

Article



Effect of Self-Adhesive Resin Cement Film Thickness on the Shear Bond Strength of Lithium Disilicate Ceramic–Cement–Tooth Triplex

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Abstract: Cement film thickness may have an impact on the shear bond strength (SBS) of lithium disilicate dental ceramics luted to human enamel with resin cement. The objective of this study was to evaluate SBS of lithium disilicate ceramics adhered to enamel using resin cement at different thicknesses. In total, 50 ceramic specimens ($3 \times 3 \times 3$ mm) and 50 premolar teeth were prepared and randomly assigned to 5 groups (n = 10 each). Ceramic specimens were designed with five cement film thicknesses (50 µm; 100 µm; 150 µm; 200 µm; and 300 µm). Teeth surfaces (4×4 mm) were prepared with a high-speed handpiece mounted on a dental surveyor. Ceramic specimens were cemented to teeth with resin cement ($3M^{TM}$ RelyXTM U200, Resin-Self-Adhesive-Cement). The specimens were then thermocycled for 6000 cycles with a 30 s dwell time and a 5 s transfer time in water ($5 \circ C$ and $55 \circ C$). A Universal-Testing-Machine was used to measure SBS (MPa). Statistical analysis in SPSS included Anova and Tukey's tests. The SBSs of ceramics adhered to teeth revealed significantly different values across all test groups (p = 0.000). The findings showed that as cement layer thickness increased, so did the SBS. The cement spaces at 50 and 300 µm had the lowest SBS (9.40 + 1.15 MPa) and maximum SBS (21.98 + 1.27 MPa), respectively. The SBS of the lithium disilicate ceramic luted to natural human enamel increased along with the cement layer thickness.

Keywords: shear bond strength; cementation; dental ceramics; lithium disilicate; resin cement; ceramic veneers

1. Introduction

Several dental ceramics have lately been refined in terms of mechanical and esthetic features for use in restoring ceramic crowns and laminate veneers at varying thicknesses and translucencies [1]. Due to the ongoing advances in their mechanical qualities brought on by superior microstructures and new processing techniques, which are reflected in the high lifetime of such restorations, glass-ceramics are now widely employed in prosthetic dentistry [2]. Because they combine the advantages of indirect restorations, such as strength, longevity, biocompatibility, esthetics, and treatment predictability, lithium disilicate ceramics have grown to dominate among these materials and, therefore, has frequently been utilized for dental restorations like crowns, bridges, and veneers [3].

The chemical compound lithium disilicate $(Li_2Si_2O_5)$ is a glass ceramic that closely imitates the appearance of real human teeth owing to its excellent optical properties such as absorptivity, transmission, crystallinity, and refraction and reflection of light [4,5]. The unique microstructure of $Li_2Si_2O_5$ is made up of several randomly orientated, tiny,



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Copyright: © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). interconnecting, needle-like plate crystals of 3–6 μ m in length [6]. This microstructure results in cracks being deflected, blunted, and/or branching, which inhibits cracks from developing and significantly affects the material's optical and physical properties [3,7]. Additionally, these ceramics have superior physical and mechanical capabilities which are primarily brought about by the reduction in size of the platelet-shaped crystals (length range from 1.5 to 3 μ m) and the increase in crystal interlocking [2,8].

Computer-aided design/computer-aided manufacturing (CAD–CAM) technology is crucial for the planning and production of all ceramic restorations, especially ceramic laminate veneers, due to its simplicity, speed, and precision [8]. The CAD is utilized for the customized designing of the dental restoration using special softwares; thereafter, the virtual designing is sent to the CAM milling machine for processing the ceramic blocks according to the virtually designed dental restoration [8]. During processing, the lithium disilicate undergoes a two-stage crystallization process producing the definitive restoration with a fine-grain glass ceramic containing 70% crystal volume lithium disilicate, $Li_2Si_2O_5$, with an approximate crystal size of 1.5 µm incorporated in a glass matrix which gives the restoration its mechanical and esthetic properties such as its high strength and its range of translucencies and colors [4–8].

CAD–CAM technology now enables specialists to digitally configure the cement space using software settings in a variety of materials [9]. Various settings have been reported to influence the ultimate quality of cemented ceramic restorations [2,9,10].

