Research Article

Shear bond strength and internal adaptation of lithium disilicate veneers luted to enamel using three different luting agents

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Abstract: Background: Lithium disilicate veneers rely on both strong resin–ceramic bonding and intimate adaptation for long term success. Universal adhesives containing 10-Methacryloyloxydecyl Dihydrogen Phosphate may enhance the performance of contemporary resin cements, yet evidence for their effect on bioactive and self-adhesive luting agents remains limited. Aim: To compare the shear bond strength (SBS) and internal adaptation of IPS e.max CAD veneers luted to enamel with three resin cements — bioactive dual cure (Predicta), light cure (Variolink Esthetic LC) and self-adhesive (RelyX U200) — used with or without a universal bonding agent. Materials and Methods: Forty-eight sound third molars were prepared to mid enamel, CAD/CAM veneers were etched with 10% hydrofluoric acid and silanated, and teeth were etched with 37 % phosphoric acid. Specimens were allocated randomly (n = 8 per subgroup) to one of six cement–bonding combinations. Bonded subgroups received a 10 MDP universal adhesive; unbonded subgroups did not. After cementation and light curing, all samples were thermocycled 5,000X between 5 °C and 55 °C. SBS was measured using a universal testing machine (1 mm min^{-1}), failure modes were classified at 20× magnification, and marginal/internal gaps were quantified by micro computed tomography (micro-CT). Results: Predicta exhibited the highest SBS in both bonded (140 ± 24 MPa) and unbonded (80 ± 60 MPa) conditions. Variolink LC showed the lowest SBS, especially when unbonded (26 ± 35 MPa). RelyX U200 displayed moderate SBS that increased significantly with bonding ($p < 0.05$). Micro CT revealed smaller gap volumes for RelyX U200 overall, while Predicta's adaptation improved notably when bonded. Failure analysis showed predominantly cohesive failures for Predicta and Variolink; RelyX U200 shifted from cohesive to adhesive failures when adhesive was applied ($p < 0.05$). Conclusion: Applying a universal bonding agent significantly improved both bond strength and internal adaptation of resin cements, particularly Predicta and RelyX U200. Predicta cement combined with 10 MDP adhesive delivered the most favorable enamel–ceramic interface, supporting its clinical use for lithium disilicate veneers.

Keywords: Dental Bonding; Dental Veneers; Resin Cements; Silicates; Tomography; X Ray Computed.

Introduction

The esthetic restoration has been developed over the past decade and the lithium disilicate ceramics, particularly IPS e.max, have become the material of choice for esthetic restorations, due to their optical qualities, mechanical strength, and durability [1].

The long-term success of IPS e.max restorations relies heavily upon the strength and stability of the bond between the restoration and underlying substrate material [2]. However, achieving a durable and effective bond is a complex process influenced by numerous variables [3], including the type of surface treatment applied to both the ceramic and the underlying substrate, the nature of the substrate material itself, and the physicochemical behavior of the luting agent, Surface treatments such as hydrofluoric acid, especially in high concentrations such as 10%, modify the fitting surface of the ceramic's topography and chemical composition, subsequently enhancing micromechanical retention (3). Similarly, the characteristics of the substrate such as the enamel surface, play a role in interfacial bonding performance due to differences in composition, surface energy, and interaction with the cement (4). The cementation process creates reliable retention and a tough seal of the space between the tooth and the restoration; therefore, the characteristics of the luting cement also play a critical role in the success of the restoration (5). Dual-cure adhesive cements combine the advantages of light and chemical curing, ensuring complete polymerization even in areas with limited light access, thus offering high bond strength when bonded to restoration or enamel (6) (7). Self-adhesive resin cements simplify clinical application by eliminating the need for separate etching and bonding steps, making them attractive for time-sensitive procedures, and a study found that the self-adhesive resin cement exhibit the best shear bond strength when bonded to restorations but lower results when bonded to enamel (7). The emergence of bioactive materials offers the potential for ion release to aid hard tissue healing; however, their long-term bonding efficacy with IPS e.max remains unclear (8). Although putative advances in luting techniques and strategies are available, existing standardized data comparing bond strength, marginal and internal fit across different luting agents and core substrates, and the effect of bioactivity of bioactive materials upon the bond strength and their behaviours in general, are limited. Addressing these information gaps is therefore important to understand the optimal combinations for durable and aesthetically successful restorations, ultimately supporting improved clinical outcomes.

