain Primer (Silane primer, Bisco)	Pre-Hydrolyzed Silane Primer, Alcohol, Acetone.
Single Bond Universal (3M ESPE)	10-Methacryloyloxydecyl Dihydrogen Phosphate, Phosphoric Acid Ester Monomer, 2-Hydroxyethyl Methacrylate, Silane, Dimethacrylate, Vitrebond Copolymer, Filler, Ethanol, Water, İnitiators, Silane.
Clearfil Universal Bond (Kuraray)	Ethanol, 2-Hydroxyethyl Methacrylate, 10-Methacryloyloxydecyl Dihydrogen Phosphate, Hydrophilic Amide Monomers, Colloidal Silica, Silane Coupling Agent, Sodium Fluoride, Dl-Camphorquninone, Water.
Monobond Etch&Prime (Ivoclar Vivadent)	Butanol, Tetrabutylammonium Dihydrogen Trifluoride, Methacrylated Phosphoric Acid Ester, Bis(Triethoxysilyl)Ethane.

Table 1: Materials used in this study.

Sample preparation and shear bond strength

Forty cylindrical wax-ups (5mm in diameter and 2mm in height) were created and invested using Empress 2 speed investment material (Ivoclar Vivadent, Schaan, Liechtenstein). The preheating cycle was conducted at 850°C for 60 minutes. The molds were then transferred to the furnace, where they were press-filled with IPS e.max® Press ingot material at 915°C for 20 minutes. After divesting, the ceramic cylinders were embedded in Polyvinyl Chloride (PVC) tube with self-curing acrylic resin, ensuring that the LD surfaces remained exposed. Once embedded, the exposed LD surfaces were polished with 600-grit silicon carbide sandpaper to standardize them.

The samples were randomly divided into 4 experimental groups according to the surface treatment (n=10): 1. HSil (HF + Silane) group: The surface was etched for 20 seconds with HF (9.5% Porcelain Etchant, Bisco, Illinois, USA). Subsequently, the acid was removed with a water jet, and the specimens were placed in an ultrasonic bath for 5 minutes to eliminate the hexafluorosilicates formed. Then, the surface was dried with air, followed by the application of a layer of silane (Silane, Porcelain Primer, Bisco,

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Schaumburg, IL, USA) using SDI microbrushes (SDI Limited, Bayswater, Victoria, Australia), and allowed to dry for 5 minutes. 2. HSBU group: The surface was etched with HF using the same procedure as in the Group 1. After etching, a layer of Single Bond Universal adhesive (3M ESPE, St. Paul, USA) was placed and rubbed on the surface for 10 seconds. Excess solvent was removed using a blast of air for 5 seconds. 3. HCFU group: The surface was etched with HF using the same procedure as in the Group 1. After etching, a layer of Clearfil Universal adhesive (Kuraray, Tokyo, Japan) was placed and rubbed on the surface for 10 seconds. Excess solvent was removed using a blast of air for 5 seconds. 4. MEP group: A layer of MEP (Ivoclar-Vivadent, Schaan, Liechtenstein) was applied and rubbed on the surface for 20 seconds. The material was allowed to react for another 40 seconds. The material was then rinsed completely with a stream of water and air. Finally, the surface was dried using a blast of air for 10 seconds.

Once the surface treatment was carried out, a cylindrical silicone addition matrix with 2 holes of 1.5mm internal diameter was placed on the surface of the ceramic and for the HSBU and HCFU groups the adhesive layer was photopolymerized for 20 seconds. Then, the holes were filled with Choice 2 resin cement (Bisco, Illinois, USA) and light-cured for 20 seconds with a light-curing unit (VEGA light-curing unit (Dentac, Istanbul, Turkey)) with an intensity of 1000 mW/cm^2. After photopolymerization, the silicone matrix was removed to expose two cylinders of resinous cement bonded to the ceramic surface.

Half of the samples were stored in distilled water at 37°C for 24 h, while the remaining samples were kept in distilled water for 6 months. Following storage, all samples underwent a SBS test using a universal mechanical testing machine (Instron 1165, Massachusetts, USA) with a crosshead speed of 1.0 mm/min. The bond strength, expressed in MPa, was calculated by dividing the applied load (in Newtons) by the interface area of the bond (in mm^2). After the shear test, the bond surfaces were examined under a stereomicroscope to identify the modes of failure: adhesive, cohesive, or mixed. All observations were recorded by a single observer.

Statistical analysis

Statistical analyses were conducted using Sigma Plot 12.0 software. The data were evaluated for normal distribution and variance homogeneity. A two-way analysis of variance (ANOVA) was used to assess the effects of surface treatment protocols and storage duration on SBS. The significance level for all tests was set at $\alpha < 0.05$.