Durability is one of the most important aspects of any dental treatment, and proper ceramic veneer cementation is necessary for long-term durability and success. During the cementation process, the most recommended protocol for luting a ceramic restoration is to use resin-based cements in conjunction with adhesive procedures [11]. This suggestion aims to form a clinically durable link between resin cement and tooth tissues as well as between resin cement and ceramic restoration [12]. Various types of resin cements are available and are generally categorized based on the application protocols: total etch and rinse systems (conventional), self-etch systems, and self-adhesive systems (all-in-one cement systems) [13]. To minimize the steps of cementation and simplify the clinical procedures, self-adhesive resin cements are preferred. Besides simplicity, moisture tolerance, dimensional stability, and a rapid setting time, self-adhesive cements rarely display postoperative sensitivity and usually provide good esthetics [13]. They also have the potential for mechanical and chemical bonding and function by strengthening the ceramic as well as the tooth surface especially with veneer restorations [12,14]. Contrarily, a crucial element affecting the success and longevity of adhesively cemented ceramic restorations is the vertical mismatch, or cement thickness, between the restoration and the tooth preparation [12]. It has been documented in the literature that the resin cement layer thickness for ceramic crowns should be in the range of 50–100 μ m [11–14]. Increasing the cement layer thickness to 450 and 500 µm has been associated with residual stresses that severely impaired bonding characteristics [14].

Debonding of porcelain veneers, among other factors, has been identified as a cause of restoration failure over the years. The outcomes of investigations conducted in vitro and in vivo showed that the cement layer thickness can influence binding strength and adhesive contact behaviour [15]. The thickness of the cement layer has been connected to the fracture resistance of adhesively cemented ceramic restorations [15]. In order to accommodate the luting resin cement, which ensures sufficient hydrostatic pressure and promotes cement flow during cementation, there must be clearance between the tooth surface and the veneer [16]. Furthermore, as stated by Pilo et al., the cement escape pathway between the crown restoration is exactly matched to the prepared tooth surface leading to the development of early occlusal connections, insufficient proximal contacts, marginal inconsistencies, and a lack of coziness [17]. This highlights the fact that prosthetic ceramic strength is multimodal by incorporating a ceramic–cement–tooth substrate triplex

and not just a function of the material [12–15]. Being an integral part of the triplex, cement thickness is crucial to the endurance of a restoration.

Although the adhesive capacity of veneer resin cements has received a lot of attention, few of these studies considered cement thickness in this specific setting [12–16,18–20]. The impact of cement gap/space on the adhesive strength of dental ceramics to human enamel has only been examined in a small number of investigations. Determining the shear bond strength of lithium disilicate ceramics adhered to human enamel using five various cement film thicknesses of self-adhesive resin cement was the aim of this in vitro study. The null hypothesis stated that the bonding strength of the luted ceramic specimens will be unaffected by the thickness of the cement layer.

2. Results

2.1. Shear Bond Strength

The shear bond strength of lithium disilicate ceramics adhered to human enamel with five different cement film thicknesses was examined in the current in vitro study. With an ANOVA, the shear bond strength of the ceramic that was adhered to the test subjects' teeth revealed significantly different values across all test groups (p = 0.000). The findings showed that as cement layer thickness increased, so did the shear bond strength. The lowest (9.40 \pm 1.15 MPa) and highest (21.98 \pm 1.27 MPa) shear bond strengths were recorded for the 50 µm and 300 µm cement spaces, respectively. The complete descriptive statistics are presented in Table 1.

Table 1. Descriptive statistics and one-way analysis of variance (ANOVA) for shear bond strength values of tested specimens.

Cement Space (µm)	Mean (MPa)	Std. Deviation	Std. Error	95% Confidence Interval for Mean		Minimum	Maximum	* Anova <i>p-</i> Value
				Lower Bound	Upper Bound			
50	9.40	1.15	0.36	8.58	10.23	8.06	10.64	
100	12.86	1.86	0.58	11.53	14.19	10.81	15.52	
150	14.22	1.70	0.53	13.00	15.44	12.38	16.73	0.000
200	16.70	1.43	0.45	15.67	17.72	15.26	18.97	
300	21.98	1.27	0.40	21.08	22.89	20.07	23.51	

* With p < 0.05, the p value became significant.

Table 2 compares the shear bond strength results for the five groups of tested cement film thickness on a one-to-one basis using the Post Hoc Tukey's HSD test. The test findings revealed substantial variations in shear bond strength (p < 0.05) between all groups, except for the cement gap from 100 µm to 150 µm (p = 0.270).

Table 2. Multiple comparisons of mean difference values with Post Hoc Tukey's HSD test.