The null hypothesis of this *in vitro* study is that the bioactivity of the luting agent has no effect on the interfacial bond strength or the internal adaptation of lithium disilicate restorations.

Materials and Methods

Materials

The study's protocol, which complied with the Helsinki Declaration, was approved by the Human Ethics Committee of the University of Mosul College of Dentistry (reference number: 6943). Human enamel was used as the core substrate. Three types of luting agent was used in this study , they are listed in the table below:

Table 1: luting agent used for the study.

Luting agent (brand)	Type / Curing mode	Key components (as provided)	Manufacturer (city, country)
Predicta™ Bioactive Luting Cement	Bioactive resin cement; dual-cure	bis-GMA, UDMA; bioactive glass fillers containing calcium phosphate ($Ca_3(PO_4)_2$) and fluoride (F^-)	Parkell, Edgewood, New York, Untied American state
RelyX™ U200	Conventional resin cement; light-cure	bis-GMA, UDMA, TEGDMA; barium glass fillers; ytterbium trifluoride (YbF_3)	Ivoclar Vivadent AG, Schaan, Liechtenstein
Variolink® Esthetic LC	Self-adhesive resin cement; dual-cure	Methacrylate monomers; zinc oxide (ZnO) base; peroxide catalyst	3M Oral Care (3M ESPE), St. Paul, Minnesota, Untied American state

The equipment used in the study: includes the followings: five-axis milling machine: MAXX NX (Robots & Design, Korea), Universal testing machine: GT KS20 (Gester, China), Cone beam micro-CT scanner: LOTUS inVivo (Tehran, Iran) and Stereomicroscope (20× magnification): Optika (Italy).

Tooth selection, storage and preparation for the sample

Forty-eight sound, freshly extracted human third molars with comparable mesio-distal and bucco-lingual dimensions were collected after informed patient consent. After calculus removal (ultrasonic scaler) and prophylaxis with a slow speed handpiece and brush (pumice), crowns at 10× magnification were scored using ICDAS II. Teeth exhibiting any caries lesions (ICDAS codes 1 – 6) on the buccal surface were excluded, and only ICDAS score 0 (sound) specimens were retained and stored in distilled water at (22 ± 1 °C) for ≤ 1 month, following ISO TS 11405 recommendations. The buccal surface was cut using a low-speed precision diamond saw (Isomet 1000, Buehler Ltd., USA) under water cooling to fabricate the (4 mm diameter × 2 mm thickness) enamel surface.

Samples grouping

The total sample size consisted of 48 specimens, which were divided into two main groups based on the use of an adhesive bonding agent. In the first group, specimens were luted with the application of an adhesive bonding agent, while in the second group, specimens were bonded without adhesive. Each of these two groups (n = 24) was further subdivided into three subgroups (n = 8 per subgroup), according to the type of luting agent used.

Veneer fabrication

CAD/CAM veneers were milled from IPS e.max CAD lithium disilicate blocks primarily composed of lithium disilicate ($Li_2Si_2O_5$) embedded in a glassy silica (SiO_2) matrix with aluminum oxide (Al_2O_3), potassium oxide (K_2O), phosphorus pentoxide (P_2O_5), and zirconium oxide (ZrO_2). Lithium-disilicate veneers (4 mm diameter × 2 mm thickness) were CAD-designed and milled using a five-axis MAXX CAD/CAM device (Robots and Design Co., Ltd. (South Korea)), then crystallized according to the manufacturer's schedule (850 °C/20 min).

Surface treatment of veneers

Intaglio surfaces were etched : 10% hydrofluoric acigel [3] ((HF) — FGM, Brazil the patch no. 443324 and the expair date 2025-6-20) fllowed with Monobond N silane coupling agent (Ivoclar Vivadent, Liechtenstein. The patch no.3004424 and the expair date 2024-14).