Results

(Table 2) and (Figure 1) show the SBS values of the different surface protocols evaluated, both at 24 h and at 6 months. At 24 h, the highest value was for the HSil group (29.29 ± 4.40), while the lowest value was for the group where MEP was applied (21.39 ± 5.54). The statistical analysis revealed that there are no statistically significant differences between the groups ($p \geq 0.228$).

Surface treatment protocol	Shear bond strength	
	24 h (Mean ± SD)	6 months (Mean ± SD)
Monobond® Etch & Prime	21.39 (5.54)	19.00 (5.0)

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SingleBond™ Universal	23.50 (6.0)	22.87 (10.38)
CLEARFIL™ Universal	21.00 (6.5)	15.89 (7.15)
HSil	29.29 (4.40)	20.00 (6.0)
There were not statistically significant differences in any of the possible comparisons (p>0.05).		

Table 2: Shear bond strength of the different lithium disilicate adhesion protocols; SD = standard deviation.

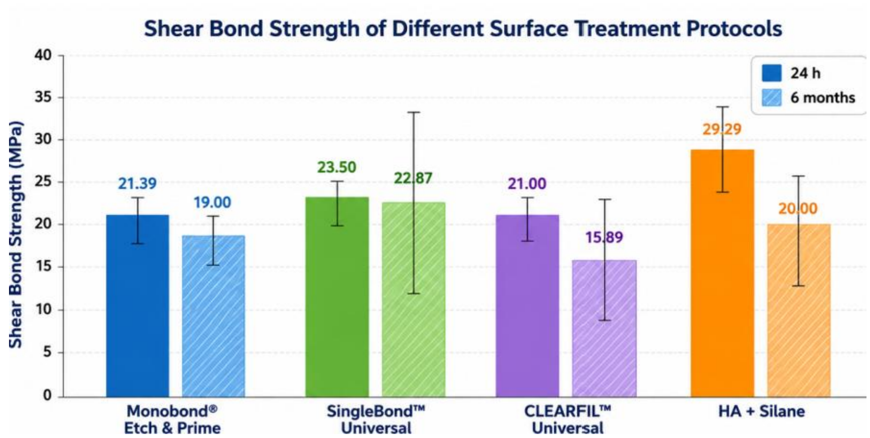

Figure 1: Shear bond strength of different surface treatment protocols after 24 h and 6 months of storage; HSil= HA + Silane.

After 6 months of aging, the highest SBS was observed in the Single Bond™ Universal group (22.87 ± 10.38), while the lowest was in the Clearfil™ Universal group (15.89 ± 7.15). Statistical analysis indicated no significant differences between the groups ($p \geq 0.159$). Additionally, none of the materials exhibited a statistically significant decrease in bond strength after 6 months of storage ($p \geq 0.079$).

(Figure 2) shows the percentages of the failure mode distribution observed after the SBS test at 24 h storage. The HSBU (Single Bond Universal), HCFU (Clearfil Universal Bond), and MEP groups had a predominance of adhesive failures (Figure 3). The failure mode after 6 months of storage was adhesive type for all groups.

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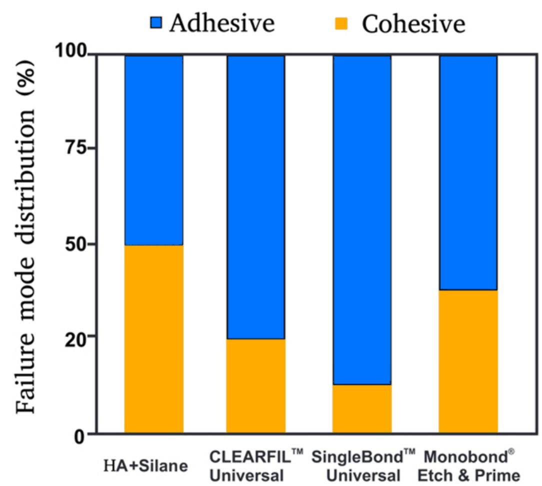

Figure 2: Failure mode distribution after bond strength test at 24 h of storage. The failure mode after 6 months of storage was adhesive type for all groups; HSil= HA + Silane.

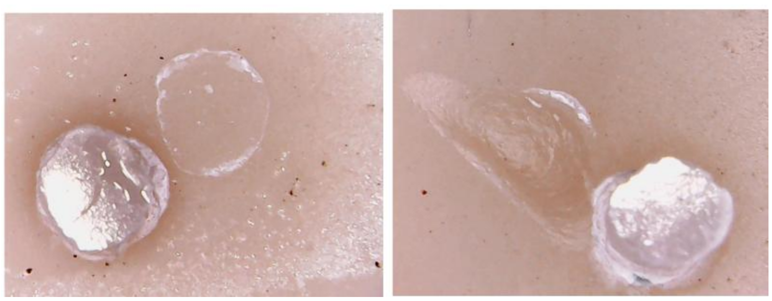

Figure 3: Representative image of an adhesive (left) and cohesive (right) failure mode.