Cement Space	50	100	150	200	300
50	-	-3.45 *	-4.81 *	-7.29 *	-12.57 *
100	3.45 *	-	-1.36	-3.83 *	-9.12 *
150	4.81 *	1.36	-	-2.47 *	-7.76 *
200	7.29 *	3.83 *	2.47 *	-	-5.28 *
300	12.57 *	9.12 *	7.76 *	5.28 *	-

* Mean difference values, significant with p < 0.05.

The groups (50–300 μ m) and (100–150 μ m) were found to have the largest (12.57) and lowest (1.36) mean differences for the shear bond strength, respectively (Table 2).

2.2. Digital Microscopic Evaluation

According to the examination of failure modes (Figure 1), 82% of the failures occurred at the cement–ceramic contact, with some specimens experiencing mixed failures (18%). The cement and tooth connection did not experience any failure.



Figure 1. (**a**): De-bonded tooth surface image for adhesive failure; (**b**): De-bonded ceramic surface image for adhesive failure at ceramic–luting cement junction; (**c**): De-bonded tooth surface image for mixed failure; (**d**): De-bonded ceramic surface image for mixed failure.

3. Discussion

The current in vitro investigation evaluated the shear bond strength of lithium disilicate ceramics cemented to natural human enamel at five different cement gap/film thicknesses. Using a distinguished CAD/CAM ceramic model, it was demonstrated that the binding strength of the lithium disilicate ceramic to tooth enamel varied significantly between the five different cement gaps tested. The cement layer thickness has influenced the bond strength and adhesive contact behavior, according to the current in vitro finding. Thus, the null hypothesis was rejected. Despite the fact that the impact of cementation on bond strength has been researched previously [10-16], the current study implemented a distinctive digitally designed standardized model to control cement film thickness that represents various clinical scenarios of different cement spaces. This unique model incorporated digitally designed vertical stops with five different heights (50 μm; 100 μm; 150 μm; 200 μ m; and 300 μ m) for standardized cement space, which allowed for a controllable and reproducible cementation procedure, while maintaining the other factors at a constant. In addition, to simulate oral conditions, the present laboratory settings incorporated thermal cycling for all test specimens since aging affects the bonding characteristics of dental ceramic restorations to the tooth enamel [21]. Moreover, the bond strength has been measured in present work using the shear bond test, which is a reliable test that has been successfully applied in other studies to measure the binding strength of ceramics adhered to enamel [22-24].

A key factor in the clinical effectiveness of these ceramic restorations is a stronger contact between the two components, tooth enamel and dental restoration. To accomplish this, a luting/cementing material that is adherent to both human enamel and ceramic in quality is necessary [25]. Therefore, adhesive resin cements are the material of choice for luting ceramic restorations since these biomaterials depend on adhesion not only for retention but also for resistance [25,26]. The Rely X U200 Automix employed in this study is a self-adhesive resin cement that is made up of methacrylate monomers that contain phosphoric acid groups, which are known to act as crosslinkers in adhesive systems. These multipurpose phosphoric acid methacrylates dissociate in an aqueous solution into methacrylate and acidic phosphoric acid, resulting in a high bond strength between the polymeric cements and the tooth. They demineralize and infiltrate the tooth surface and provide micromechanical retention with the hydroxyapatite of the hard tissues [27]. On the other hand, applying silane coupling agents to the restoration's intaglio surface improves the binding between the resin cement and the inorganic ceramic material. In order to promote the adhesion between the two dissimilar materials, silane coupling agents will provide an interphase zone, which is the region between the organic polymers of the cement and the inorganic lithium disilicate. This is made easier by the fact that the silane coupling agents have inorganic reactive groups on silicon, which are known to attach well to the majority of inorganic ceramic substrates, particularly if those substrates contain silicon, such as lithium disilicate [28]. The metal hydroxyl groups on the surface of the inorganic ceramic can coordinate with the alkoxy groups on silicon to generate silanols, which then hydrolyze to form an oxane bond. The polymer matrix of the self-adhesive resin cement is capable of forming strong bonds with the organo-functional group and the alkoxy group [27,28]. Besides enhanced bonding to tooth enamel and ceramic substrate, the self-adhesive resin cement Rely X U200 was chosen because it requires simple and fast cementation steps combined with ease of handling.