Core preparation

Enamel surfaces were flattened with a flat-end diamond bur (236 C Lusterdent Medical Instrument Co., Ltd. China) under water cooling (400,000 rpm, 50–60 mL min^{-1}, 60 s). After ultrasonic cleaning for 10 min (by using Heavy Duty Cleaning, 600 ml Tank, Digital Timer (MGUC500)) and air-drying (10 s), all cores were etched with 37% phosphoric acid gel (H_3PO_4) (Transene Company, Inc. (USA) the patch no. : 2322577 with expire date: 2026-12-01) then one subgroup bonded with G-Premio Bond(G-Premio BOND GC Corp. (Tokyo, Japan) the patch no. 2407267 with expire date: 2026-07-25), a universal one-bottle adhesive containing 4-MET, 10-MDP, DMA, phosphoric acid ester monomers, and organic solvents. The other subgroup bonded alone with no bonding agent.

Luting procedure

Cements were mixed/dispensed per manufacturer instructions, applied to the veneer intaglio, and seated under a 1 kg axial load for 10 s [9] using a custom surveyor jig to standardize film thickness. Excess was removed and light polymerization was completed with an LED unit (1000 mW cm^{-2}) through the ceramic for 40 s per surface.

Thermocycling (artificial ageing)

All 48 bonded specimens underwent 5,000 thermal cycles between 5 °C and 55 °C with a 30 s dwell and 10 s transfer time (ISO TR 11405), simulating approximately six months of oral ageing [10].

Shear-bond strength (SBS) test

Thirty specimens (5 per cement × bonding condition) were mounted in a jig and loaded with a chisel-shaped blade at 0.5 mm min^{-1} until failure (universal tester). SBS (MPa) was calculated as:

SBS = F/A,

where F = failure load (N).

$A = \pi r^2$ (r = 2 mm)

Failure mode analysis

Deboned interfaces were inspected under a stereomicroscope at 20X (Optika microscopes; Ponteranica; Italy) and categorized as adhesive, cohesive, or mixed.

Micro-CT evaluation and internal adaptation

A total of eighteen representative specimens were selected, with six specimens allocated to each luting agent group. Within each group, three specimens were assigned to the bonded subgroup and three to the unbonded, they were scanned with an in vivo X-ray Micro-Computed Tomography (micro-CT) scanner (LOTUS inVivo, Be hin Negareh Co., Tehran, Iran). LOTUS-inVivo has a cone beam micro-focus X-ray source and a flat panel detector. In order to obtain the best possible image quality, the X-ray tube voltage and its current were set to 80 kV and 90 μA, respectively and the frame exposure time was set to 2 seconds by 3.7 magnification. Total scan duration was 30 minutes. Slice thicknesses of reconstructed images were set to 25 micrometers. The protocol settings process was controlled by LOTUS-inVivo-ACQ software. The acquired 3D data was reconstructed using LOTUS inVivo-REC by a standard Feldkamp, Davis, Kress (FDK) algorithm. Perpendicular to the X-ray beam (70 kV, 200 μA, 25 μm voxel). 3-D reconstruction (FDK algorithm; LOTUS-inVivo-REC) allowed measurement of marginal and internal gap volumes (μm^3) using threshold-based segmentation.

Statistical analysis

Data normality and homoscedasticity were verified (Shapiro–Wilk, Levene). Shear-bond strength and gap volume were compared by two-way ANOVA (cement × bonding) with Tukey post-hoc tests ($\alpha = 0.05$). Failure-mode distributions were analyzed using Fisher–Freeman–Halton exact tests; Cramér's V quantified effect size. Analyses were performed in GraphPad Prism 10.2 (GraphPad Software, USA).

Results

Shear-bond strength

For the bonded condition, assumptions for parametric testing were met across groups; therefore, a one-way ANOVA followed by Tukey's HSD was used. Pairwise comparisons showed that Predicta™ Bioactive Luting Cement produced significantly higher SBS than both RelyX™ U200 and Variolink® Esthetic LC (Tukey-adjusted p's < 0.05), and RelyX U200 was significantly higher than Variolink Esthetic LC (adjusted $p < 0.05$). For the unbonded condition, given the non-normality in the Variolink group (Shapiro–Wilk $p = 0.024$) and extreme outliers within RelyX U200 (values > 300 MPa), we applied a Welch ANOVA with Games–Howell post-hoc (confirmed by a sensitivity Kruskal–Wallis/Dunn analysis). The same direction of differences was observed: Predicta Bioactive > RelyX U200 > Variolink Esthetic LC, with Predicta significantly higher than both comparators and RelyX significantly higher than Variolink after multiple-comparison correction (adjusted p's < 0.05). Robust and nonparametric sensitivity analyses yielded unchanged conclusions. The shear bond strength results are shown in Figure 1.

