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Comparative analysis of fracture resistance and film thickness between flowable composites and resin cement luted lithium disilicate ceramics

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Objectives: This study investigated the fracture resistance and film thickness of the different composite cements that can be used with lithium disilicate-based ceramic restoration (IPS e.max CAD).

Materials and Methods: Twenty-five (25) IPS e.max CAD discs (A2, shade HT/C14) with a 10 mm diameter and 1 mm thickness were randomly assigned to five experimental groups (n = 5) according to luting agent G-ænial™ Universal Injectable (GC Corporation, Japan), Clearfil™ AP-X Esthetics FLOW (Kuraray Noritake, Japan), Beautifil injectable X (Shofu Inc., Japan), and Filtek™ Supreme Flowable (3M Oral Care, USA). Flowable composites were bonded between the ceramic disc and dentin. The control group sample was dual-cured resin cement, Multilink N (Ivoclar Vivadent, Liechtenstein). A universal testing machine (Model LR10K; Lloyd Instruments, Fareham, UK) was used to conduct a three-point bending test to determine the fracture resistance. The film thickness was analyzed using scanning electron microscopy (SEM, JSM 6610LV, JEOL, Peabody, PA, USA). The data were analyzed using a one-way ANOVA.

Results: Flowable composites presented fracture resistance values comparable to the resin cement, with acceptable film thickness values meeting ISO requirements, except for Filtek™ Supreme Flowable. However, no statistically significant differences were found among groups ($p>0.05$)

Conclusions: Flowable composites could be potential alternatives for ceramic luting, although further studies are needed to confirm their long-term clinical performance.

Keywords: ceramic, composite cement, film thickness, fracture resistance

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Introduction

A luting cement is a substance that is employed to secure indirect restorations to prepared tooth surfaces by filling minute cavities between the restorations and the tooth structures. This mechanically locks the restoration in place to prevent dislodgment [1, 2]. Resin cements are composite materials that have distinct chemical compositions. They are composed of a resin matrix (e.g., Bis-GMA or urethane dimethacrylate) and fine particles of inorganic fillers. Initially, they are distinguished from restorative composites by their low filler content (50–70%wt glass or silicon dioxide) and viscosity. The filler concentration in the resin cement is reduced to accommodate a thin film thickness and a longer working time [3].

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In addition, the mechanical properties are correlated with the quantity of filler; the mechanical strength decreases as the number of fillers decreases [4-6].

The increasing interest in using flowable composites as adhesive luting is to benefit from their physical properties; being more filler-loaded than resin cements, and their improved cost benefits compared to resin cements [7]. Most recently, highly filled flowable composites, including 65-75% fillers by weight, have been introduced for direct restorations and indirect cementation. Besides maintaining low viscosity, these materials have relatively comparable mechanical and optical properties with paste-type composites[8]. For light-curing resin cements, the light transmission rate is influenced by the thickness of the restoration; hence, these cements are recommended for bonding translucent restorations with thicknesses of less than 2 mm [9, 10]. Consequently, flowable composites may exhibit mechanical and optical properties similar to those of resin cements, particularly in terms of viscosity, filler content, and clinical handling characteristics.

A novel type of highly filled flowable composite has recently been developed—for example, G-aenial Universal Injectable (GC, Tokyo, Japan). It is distinguished by its high viscosity and is purported to have improved mechanical properties that are comparable to those from conventional composite restorative materials [11, 12]. In contrast to traditional paste-type composites, the highly filled flowable resin contains nano-sized fillers. The surface of the resin has been modified to reduce its viscosity for placement, thereby enabling the composite to be used in load-bearing restorations [13, 14]. On the other hand, Clearfil™ AP-X Esthetics FLOW (CF, Kuraray, Osaka, Japan) had a high filler content (75 wt% or 59% volume). Additionally, CF was reported to to provide superior mechanical properties making it suitable even for posterior restorations.