Without a doubt, the cement film thickness of resin cement is regarded as a key factor for the success of indirect restorations [14,28]. The results of the current research revealed that when the cement film thickness increases, so does the bonding strength between the ceramic and the teeth. In other words, the shear bond strength was directly related to the cement film thickness. Contrarily, using cement thicknesses of 60 μ m, 120 μ m, and 180 μ m, Tribst et al. demonstrated that the thickness of the cement layer did not affect the immediate bond strength of lithium disilicate restorations but inversely influenced the bond strength for the aged specimens tested. Such contradiction could be attributed to the difference of the resin cement type used. The authors have used Variolink II which is a dual cure resin cement containing Bis-GMA as its adhesive monomer while Rely X 200 contain methacrylated phosphoric ester resin groups known to show high hydrophilicity among dental resins [29]. The increased water absorption observed for self-adhesive resin cement, such as the currently used Rely X 200, contribute to the cement hygroscopic expansion especially with thicker cement films after aging. This expansion compensates for the initial polymerization shrinkage, with its lowest stress values present in the thinnest cement layer of just 25 μ m, and increases correspondingly with greater thicknesses (100 μ m, 200 μ m, and 400 µm) [29]. Therefore, using thicker cement layers after simulating aging may have a positive therapeutic impact, producing expansion stress that may influence the closure of the marginal gap and encourage restoration and tooth retention. This logical argument could explain why the bond strength increased with the increase in the cement gap in the current study's results [14,29]. Additionally, the ceramic blocks in the work of Tribst et al. were bonded to dentin and not to enamel as the present study. High failure rates of ceramic veneer have been associated with bonding to exposed dentin surfaces [30]. Moreover, porosities originate in bulk materials while applying pressure during cementation, which is more common in thicker layers of resin cement [29]. These porosities may also be a contributing factor to the decreased binding strength in the group of cement layers with a thicker consistency in the Tribst et al. experiment [14]. The fact that the current study used digitally designed vertical stops for standardized cement film thickness allowed the cement

to flow freely out of the sides of the specimens which in turn avoided any air trapping and reduced the hydrodynamic pressure built up during cement setting.

Earlier research has displayed a connection between bond strength and cement layer thickness [18]. Although an increase in the thickness of the cement layer did not adversely affect the immediate bond strength [14], a thin cement coating is still preferred for improved restoration from the mechanical response, as evidenced by the previously established substantial association between lower ceramic fracture load and thicker cement layer [29]. A thick cement would have more volume of material, and consequently more magnitude of residual stress will be present which will eventually weaken the bond strength after aging [14]. Additionally, a thicker cement increases stress at the walls of the tooth cavity due to exaggerated polymerization shrinkage relative to increased material volume. As a result, the mechanical behavior of adhesively cemented ceramic restorations is greatly influenced by the thickness of the cement layer [14,18,29]. The literature states that during the cementation procedure, the resinous cement should completely cover the space between the restoration and the tooth with no marginal deficiency [31,32]. However, the luting cement type and sitting force utilized can both significantly alter the cement film's thickness [14]. According to the findings, a thinner cement layer of $<100 \mu m$ resulted in weak bonding, whereas a bigger cement layer of $>300 \,\mu\text{m}$ may result in an open margin and cement exposure to the oral environment [14,29]. Therefore, a cement layer of 150–200 μ m could be recommended for resin luting cement gap.

Examination of the de-bonded teeth and ceramic surfaces revealed that the majority of the failures were adhesive failures at the junction of the cement and ceramic specimens. This observation could be due to the fact that all the teeth specimens were sound with intact enamel. Adhesive systems available in the market promote better interaction/bonding of the luting cement with the enamel [33]. As explained earlier, these resin cements demineralize and infiltrate the tooth surface and provide micromechanical retention with the hydroxyapatite of the hard tissues [27] which explains the absence of failure at the cement-tooth interface. Furthermore, the self-adhesive resin cements are hydrophilic materials with increased water sorption, and due to the high amount of acidic phosphate functional monomers, they may be sensitive to hydrolytic breakdown, especially at the resin cement-ceramic interface. Through hydrolytic breakdown, the passage of water molecules into the bonding contact may make the connection less strong [34]. Additionally, during thermocycling procedures, the thermal stress caused by the different coefficients of thermal expansion between adhesive resin cement and lithium disilicate ceramic may have accelerated further hydrolytic degradation of the bonding interface, which could account for the dominant adhesive failure mode observed in the current study at the cement-ceramic interface [35].