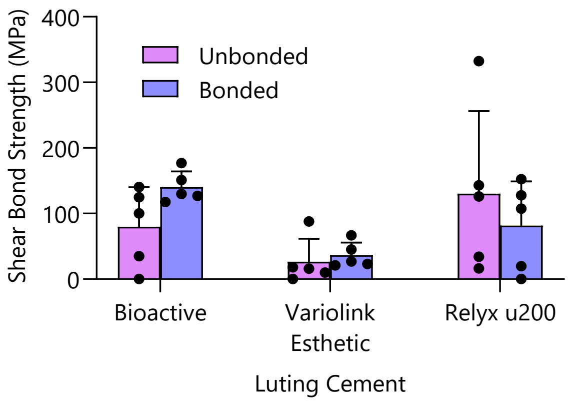

Figure 1: Shear bond strength (MPa) of three types of luting cements under bonded and unbonded conditions. Each bar represents the mean ± standard deviation (SD), with individual data points shown. Bonded groups are shown in blue, unbonded in pink.

Internal adaptation

For unbonded specimens, all groups met normality; one-way ANOVA with Tukey's HSD demonstrated that Predicta Bioactive exhibited significantly larger internal gaps than both RelyX U200 and Variolink Esthetic LC (adjusted *p*'s < 0.05), while RelyX U200 had the smallest gaps and was significantly lower than Variolink (adjusted $p < 0.05$ or nonsignificant if your post-hoc shows otherwise). In the bonded condition (Predicta borderline normality), ANOVA (Welch if variances unequal) with Tukey/Games–Howell indicated that RelyX U200 showed smaller and more consistent gaps than Predicta (adjusted $p < 0.05$), and RelyX was smaller than Variolink (adjusted *p* as returned by your post-hoc). The slight increase in internal gap for Variolink when bonded, compared with its unbonded counterpart, did not reach statistical significance (adjusted $p > 0.05$). Internal adaptation, measured as the gap in micrometers between the luting cement and the internal surface, is presented in Figure 2.

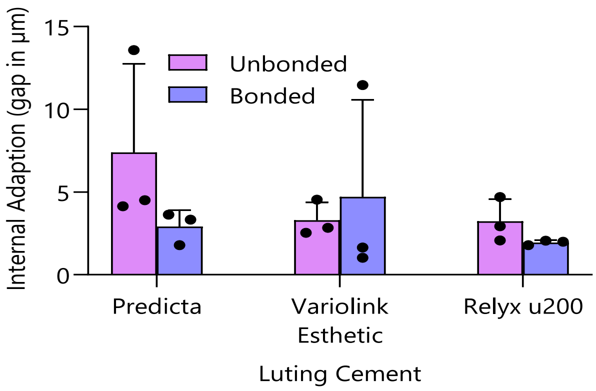

Figure 2: Internal adaptation (measured as gap in µm) of three luting cements under bonded and unbonded conditions. Bars represent the mean ± standard deviation (SD), and individual data points are shown. Bonded groups are shown in blue, unbonded in pink.

Failure mode analysis

The distribution of failure modes is summarized in Figure 3. Both Bioactive and Variolink Esthetic cements exhibited predominantly cohesive failures, with no clear difference between bonding conditions, as is shown in Figures 4 and 5. However, RelyX U200 showed a significant shift in failure patterns, with the bonded group presenting an increased frequency of adhesive failures ($p < 0.05$, chi-square test), as is shown in Figure 6. This change suggests that bonding may affect the failure mechanism, potentially through improved interfacial interaction or altered stress distribution.