Discussion

The SBS test stands out as the optimal methodology for assessing bond strength, particularly applicable to fragile materials including glass ceramics and enamel. Unlike other methods, it does not necessitate the sectioning of specimens. This is fundamental, as sectioning such brittle materials for micro tensile bond strength tests frequently leads to a major occurrence of cohesive failures [16,17]. Resin bonding necessitates several pre-treatment steps on the intaglio surface of the restoration to establish a close bond between the ceramic surface and the luting agent. In contrast, the traditional surface treatment protocol for glass ceramics, encompassing feldspathic, leucite-enhanced, and LD materials, involves HF etching followed by the application of a silane coupling agent [18]. The current research accepted the null hypothesis, as the bond strength of LD to resin cement remained unaffected by both the surface treatment and aging time.

After 24 h, the results indicated that the HSil (HF + silane) group had the highest mean SBS (29.29 ± 4.40), whereas the MEP group exhibited the lowest mean value (21.39 ± 5.54). Despite these differences, no statistically significant variation was observed among the groups. The high bond strength in the HSil group could be attributed to the application of silane without additional adhesive, which might enhance

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the bonding by HF's ability to create micro-retentions on the ceramic surface, facilitating the flow of resin cement [19-21]. The effectiveness of the silane agent may be due to its formation of three-dimensional siloxane bonds between hydroxyl groups (-OH) in the silica-based ceramic and methacrylate groups in both the adhesive and resin cement [22].

HF (5–10%) is commonly used to treat the inner surface of ceramic restorations. It partially dissolves the ceramic glass matrix, exposing the crystalline structure and promoting mechanical interlocking between the ceramic and resin cement. Without silane treatment, the bond strength of LD relies on micromechanical retention created by HF etching. The application of silane improves the chemical bond between LD and resin cement, thereby enhancing bonding strength. The study suggests that the micromechanical retention resulting from HF etching plays a more critical role in bond strength than the chemical bonding provided by silane. Therefore, HF pretreatment may be essential for optimizing the bonding potential of ceramic restorations [23].

For both the HSBU and HCFU groups, the quantity of silane present in the tested universal adhesives may not be sufficient to achieve the same effect as when applied separately prior to the universal adhesive, potentially resulting in higher bond strength [24]. Consequently, the findings of this study advocate for the independent application of silane, irrespective of its presence within the universal adhesive solution. Similar conclusions were drawn in the study by Kavalacharla et al., where they highlighted that applying silane before the universal adhesive led to superior bond strength. Moreover, they recommended extending the etching time from 20 to 60 seconds if silane application is omitted, as adhesion to the ceramic surface then solely relies on the micromechanical retention facilitated by HF etching [22]. According to the manufacturer's recommendations, separate primers for adhesion to silicate ceramics are not required when using Clearfil Universal Bond and Single Bond Universal– all of which are universal adhesives included in this study. However, findings from a previous meta-analysis indicated that the silane incorporated within universal adhesives may not be as effective as a silane coupling agent applied independently in optimizing the bond between ceramic and resin cement [8]. This was in agreement with the finding of this in vitro study.

The reduced efficacy of universal adhesives when utilized as ceramic primers may be attributed to the limited stability of the silane coupling agent within the acidic aqueous adhesive solution. In the presence of water, the silane groups (triethoxysilane) undergo hydrolysis, converting into silanol groups (silanol), which can adhere to and chemically bond with glass surfaces [25]. Subsequently, the silanol groups may undergo dehydroxylation and condensation to form siloxane oligomers that lose their ability to bond with glass [26]. Additionally, it has been observed that interactions between the various monomers present in the universal adhesive may impact the silane's ability to couple with silica. For instance, the presence of Bisphenol A diglycidyl methacrylate (BisGMA) can inhibit the condensation reaction between the silanol group and the substrate [27]. Regarding the HF concentration utilized in this investigation (9.5%) compared to the manufacturer's recommended concentration (5%), no significant disparity in bond strength or surface morphology could be discerned when applied for 20 seconds [28].

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In addition, it has been demonstrated that combining non-silane-containing universal adhesives with silane can result in higher bond strength compared to using universal adhesives alone. This phenomenon may occur because the acidic monomer 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) present in universal adhesives can bond to metals and neutralize silane, rendering it unstable over time [29]. Therefore, it can be concluded that using silane primers independently after HF treatment is more effective than relying solely on silane-containing universal adhesives.