Fracture resistance is a fundamental mechanical property that indicates a material's capacity to endure functional loads without experiencing catastrophic failure. In dentistry, this is especially crucial for brittle materials like ceramics, which are commonly used in restorative procedures. The fracture resistance of lithium disilicate ceramics is governed by several factors. One key factor is restoration thickness; an adequate thickness significantly enhances the strength of lithium disilicate restorations [16]. The manufacturing technique also plays a vital role. CAD/CAM-fabricated restorations have demonstrated superior fracture resistance compared to pressable techniques, even when using the same material [17]. Restoration design further influences stress distribution. For instance, onlays made from IPS e.max CAD exhibited higher fracture resistance than crowns made from the same material [18]. Tooth preparation design—such as taper angle, margin type, and occlusal reduction—also affects the outcome. Studies have reported that occlusal veneers present lower fracture resistance compared to adhesive crowns [19]. In addition, the mechanical properties of resin cement, including its composition and polymerization characteristics, are important determinants of overall restoration performance. Notably, research has shown that the mean fracture resistance of lithium disilicate anterior crowns varied significantly based on the resin cement used [20], Despite these insights, only a limited number of studies have comprehensively investigated how different flowable composites used as luting cements influence the fracture resistance of ceramic restorations bonded to tooth structures.

In addition, film thickness of the luting agent at the tooth-cement-restoration interface represents a key aspect for a successful treatment prognosis. A thicker film is more prone to wear, leading to consequent marginal misfit [21]. Reduced film thickness has been linked to increased fracture resistance of all ceramic restorations, improved bond strength and low water sorption [22, 23]. As previously mentioned and also according to ISO standard 4049:2019, film thickness has been researched and described as ideal between 5 and 25 μm and in any event it shall exceed 50 μm [24]. Film thickness is closely related to the flowability and viscosity of the material; flowable composites typically demonstrate lower viscosity and better flow characteristics than conventional resin cements, which may allow for thinner and more uniform luting layers. These material properties, including filler content, resin matrix composition, and rheological modifiers, affect both the ease of application and the clinical performance of the luting agent.

Therefore, the null hypothersis of this study were i) there is no significant difference in fracture resistance among the tested composite cements for lithium disilicate-based ceramic restorations. ii) there is no significant difference in film thickness among the tested composite cements for lithium disilicate-based ceramic restorations.

Materials and Methods

Material preparation

1. Sample collection

In this study, twenty-five extracted human third molars without carious lesions, cracks, or restorations on the enamel and dentin surfaces were used and collected under a protocol reviewed and approved by the university ethics committee (COE.No.MU-DT/PY-IRB 2023/059.1912). All collected teeth were stored in 0.1% thymol solution and used within 6 months after extraction. The sample size was calculated from the estimated effect size (95% power and 5% error), resulting in N = 5 (G*Power 3.1).

2. Resin luting cement preparation

The following materials were tested: flowable composites, and resin cements. Their compositions, instructions, and manufacturers are described in Table 1 and 2. The flowable composites and resin cements were used at room temperature and handled according to the manufacturers' instructions.

Table 1 Manufacture, classification, and composition of materials used in this study

Resin-based luting agent	Manufacturer	Type	Monomer composition	Filler content
G-ænial™ Universal Injectable (GI)	GC Corporation, Tokyo,Japan	Highly filled flowable composite	UDMA, bis-GMA, methacrylatemonomers	Filler load 69% wt / 46%vol[1]
Clearfil™ AP-X Esthetics FLOW	Kuraray Noritake, Japan	Highly filled flowable composite	TEGDMA, hydrophobic aromatic dimethacrylate,	Filler load 75% wt / 59% vol

Table 1 Manufacture, classification, and composition of materials used in this study (continued)

Resin-based luting agent	Manufacturer	Type	Monomer composition	Filler content
Beautifil injectable X (BI)	Shofu Inc. Kyoto, Japan	Flowable composite	Bis-GMA, TEGDMA, Bis-MPEPP	Filler load 64% wt / 42%vol
Filtek^{TM} Supreme Flowable (FF)	3M Oral Care,St. Paul, MN, USA	Flowable composite	Procrylat, BisGMA, and TEGDMA resins	Filler load 65% wt / 46%vol
Multilink N (MN)	Ivoclar Vivadent, Schaan, Lichtenstein	Dual cured luting resin cement	dimethacrylate and HEMA (30.5% wt)	Filler load 68.5% wt/ 40% vol.