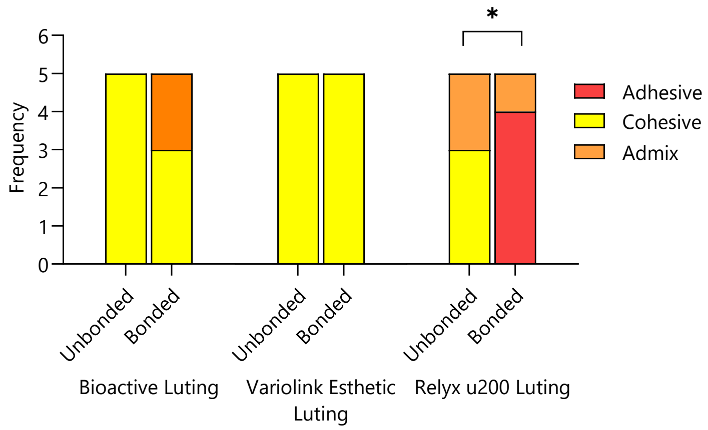

Figure 3: Failure mode distribution of luting cements under bonded and unbonded conditions. Bars show the frequency of adhesive (red), cohesive (yellow), and admixed (orange) failures. A statistically significant difference was observed between bonded and unbonded groups in RelyX U200 ($p < 0.05$, chi-square test).

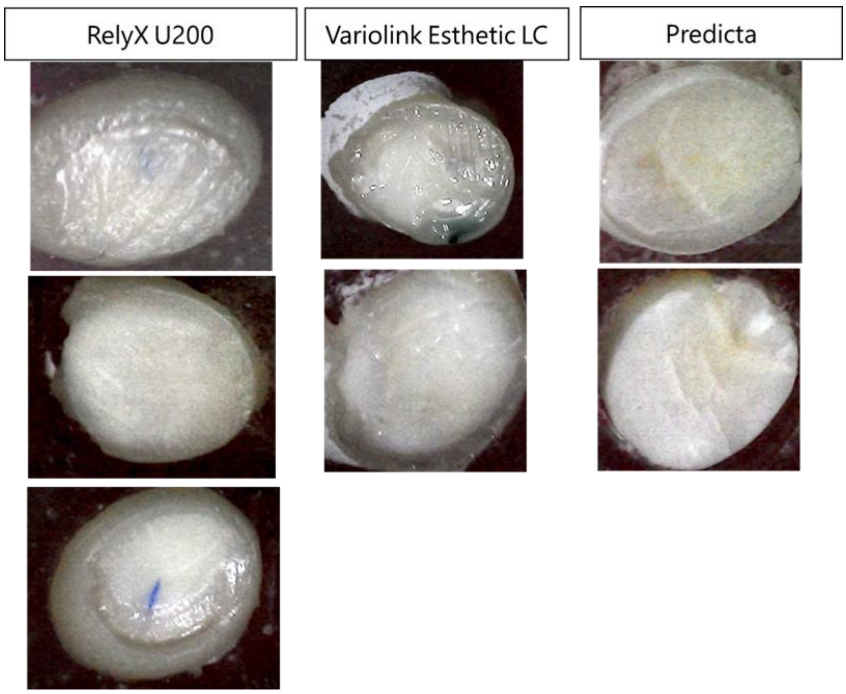

Figure 4: Representative stereomicroscopic images showing the internal adaptation of lithium disilicate veneers luted to enamel using three different resin cements: RelyX U200, Variolink Esthetic LC, and Predicta. Differences in marginal integrity and gap formation can be observed among the tested materials.

Internal adaptation

Micro-CT analysis revealed clear differences in internal fit between bonding protocols and cements Figure 5. Bonded Predicta exhibited the most favourable interface, presenting a thin, continuous cement film with no detectable internal gaps. Omission of the adhesive tripled the gap volume and introduced a continuous interfacial void. Variolink Esthetic LC showed an irregular but closed interface when bonded;

removal of the adhesive produced the largest discontinuities and occasional bulk voids, correlating with that subgroup's low bond strength. RelyX U200 demonstrated satisfactory adaptation in both protocols; however, the bonded subgroup displayed a slightly more uniform cement layer and fewer internal voids, consistent with its 60 % higher shear-bond strength. Overall, application of the 10-MDP universal adhesive reduced gap volume and maximum marginal gap for all three cements, with the greatest improvement observed in the bioactive Predicta system.