The thickness of the cement is one of many other factors that are important for the performance of lithium disilicate frameworks [36], and the current work has displayed the significance of this cementation parameter on the success and durability of these ceramic restorations. However, there are certain limitations in the present work to be addressed. The use of square morphology specimens with flat side surfaces does not represent the real restoration shape in clinical scenarios. A more accurate way to assess the mechanical properties of bond strength may have been to create test specimens in the shape of curved ceramic veneers, which would have mimicked the clinical situation. Another limitation exists due to the fact that the specimens used in the current investigation were tested until failure under constant vertical tension. The frequent changes in temperature and moisture that the intraoral restorations experience affect their mechanical performance. More research with different times/durations of ageing, an inclusion of lap shear, tensile stress, and peel stress testing's, is needed. Moreover, the use of one type of cement limits the expansion of the present findings. Though, an in vitro study that properly models and predicts the clinical event is doubtful. The results of this in vitro experiment should not be directly extrapolated to the clinical performance of restorative materials due to the constraints indicated above. Nevertheless, cement film thickness is one of the crucial but

frequently disregarded cementation features that influences both the short- and long-term bond strength and so jeopardizes the restorative prognosis.

4. Materials and Methods

College of Dentistry Research Center (CDRC), King Saud University (CDRC Reg. # IR 0433), and the institutional review board of King Saud University medical city (IRB Reg. # E-22-7203) gave their consent prior to the study's start. From September 2022 to April 2023, the study was conducted in the Department of Prosthodontics and CDRC, College of Dentistry, King Saud University, Riyadh, Saudi Arabia.

4.1. Sample Size Determination

According to "G*Power 3.1.9.7, Heinrich Heine University Düsseldorf, Germany", the total sample size for determining the shear bond strength of five test groups having five different cement film thicknesses was 50 (n = 10). The parameters used were alpha 0.05, power 0.95, and effect size 0.77 [14].

4.2. Ceramic Specimen Preparation

Fifty specimens ($3 \times 3 \times 3$ mm) of all ceramic lithium disilicate (Ivoclar Vivadent IPS E.max CAD) (Table 3) were fabricated using CAD/CAM technology following the manufacturer's instructions. Exocad's Dental CAD TruSmile Module was used to design the ceramic blocks with vertical stops placed at the corners of the cubic block with different heights (Figure 2) to reflect the desired different cement film thicknesses creating five groups as follows (n = 10):

Group 1: with 50 μ m cement film thickness. Group 2: with 100 μ m cement film thickness. Group 3: with 150 μ m cement film thickness. Group 4: with 200 μ m cement film thickness. Group 5: with 300 μ m cement film thickness.

Table 3. Details of the materials used in the research study.

Material	Trade Name	Manufacturer	Composition	Lot Number
Lithium- disilicate	IPS, Emax, CAD	Ivoclar, Vivadent	quartz, lithium dioxide, phosphor oxide, alumina, potassium oxide, and other components	yB54FS
Self-adhesive resin cement	RelyX™ U200	3M TM	Methacrylate monomers containing phosphoric acid groups, methacrylate monomers, alkaline fillers, silanated fillers, zirconia/silica fillers, initiators, stabilizers, rheological additives.	UPC 4035077033213
Silane Coupling Agent	RelyX™ Ceramic Primer Silane Coupling Agent	3M TM	Ethanol, water, methacryloxypropyltrimthoxysilane	UPC 30605861033191

After software designing, glass ingots of $Li_2Si_2O_5$ were then digitally machined using diamond milling burs within the enclosed programmed milling machine (Diamond RFID, Amann Girrbach) producing the desired shape of ceramic blocks which were then fired within the furnace (Ivoclar Vivadent Furnace) at 850 °C resulting in the final E.max restoration.



Figure 2. Computer-aided designed ceramic block with cement space controlled by varying heights of vertical stops (50 μ m; 100 μ m; 150 μ m; 200 μ m; 300 μ m) incorporated in the design for standardizing the cement space.

4.3. Preparation of Teeth Specimens

Fifty recently extracted, caries-free, premolar teeth of similar size from humans were used in this study. Young adults' teeth were extracted for orthodontic reasons. The teeth were sterilized with 5.25% NaOCl solution, washed with an ultrasonic cleaner, placed in a container with 0.5% Chloramine T antimicrobial preservative (Delchimica Scientific Glassware, Naples, Italy), and used within eight weeks of extraction.

The anatomic crown and 2 mm of coronal root section were exposed when the teeth were placed in 2 cm diameter auto-polymerizing acrylic resin blocks (Ortho-Resin, Degu-Dent GmbH, Hanau Hessen, Germany). The dental surveyor (J. M. Ney Company, Hartford, Connecticut, USA) was equipped with a high-speed hand piece to prepare the teeth surfaces (4×4 mm) for standardization. After just removing 0.5 mm of enamel, a wheel diamond bur was used to create a clean and level 4×4 mm tooth surface on the buccal surface of the teeth (Figure 3). The 50 mounted prepared premolar teeth were randomly distributed into 5 groups based on the cement film thickness that will be utilized for luting the ceramic blocks.