Table 2 Manufacture of materials used in this study

Material	Type	Manufacturer
Clearfil SE bond	Self-etching adhesives	Kuraray Noritake. Osaka, Japan
Single Bond Universal	Universal adhesive	3M Oral Care,St. Paul, MN, USA
Scotchbond Universal Etchant	Etchant gel	3M Oral Care,St. Paul, MN, USA
Monobond N	Ceramic primer	Ivoclar Vivadent, Schaan, Lichtenstein
IPS Ceramic Etching gel	Hydrofluoric acid	Ivoclar Vivadent, Schaan, Lichtenstein

3. Tooth preparation

The occlusal third of the crown was sectioned using a low-speed diamond saw (Diamond blade 4-inch series HC, PACE, USA) under water cooling. Teeth with a dentin diameter of less than 9 mm were excluded. A smear layer was created by manually finishing the surface with 600-grit silicon carbide (SiC) grinding paper (Buehler, Buehler Ltd, Lake Bluff, Illinois, USA) under running water for 60 seconds.

4. Preparation of lithium disilicate ceramics slice (LDS, IPS e.max CAD)

The IPS e.max CAD HT CAD/CAM blocks (LDS, IPS e.max CAD, Ivoclar Vivadent, Schaan, Liechtenstein; SiO_2, Li_2O, K_2O, P_2O_5, ZrO_2, ZnO, Al_2O_3, MgO, coloring oxides; HT A2/C14) were used. The block dimensions were 18 mm in length, 14.5 mm in width, and 12.4 mm in height. Each block was sectioned into 15 pieces (1 × 10 × 10 mm) by using a low-speed diamond saw (Diamond blade 4-inch series HC, PACE, USA). The ceramic discs were then crystallized in an Ivoclar Vivadent ceramic furnace (Programat® P300) to complete the restoration process. Flat LDS surfaces were prepared by manually grinding with wet 600-grit silicon carbide (SiC) paper 60 s. Then, the blocks were cleaned in an ultrasonic bath (Bandeloin DT-156BH, Germany) of distilled water for 10 minutes to ensure a contaminant-free ceramic surface.

5. Cementation of lithium disilicate discs to the tooth substrate

The cementation was performed in a controlled room at 25°C. The surfaces of all ceramic discs were etched with 4.5% hydrofluoric acid (IPS Ceramic Etching Gel, Ivoclar Vivadent) for 20 seconds, subsequently washed with a spray jet and water for 30 seconds, and then placed in an ultrasonic bath containing distilled water for 5 minutes. Following this, a silane coupling agent (Monobond N, Ivoclar Vivadent) was applied using a size M microbrush (3M Oral Care, St. Paul, MN, USA) and allowed to react for 60 seconds. The remaining excess was dispersed with a strong stream of air for 10 seconds.

Group A; The flowable composite.

Clearfil SE Bond (Kuraray Noritake) primer and bonding were applied on the tooth surface according to the manufacturer's instructions. The light intensity of the curing unit (Bluephase G2, Ivoclar Vivadent, Schaan, Liechtenstein) was verified using a calibrated light meter (bluphase® meter, Ivoclar Vivadent, Liechtenstein) before use. The device was operated at an intensity of approximately 1,000 mW/cm² for 10 seconds. Four groups of flowable composites were used as follow: ***Filtek™ Supreme Flowable*** (**FF**, 3M Oral Care,St. Paul, MN, USA), ***G-ænial™ Universal Injectable*** (**GI**, GC Corporation, Tokyo, Japan), ***Clearfil™ AP-X Esthetics FLOW*** (**CF**, Kuraray Noritake, Japan), ***Beautifil injectable X*** (**BI**, Shofu Inc. Kyoto, Japan). The materials were applied to the intaglio surface of the pretreated ceramics and then seated onto the tooth surface. All specimens were luted by applying a controlled force of 50 N for 3 minutes. The specimens were light-cured for 60 seconds per surface at an intensity of 1,000 mW/cm² using a light-curing unit (Bluephase® G2 LED curing light, Ivoclar Vivadent, Schaan, Liechtenstein).