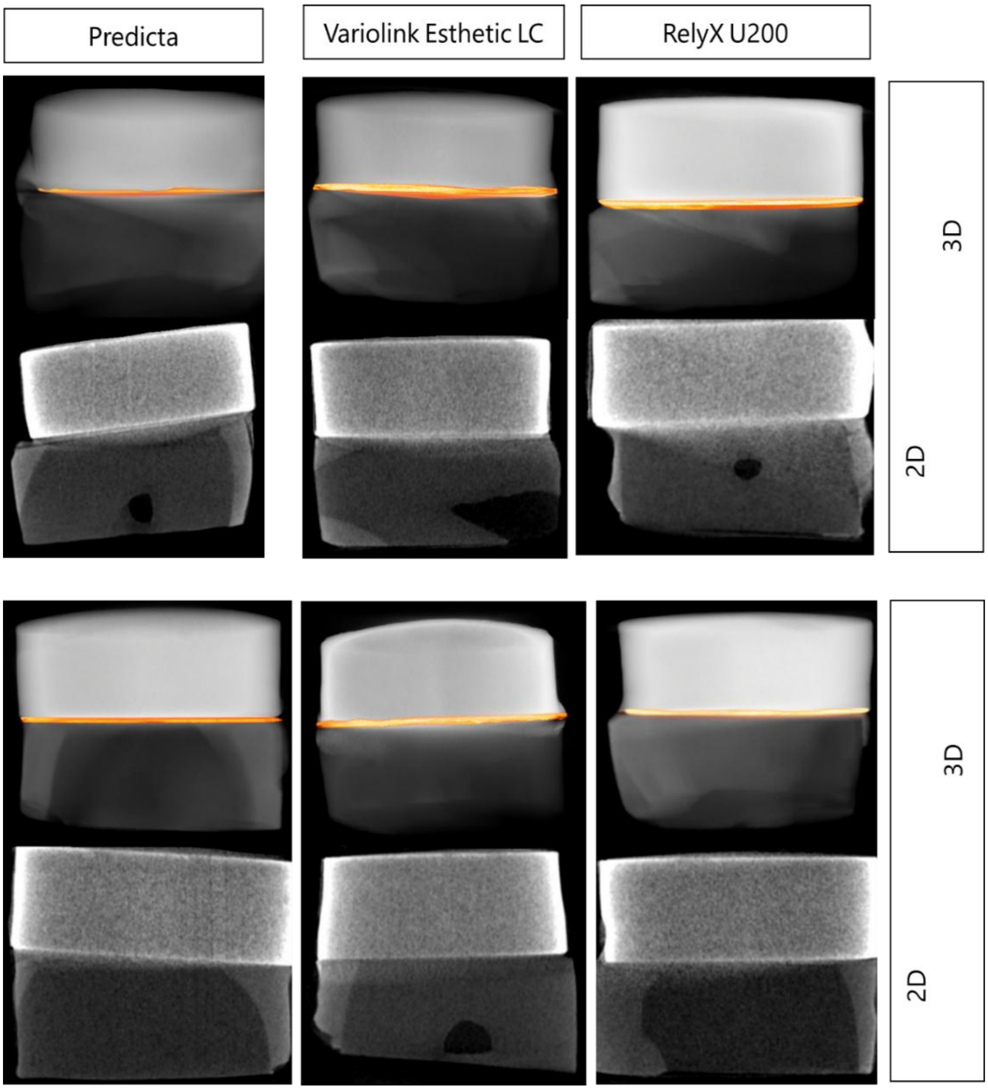

Figure 5: Representative micro-CT images illustrating the internal adaptation of lithium disilicate veneers luted with three resin cements under bonded and un bonded protocols. Columns display Predicta Bioactive, Variolink Esthetic LC, and RelyX U200. For each cement, the upper image is a false colour three-dimensional reconstruction (orange = cement layer); the lower image is the corresponding sagittal two dimensional slice. The upper block shows bonded specimens, the lower block un bonded specimens. Scale bar = 1 mm.

Discussion

Enamel is a structure composed of approximately 96% impure hydroxyapatite by weight, with a highly organized crystalline and prismatic structure. Enamel does not contain organic resin matrices or methacrylate groups, which are critical for chemical bonding with resin-based luting agents [11]. Phosphoric acid etching creates a microporous surface by selectively demineralizing the enamel rods. Resin cement or adhesive materials then flow into these porosities, forming an intact hybrid layer which forms resin tags upon polymerization, creating micromechanical interlocking [11, 12].