Figure 3. Tooth specimen mounted in resin block with ceramic block cemented to the buccal surface of the tooth.

4.4. Cementation of the Ceramic Specimens to the Teeth

Utilizing self-adhesive resin cement (RelyXTM U200 Automix Self-Adhesive Resin Cement), the lithium disilicate ceramic specimens were luted to the prepared tooth. The intaglio surface of the ceramic blocks was etched with 5% hydrofluoric acid (IPS Ceramic Etching Gel, Ivoclar, Vivadent, Liechtenstein) for 1 min prior to starting the automix syringe. As directed by the manufacturer, silane coupling agent was then applied using a micro brush. The self-adhesive resin cement does not require any surface preparation of the tooth. The automixed cement had been applied to the ceramic bonding surface with the syringe's tip, and the ceramic block was finger pressed and bonded to the prepared tooth surface. Before being exposed to a high-intensity (~1200 mW/cm²), LED light-curing unit for 40 s (Bluephase G2, Ivoclar Vivadent, Schaan, Liechtenstein), any surplus was removed with a micro brush (Figure 3).

4.5. Thermocycling of Specimens

All the samples were kept in distilled water at 37 °C for 24 h to mimic the clinical setting. They were then thermocycled (Huber, SD Mechatronik Thermocycler, Germany) for 6000 cycles with a 30 s dwell time and a 5 s transfer time in water (5 °C and 55 °C).

4.6. Mounting and Testing

All specimens were set up in the Universal Testing Machine (Instron, model 8500 Plus; Dynamic Testing System; Instron Corp.), and the angulation was changed so that the ceramic block was parallel to the beveled arm (Figure 4A,B). All specimens were tested using UTM with a beveled arm at a crosshead speed of 0.5 mm/min, and the maximum force required to unseat each ceramic block was noted until failure in order to compute shear bond strength. A constant loading rate of 0.5 mm/min was employed until failure. The bond failure point was defined as the point at which the ceramic block that was adhered to the enamel de-bonded with the highest force recorded. The strength of the shear bond was measured in megapascals (MPa).





Figure 4. (**A**): Application of load for shear bond testing in universal testing machine; (**B**): A diagrammatic representation of a universal testing machine's shear bond test.

4.7. Digital Microscopic Examination

The De-bonded samples were inspected by two blinded examiners under a digital microscope (HIROX, KH-7700, Digital microscope system, Tokyo, Japan) at a magnification

of $50 \times$ to study the surface topography of the teeth and ceramic surfaces after debonding of the ceramic specimens luted to the teeth specimens. The surfaces were evaluated for the types of failure modes, i.e., adhesive failure (de-bonding at the cement–ceramic or cement–tooth interface), cohesive failure (de-bonding because of the failure within the cement), or mixed failure (adhesive failure + cohesive failure).

4.8. Statistical Analysis

The statistical program for social sciences (SPSS, version 22, Chicago, IL, USA) was used for the statistical analysis. For each of the five test groups of cement film thicknesses, shear bond strengths were averaged, and their standard deviations calculated. The Shapiro–Wilk test was used to determine the data's normality, and it was found to be normally distributed. One-way ANOVA and the Post Hoc Tukey's HSD test were used to evaluate the data. Descriptive statistics for the failure mode data were presented as percentages. $p \leq 0.05$ was chosen as the significance value.

5. Conclusions

Within the limitations of the current study, the following conclusions could be drawn:

- 1. Varying the thickness of resin cement affects the shear bond strength of the lithiumdisilicate ceramic luted to natural human enamel.
- 2. As the cement layer thickness increased, so did the shear bond strength of the lithium disilicate ceramic luted to natural human enamel.
- 3. The failure mode was predominantly adhesive failure at the cement–ceramic interface.

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References

- 1. Moshaverinia, A. Review of the modern dental ceramic restorative materials for esthetic dentistry in the minimally invasive age. *Dent. Clin. N. Am.* **2020**, *64*, 621–631. [CrossRef]
- Rexhepi, I.; Santilli, M.; D'Addazio, G.; Tafuri, G.; Manciocchi, E.; Caputi, S.; Sinjari, B. Clinical applications and mechanical properties of cad-cam materials in restorative and prosthetic dentistry: A systematic review. J. Funct. Biomater. 2023, 14, 431. [CrossRef]
- 3. Aslan, Y.U.; Uludamar, A.; Özkan, Y. Retrospective analysis of lithium disilicate laminate veneers applied by experienced dentists: 10-year results. *Int. J. Prosthodont.* **2019**, *32*, 471–474. [CrossRef]
- 4. Kaur, K.; Talibi, M.; Parmar, H. Do you know your ceramics? Part 3: Lithium disilicate. Br. Dent. J. 2022, 232, 147–150. [CrossRef]
- 5. Zarone, F.; Di Mauro, M.I.; Ausiello, P.; Ruggiero, G.; Sorrentino, R. Current status on lithium disilicate and zirconia: A narrative review. *BMC Oral Health* **2019**, *19*, 134. [CrossRef]