Group B; resin cement.

On the dentin surface, the mixed Multilink Primer A/B (Ivoclar Vivadent, Schaan, Lichtenstein) was applied to all prepared surfaces using a size M microbrush. The primer was lightly scrubbed into the dentin for 15 seconds. Excess Multilink Primer was dispersed with a strong stream of air until the mobile film disappeared, as the primer was self-curing. The cement was mixed and dispensed onto the intaglio surface of the pretreated disc, and the discs were seated onto the pretreated tooth surface under a constant load of 50 N for 3 minutes. The resin cement was light-cured in the same manner with the flowable composite group.

All the bonded specimens were stored in distilled water for one week at a constant temperature of 37°C.

6. Measurement of fracture resistance

To conduct the fracture resistance test, the stored specimens were trimmed into bar-shaped samples (2 × 2 × 8 mm³; n = 3 per specimen) using a low-speed diamond saw. During this step, the enamel was completely removed. The trimmed specimens from central area (15 per subgroup) were used for the fracture resistance test (10 per subgroup) and the film thickness measurement (5 per subgroup). The three-point bending test was performed using a universal testing machine (Model LR10K; Lloyd Instruments, Fareham, UK) at a crosshead speed of 1 mm/min.

The trimmed specimens, with the ceramic positioned on the top side, were placed in a jig and then loaded until fracture. The jig, consisting of two triangular prisms mounted in parallel with a 5 mm distance between centers and a third prism centered between and parallel to the other two, was used to support the specimens. The load–deflection curves obtained from these tests were carefully examined for any discontinuity to determine whether the ceramic and dentin fractured simultaneously, and the load (N) at fracture was determined.

Fracture resistance values were averaged from two specimens from each tooth and used to represent the fracture resistance for each sample.

7. Measurement of film thickness

Five trimmed specimens per subgroup were used for film thickness measurement. The specimens were polished using abrasive SiC papers in ascending grit sizes (500, 1000, 1200, and 2500 grits, respectively), followed by dehydration through immersion in ethanol solutions of increasing concentrations (60%, 80%, 90%, and 100%) for 2 minutes each. To evaluate the ceramic–cement–dentin interface thickness (11), the analysis was performed using SEM. Photomicrographs of the cross-sections were taken at 200× and, if necessary due to reduced film thickness, at 500× magnification. Film thickness values were obtained directly from the microscope's imaging software at the thickest, thinnest, and midmost regions of the luting agent. The mean of the three measurements was calculated and used to represent the film thickness for each sample.

Statistical Analysis

The means and standard deviations of fracture resistance values (N) and film thickness (μm) were calculated using descriptive statistics. All data were organized and analysed for homogeneity of variance and normal distribution using the Levene test and Kolmogorov–Smirnov test, respectively. The data were normally distributed and showed homogeneity of variance, a one-way ANOVA was conducted. A *p*-value of less than 0.05 was considered statistically significant

Results

The fracture resistance values (N) and the film thickness (μm) of e.max CAD bonded with different resin-based luting composites are summarized in Table 3. Among the tested materials, no statistically significant differences were found among the groups for both parameters ($p>0.05$). The result suggested that all tested flowable composites, highly-filled flowable composite and the resin cement provided comparable fracture resistance and flim thickness when used with e.max CAD.

Table 3 shows mean fracture resistance (N), film thickness (μm) and standard deviation of different resin composites bonded with E.max CAD. No statistically significant differences were found among the groups (*p*>0.05)

	Composite cements				
	Flowable composite				Resin cement
	Filtek™ Supreme Flowable	G-ænial™ Universal Injectable	Clearfil™ AP-X Esthetics FLOW	Beautifil injectable X	Multilink N
Fracture resistance (N)	219.62 ± 17.77	206.323 ± 15.31	207.453 ± 12.09	215.12 ± 20.879	214.143 ± 18.49
Film Thickness (μm)	52.55 ± 9.91	42.239 ± 15.34	38.974 ± 7.00	50.533 ± 14.32	42.231 ± 9.67

Discussion

The results of this study indicated that the fracture resistance and film thickness were not substantially influenced by the use of various flowable composite with IPS e.max CAD. Consequently, both null hypotheses were accepted (p > 0.05).