In the present study, GC-primero adhesive agent was used as it contains 10-Methacryloyloxydecyl dihydrogen phosphate (10-MDP), a functional monomer able to form stable nano-layers through chemical interaction with calcium in the core material by forming MDP-Ca salts [13]. These salts balance hydrophilicity/hydrophobicity, reducing water sorption and degradation, thereby enhancing the durability and aging resistance of the adhesive interface [13, 14].

This *in vitro* study evaluated the shear bond strength (SBS) and internal adaptation of lithium disilicate veneers bonded enamel, which underwent surface smoothening using high speed flat end bur and were cleaned using an ultrasonic device, and before the luting they were totally etched and rinsed and each group were subdivided into two groups, one subgroup with adhesive GC- premio adhesive agent and then were luted and the other subgroup were directly luted without the use of the adhesive agent.

Then all samples were luted using three different types of luting agents: bioactive (Predicta cement), light-cure (Variolink Esthetic LC), and self-adhesive (RelyX U200), with assessment of adhesive bonding protocols.

The results were interpreted based on statistically significant differences observed in both shear bond strength (SBS) and internal adaptation. Predicta Bioactive cement consistently achieved the highest SBS, with significance confirmed particularly when used with 10-MDP adhesive, while RelyX U200 showed intermediate values that improved significantly upon bonding, and Variolink Esthetic LC yielded the lowest, especially in unbonded groups. The analyses indicated that the improvement in RelyX U200 with bonding ($p < 0.05$) and the superiority of Predicta over both other cements were statistically significant. Internal adaptation results corroborated these findings, with Micro-CT data showing significantly reduced gap volumes when adhesives were applied, particularly in Predicta and RelyX U200 specimens. Additionally, failure mode distribution analysis revealed a statistically significant shift ($p < 0.05$) in RelyX U200 from cohesive to adhesive failures when bonding agent was applied, confirming that the application of adhesive had a measurable influence on interfacial performance.

The high results of Predica can be explain by that the Predicta bioactive luting cement has a phosphate-based monomer, which exhibits capacity for the precipitation of apatites in conjunction with calcium ions; this capacity enhances bonding and gap closure over time [15]. Predicta bioactive luting cement is designed to chemically interact with calcium-containing substrates such as enamel, so the results were the highest among the others, especially when used with 10-MDP containing adhesive agent, where it establishes MDP-Ca bond [13, 16, 17]. Reactive methacrylate sites or hydroxyl groups of the bonding agent have been available for bonding; these chemical groups can interact with the bioactive cement's matrix during polymerization, enhancing chemical bonding at the interface [13] .

The RelyX U200 had lower bond strength than the Predicta, putatively explained by the lack of chemical affinity between the enamel surface and the cement, the self-adhesive cement contains hybrids of resin monomers [18]. Whilst, as the enamel surface lacks the methacrylate groups, there is no chemical affinity between the RelyX U200 luting agent and enamel surface. However, the use of bonding agent with 10-MDP provides copious methacrylate sites and a well-penetrated hybrid layer, which the self-adhesive resin cement can copolymerize with, effectively turning the self-adhesive cement into a more conventional bonded resin system [12, 13, 19].

Amongst the three tested luting agents, the light-cure resin cement (Variolink Esthetic LC) exhibited the lowest shear bond strength (SBS) values, especially in the unbonded subgroups. This could be attributed to its reliance on sufficient light energy for complete polymerization, and given the 2 mm thickness of the lithium disilicate veneers used, a material known to attenuate light transmission[1], it is possible that reduced polymerization occurred at the cement interface [20]. Other factors must also be considered; for example, the absence of an adhesive bonding agent in certain subgroups may have limited the formation of a proper hybrid layer and resin tag penetration, thereby weakening the bond strength. Previous studies suggested avoiding the use light-cured cements under such restorations [22].