- Mavriqi, L.; Valente, F.; Murmura, G.; Sinjari, B.; Macrì, M.; Trubiani, O.; Caputi, S.; Traini, T. Lithium disilicate and zirconia reinforced lithium silicate glass-ceramics for CAD/CAM dental restorations: Biocompatibility, mechanical and microstructural properties after crystallization. J. Dent. 2022, 119, 104054. [CrossRef]
- 7. Fu, L.; Engqvist, H.; Xia, W. Glass-ceramics in dentistry: A review. Materials 2020, 13, 1049. [CrossRef]
- 8. Lien, W.; Roberts, H.W.; Platt, J.A.; Vandewalle, K.S.; Hill, T.J.; Chu, T.M. Microstructural evolution and physical behavior of a lithium disilicate glass-ceramic. *Dent. Mater.* **2015**, *31*, 928–940.
- 9. Spitznagel, F.A.; Boldt, J. Gierthmuehlen PCCAD/CAM ceramic restorative materials for natural teeth. *J. Dent. Res.* 2018, 97, 1082–1091. [CrossRef]
- Li, R.W.; Chow, T.W.; Matinlinna, J.P. Ceramic dental biomaterials and CAD/CAM technology: State of the art. J. Prosthodont. Res. 2014, 58, 208–216. [CrossRef]
- 11. O'Connor, C.; Gavriil, D. Predictable bonding of adhesive indirect restorations: Factors for success. *Br. Dent. J.* **2021**, 231, 287–293. [CrossRef]
- 12. Gundogdu, M.; Aladag, L.I. Effect of adhesive resin cements on bond strength of ceramic core materials to dentin. *Niger. J. Clin. Pract.* **2018**, *21*, 367–374. [CrossRef]
- 13. Ghodsi, S.; Shekarian, M.; Aghamohseni, M.M.; Rasaeipour, S.; Arzani, S. Resin cement selection for different types of fixed partial coverage restorations: A narrative systematic review. *Clin. Exp. Dent. Res.* **2023**, *9*, 1096–1111. [CrossRef]
- 14. Tribst, J.P.M.; Dos Santos, A.F.C.; da Cruz Santos, G.; da Silva Leite, L.S.; Lozada, J.C.; Silva-Concílio, L.R.; Baroudi, K.; Amaral, M. Effect of cement layer thickness on the immediate and long-term bond strength and residual stress between lithium disilicate glass-ceramic and human dentin. *Materials* **2021**, *14*, 5153. [CrossRef]
- 15. Alenezi, A.; Alsweed, M.; Alsidrani, S.; Chrcanovic, B.R. Long-term survival and complication rates of porcelain laminate veneers in clinical studies: A systematic review. *J. Clin. Med.* **2021**, *10*, 1074. [CrossRef]
- 16. Rojpaibool, T.; Leevailoj, C. Fracture resistance of lithium disilicate ceramics bonded to enamel or dentin using different resin cement types and film thicknesses. *J. Prosthodont.* **2017**, *26*, 141–149. [CrossRef]
- 17. Pilo, R.; Cardash, H.S.; Baharav, H.; Helft, M. Incomplete seating of cemented crowns: A literature review. *J. Prosthet. Dent.* **1988**, 59, 429–433. [CrossRef]
- 18. Arcila, L.V.C.; Gomes, L.C.L.; Ortiz, L.P.N.; Costa, M.M.D.; Tribst, J.P.M.; Bottino, M.A.; Saavedra, G.S.F.A.; de Melo, R.M. Effect of resin cement at different thicknesses on the fatigue shear bond strength to leucite ceramic. *Eur. J. Dent.* **2022**, 19. [CrossRef]
- 19. Aker Sagen, M.; Dahl, J.E.; Matinlinna, J.P.; Tibballs, J.E.; Rønold, H.J. The influence of the resin-based cement layer on ceramicdentin bond strength. *Eur. J. Oral Sci.* 2021, 129, e12791. [CrossRef]
- 20. Tribst, J.P.; Dal Piva, A.M.; Penteado, M.M.; Borges, A.L.; Bottino, M.A. Influence of ceramic material, thickness of restoration and cement layer on stress distribution of occlusal veneers. *Braz. Oral Res.* **2018**, 32. [CrossRef]
- 21. Eliasson, S.T.; Dahl, J.E. Effect of thermal cycling on temperature changes and bond strength in different test specimens. *Biomater. Investig. Dent.* **2020**, *7*, 16–24. [CrossRef]