Adding to the aforementioned findings, after 24 h of water storage, the MEP group displayed the lowest bond strength, prompting consideration of adjusting the application time of the one-step self-etching ceramic primer to enhance the mechanical performance of LD restorations. This variation in application time could influence the introduction of surface defects, with potential dichotomous consequences. On one hand, a longer etching time may lead to additional defects that are subsequently filled in by the resin cement, resulting in improved stress distribution and reduced failure probability. Conversely, an increase in the number, size, or irregularity of defects may impede resin cement penetration, thereby compromising adhesion and elevating the risk of failure and reduced mechanical performance [18]. MEP, comprising tetrabutylammonium dihydrogen trifluoride, trimethoxypropyl methacrylate (silane), and methacrylated phosphoric acid ester, serves as the ceramic primer [30]. Various fluoride sources have been explored for ceramic etching, including acidulated phosphate fluoride, titanium tetrafluoride, and ammonium bifluoride [31]. Tetrabutylammonium dihydrogen trifluoride in MEP, derived from ammonium bifluoride (ABF), offers a less toxic and hazardous alternative to HF [32]. Research indicates that ABF produces etching patterns similar to HF but with lower toxicity and hazard levels. While ABF demonstrated greater efficacy on Dicor castable glass ceramic than HF [15], etching current glass-matrix ceramics with HF yielded higher mean tensile bond strengths compared to ABF [33]. This aligns with the study findings, given MEP's inclusion of ammonium fluoride.

After 6 months, the highest bond strength was recorded in the Single bond™ Universal group (22.87 ± 10.38), whereas the lowest was observed in the Clearfil™ Universal group (15.89 ± 7.15). Statistical analysis revealed no significant differences among the groups ($p \geq 0.159$). The Single bond Universal adhesives incorporate hydrophilic monomers and solvents in their composition, potentially enhancing water sorption and plasticization of the adhesive interface [34]. Conversely, in the absence of adhesive usage, adequate wetting of the etched ceramic surface by the hydrophobic resin cementation material could promote better bond maturation even after water immersion, thus slowing down degradation via water absorption [24].

Furthermore, in this study, Clearfil Universal demonstrated relatively lower bonding strengths compared to Single bond Universal, consistent with previous findings. This disparity in bond strengths between the two adhesives could be attributed to the ambiguity surrounding their compositions, ratios, and concentrations. Single bond Universal contains polyalkenoic acid copolymers (Vitrebond copolymer; 3M ESPE), known to enhance bond strengths [35]. On the other hand, Clearfil Universal incorporates colloidal silica as a filler to achieve appropriate resin thickness and viscosity. However, this addition may lead to reduced adhesive layer flow, localized adhesive concentration, and void formation, potentially

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compromising bonding strength and the efficacy of silane in the Clearfil Universal group [36]. Consequently, the hypothesis that additional silane application would not impact the effectiveness of silane-containing universal adhesives is refuted. An additional silane application must be a need for universal adhesive when bonding LD to resin cements.

Regarding the distribution of failure modes observed after the SBS test at 24 h of storage, this can be attributed to various factors. The HSBU (Single Bond Universal), HCFU (Clearfil Universal Bond), and MEP (Monobond Etch & Prime) groups exhibited a predominance of adhesive failures (Figure 3). However, after 6 months of storage, the predominant failure mode for all groups was adhesive type. These results suggest that the failure occurred at the interface between the ceramic and the adhesive, rather than within the ceramic, adhesive, or the resin cement itself. Aging of bonded interfaces, especially in a moist environment like the oral cavity, can lead to hydrolytic degradation of the adhesive layer [37]. Water can penetrate the adhesive interface, causing hydrolysis of the silane coupling agents and resin matrix, leading to a reduction in bond strength over time [38]. The consistent finding of adhesive failures after 6 months indicates that the interface weakened over time due to these degradative processes. Despite no significant decrease in bond strength after 6 months, the mode of failure suggests that the bond may not be robust enough for longer-term performance without further optimization. Clinicians should be aware of these potential issues and consider additional measures, such as enhanced surface treatments or the use of more durable adhesives, to ensure long-term success of ceramic restorations.

Conclusions

In conclusion, the findings of this study suggest that while specific surface treatments may initially influence the bond strength between glass ceramic and resin cement, the long-term stability of these bonds is comparable across different treatment methods. This provides clinicians with multiple effective options for surface treatment in the cementation of ceramic restorations, contributing to better outcomes and enhanced safety in dental practice.

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