Conclusion

Predicta Bioactive Cement delivered the greatest shear-bond strength, a result that was most pronounced when the cement was paired with the 10-MDP-containing universal adhesive, supporting the premise that ionic exchange and bioactivity enhance interfacial durability. In contrast, Variolink Esthetic LC yielded the lowest bond values when no adhesive was applied, a result that can be attributed to limited light transmission through thicker or more opaque veneers. RelyX U200 produced intermediate bond strength that improved markedly with the use of an adhesive, underscoring the benefit of an additional bonding step on enamel substrates. Micro-CT analysis corroborated these mechanical findings, revealing valid results of internal adaptation across all groups but noticeably smaller marginal gaps when an adhesive was employed, particularly in the Predicta and RelyX U200 specimens. Failure-mode evaluation echoed the quantitative results: cohesive fractures predominated in groups exhibiting stronger bonds, whereas adhesive failures were more frequent in unbonded specimens, highlighting the correlation between interfacial integrity and fracture pattern.

Conflict of interest

The authors have no conflicts of interest to declare.

Author contributions

MJM Conceptualization, Data curation, Formal analysis, Finding Acquisition, Investigation, Investigation, Resources, Software, Validation, Visualization, Writing- original draft, Writing- review & editing. AEW Conceptualization, Investigation, Methodology, Project administration, Supervision, Visualization. DJM Conceptualization, Investigation, Methodology, Project administration, Supervision, Visualization, Writing, Writing- review & editing.

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Informed consent

Informed consent was obtained from all individuals or their guardians included in this study.

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القوة الرابطة القصوى والتكيف الداخلي لعدسات الليثيوم ديسليكات المثبتة على سطح المينا باستخدام ثلاثة أنواع من مواد التثبيت: دراسة مخبرية

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المستخلص:
تعتمد عدسات الليثيوم ديسليكات على قوة الربط بين الراتنج والسيراميك والتكيف الحدي الدقيق لضمان النجاح على المدى الطويل، وقد تعزز المواد اللاصقة الشاملة التي تحتوي على 10-MDP أداء مواد التثبيت الراتنجية الحديثة، إلا أن الأدلة حول تأثيرها على المواد اللاصقة الحيوية ومواد التثبيت الذاتية الالتصاق لا تزال محدودة. في هذه الدراسة، تم تثبيت عدسات IPS e.max CAD على سطح المينا باستخدام ثلاثة أنواع من مواد التثبيت الراتنجية: مادة تثبيت حيوية ثنائية التصلب (Predicta)، مادة تثبيت ضوئية التصلب (Variolink Esthetic LC)، ومادة تثبيت ذاتية الالتصاق (RelyX U200)، مع أو بدون استخدام مادة لاصقة شاملة. بعد التثبيت، خضعت العينات لدورات حرارية عددها 5,000 مرة بين 5 °C و55 °C، ثم تم قياس القوة الرابطة القصوى باستخدام جهاز اختبار عالمي، وتحليل أنماط الفشل، وقياس الفجوات الحدّية والداخلية باستخدام التصوير المقطعي المجهري (micro-CT). أظهرت مادة Predicta أعلى قوة رابطة قصوى في كل من المجموعات المربوطة وغير المربوطة، بينما سجلت Variolink LC أقل القيم، خاصة في المجموعات غير المربوطة. أظهرت RelyX U200 قوة متوسطة ازدادت بشكل ملحوظ مع استخدام المادة اللاصقة. أظهر التصوير المقطعي المجهري فجوات أقل لمادة RelyX U200 بشكل عام، بينما تحسن تكيف

في حين تحولت RelyX U200 من كسور تماسكّية إلى لاصقة عند استخدام المادة اللاصقة، أظهر تحليل الفشل كسوراً تماسكّية بشكل رئيسي لمادتي Predicta وVariolink، Predicta بشكل كبير عند الربط. تشير النتائج إلى أن تطبيق المادة اللاصقة الشاملة يحسن بشكل كبير كل من القوة الرابطة والتكيف الداخلي لمواد التثبيت الراتنجية، خاصة المادة الحيوية Predicta والمادة الذاتية الالتصاق RelyX U200، مما يجعل الجمع بين المادة الحيوية والمادة اللاصقة المحتوية على MDP-10 خيارًا مفضلاً سريريًا لعدسات اللثيوم ديسليكات.