- 22. El Mourad, A.M. Assessment of bonding effectiveness of adhesive materials to tooth structure using bond strength test methods: A review of literature. *Open Dent. J.* **2018**, *12*, 664–678. [CrossRef]
- Aker Sagen, M.; Vos, L.; Dahl, J.E.; Rønold, H.J. Shear bond strength of resin bonded zirconia and lithium disilicate—Effect of surface treatment of ceramics and dentin. *Biomater. Investig. Dent.* 2022, 9, 10–19. [CrossRef]
- 24. Zhu, J.; Gao, J.; Jia, L.; Tan, X.; Xie, C.; Yu, H. Shear bond strength of ceramic laminate veneers to finishing surfaces with different percentages of preserved enamel under a digital guided method. *BMC Oral Health* **2022**, 22, 3. [CrossRef]
- 25. Calheiros-Lobo, M.J.; Vieira, T.; Carbas, R.; da Silva, L.F.M.; Pinho, T. Effectiveness of self-adhesive resin luting cement in cad-cam blocks—A systematic review and meta-analysis. *Materials* **2023**, *16*, 2996. [CrossRef]
- Blatz, M.B.; Vonderheide, M.; Conejo, J. The effect of resin bonding on long-term success of high-strength ceramics. J. Dent. Res. 2018, 97, 132–139. [CrossRef]
- 27. Rodrigues, R.F.; Ramos, C.M.; Francisconi, P.A.; Borges, A.F. The shear bond strength of self-adhesive resin cements to dentin and enamel: An in vitro study. *J. Prosthet. Dent.* 2015, *113*, 220–227. [CrossRef]
- Pape, P.G. Adhesion promoters: Silane coupling agents. In *Applied Plastics Engineering Handbook*; William Andrew Publishing: Norwich, NY, USA, 2011; Volume 1, pp. 503–517.
- 29. Sokolowski, G.; Krasowski, M.; Szczesio-Wlodarczyk, A.; Konieczny, B.; Sokolowski, J.; Bociong, K. The influence of cement layer thickness on the stress state of metal inlay restorations-photoelastic analysis. *Materials* **2021**, *14*, 599. [CrossRef]
- Gonzaga, C.C.; Bravo, R.P.; Pavelski, T.V.; Garcia, P.P.; Correr, G.M.; Leonardi, D.P.; Cunha, L.F.; Furuse, A.Y. Enamel and dentin surface finishing influence on the roughness and microshear bond strength of a lithium silicate glass-ceramic for laminate veneers. *Int. Sch. Res. Not.* 2015, 2015, 243615. [CrossRef]
- Samy, N.; Al-Zordk, W.; Elsherbini, A.; Özcan, M.; Sakrana, A.A. Does resin cement type and cement preheating influence the marginal and internal fit of lithium disilicate single crowns? *Materials* 2022, 15, 424. [CrossRef]
- 32. Ghodsi, S.; Arzani, S.; Shekarian, M.; Aghamohseni, M. Cement selection criteria for full coverage restorations: A comprehensive review of literature. *J. Clin. Exp. Dent.* **2021**, *13*, e1154–e1161. [CrossRef]
- Naumova, E.A.; Ernst, S.; Schaper, K.; Arnold, W.H.; Piwowarczyk, A. Adhesion of different resin cements to enamel and dentin. Dent. Mater. J. 2016, 35, 345–352. [CrossRef]

- 35. Alrabeah, G.; Alomar, S.; Almutairi, A.; Alali, H.; ArRejaie, A. Analysis of the effect of thermocycling on bonding cements to zirconia. *Saudi Dent. J.* **2023**, *35*, 734–740. [CrossRef]
- 36. Comba, A.; Baldi, A.; Carossa, M.; Michelotto Tempesta, R.; Garino, E.; Llubani, X.; Rozzi, D.; Mikonis, J.; Paolone, G.; Scotti, N. Post-fatigue fracture resistance of lithium disilicate and polymer-infiltrated ceramic network indirect restorations over endodontically-treated molars with different preparation designs: An in-vitro study. *Polymers* 2022, 14, 5084. [CrossRef]

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