The literature has reported inorganic filler load values ranging between 37% and 53% by volume for flowable resin composites [3, 25]. Therefore, FF and BI, which contain 46% and 42% vol fillers, respectively, were selected to represent conventional flowable composites. On the other hand, CF with a filler content of 59% vol, was included to represent highly filled flowable composites. For GI, specific data regarding its filler volume percentage (%vol) were not available from the manufacturer; however, the material was selected based on the manufacturer's claim that it is a high-strength composite suitable for all restorative indications. In this study, filler content was reported as volume percentage (%vol) to more accurately reflect the spatial distribution of fillers within the resin matrix. Given that the mechanical and physical properties of resin composites are more strongly influenced by filler volume rather than weight, the use of %vol provides a more reliable and meaningful comparison between different materials [26].

In this study, no significant differences in fracture resistance were found between resin cements and flowable composites. This may be attributed to the dominant role of the ceramic's inherent strength, which likely outweighs the influence of the thin cement layer. These findings are consistent with those of Guess et al. (2013), who reported that the intrinsic strength of the ceramic material plays a more crucial role in resisting fracture than external factors such as cement layer thickness [27]. This study employed ceramic specimens with a 1.0 mm thickness, which aligns with previous research showing that the load at crack initiation and the time to crack propagation in chair-side CAD/CAM lithium disilicate with a 1.0 mm occlusal thickness did not differ statistically from the previous 1.5 mm recommendation [28]. Additionally, a 1.0 mm ceramic thickness is favorable in terms of light transmission. Supporting evidence demonstrated that 1.5 mm and 2.5 mm thick ceramics significantly attenuated light transmission, leading to reduced mean values of μSBS, degree of conversion, and polymerization shrinkage stress for all types of resin cements tested [29].

In this study, water storage was performed prior to specimen sectioning to ensure adequate completion of the auto-cure polymerization process. Previous studies have used a 7-day water storage period for this purpose. Immediate sectioning after cementation may disrupt the ongoing polymerization of the resin cement, potentially leading to incomplete curing and compromised bonding performance. Therefore, delayed sectioning after water storage was adopted to minimize these effects and ensure more consistent results [30, 31].

The film thickness of most tested flowable composites, excluding FF, was less than 50 μm, complying with ISO 4049:2019 standards. Although highly filled flowable composites contained a greater amount of filler, they exhibited similar film thickness to that of conventional flowable composites and resin cements. This phenomenon may be attributed to the incorporation of rheological modifiers in highly filled flowable composites, which effectively reduce viscosity. The simplest method for decreasing the viscosity of composites is to lower the viscosity of the monomer mixture itself [32]. Notably, the primary monomers used in GI and CF composites are UDMA and TEGDMA, respectively, both of which have lower molecular weights and viscosities compared to Bis-GMA. Previous studies have shown that the flowability of composites depends not only on the composition and ratio of the resin matrix [33], but also on the content, shape, size distribution, and silane treatment of the fillers [34].

Although this study evaluated the fracture resistance and film thickness of various flowable composites in comparison with resin cement, several limitations must be considered. The mechanical performance of luting agents in clinical applications is influenced by multiple factors beyond fracture resistance, including bond strength to both tooth structure and ceramic surfaces, long-term durability under cyclic loading (fatigue resistance), water sorption, solubility, and resistance to thermal and mechanical stresses [35-38]. In addition, the experimental design employed flat specimens, whereas actual restorations often involve more complex geometries. Such differences may affect light accessibility and polymerization efficiency, particularly for light-cured materials. Therefore, future investigations should include comprehensive evaluations to thoroughly validate the potential of flowable composites as alternative luting agents for ceramic restorations.

Conclusion

Within the limitations of this study, flowable composites demonstrated comparable fracture resistance and film thickness to conventional resin cement when bonded to lithium disilicate-based ceramic restorations. Flowable composites may offer a potential alternative to resin cements for ceramic luting; however, comprehensive mechanical and clinical validations are essential before their routine clinical application can be recommended.

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