

Influence of Filler Type of Preheated Composite Resin on Microtensile Bond Strength and Film Thickness When Used for Adhesive Cementation: An *In Vitro* Study

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ABSTRACT

Objective: The purpose of this study was to evaluate the effect of preheated composite resins with different filler sizes when used to cement ceramic restorations.

Material and Methods: Thirteen class II cavities were prepared in premolars, and pressed lithium disilicate inlays were luted with 2 different preheated composite resins: nanohybrid and microhybrid; a dual-polymerized resin cement was used as the control. The cement film thickness was measured by confocal microscopy, and the quality of the bonded interface by scanning electron microscopy. Ceramic disks of preheated composite resins were used to measure the μ TBS.

Results: No marginal discrepancy in the bonded interface between dentin-film cement and the ceramic restoration was observed with either preheated composite. The preheated nano- and microhybrid cements had lower film thickness values compared with the dual-polymerized resin cement at the axial walls ($P < .05$); median \pm standard deviation values of cement film thickness ranged from 77.7 ± 33.6 to 115.3 ± 52.3 μ m for preheated microhybrid cement and from 76.1 ± 34.0 to 82.7 ± 31.7 μ m for preheated nanohybrid cement ($P > .05$); the highest values of cement film thickness were at the floor of the cavity, with higher formation of voids in the dual-polymerized resin cement. No significant differences ($P > .05$) were found for the median values of the VHN of the film cement and for the μ TBS between the preheated nano- and microhybrid cements. Cohesive failure was the most common, followed by mixed failure.

Conclusions: Preheated composite resins with different filler sizes resulted in similar cementation of pressed lithium disilicate inlays.

Keywords: Preheat Composite Resin; Filler Type; Lithium Disilicate Restorations; Film Cement; Microtensile Bond Strength

Abbreviations: VHN: Vickers Hardness Number; μ TBS: Microtensile Bond Strength

Introduction

The cementation procedure and cementation agent are critical factors that influence the quality and longevity of a fixed prosthesis, and improper manipulation of the luting agent can affect its physical and mechanical properties [1]. Recent innovations in the cementation procedure, including preheating composite resin, have been reported to increase the durability and clinical behavior of fixed prostheses [2]. Preheating to modify properties has been applied to glass ionomer cements and increases the viscosity [3,4] and accelerates the setting reaction by increasing the ion diffusion rate [5,6]. Preheated composite resins have also been used as luting agents for the cementation of partial coverage restorations, as preheating reduces their viscosity, increasing flowability and minimizing film thickness [7-12]. Advantages of preheated composite resins have been reported to include improved marginal adaptation [13,14], and their color has been reported to be unaltered by preheating [15,16]. However, evidence is lacking on whether the filler particle size influences bonding to ceramic restorations, although the composition of the composite resin has been reported to affect its viscosity after preheating [17]. In addition, the operator's handling skills are a major factor in the cement thickness, which determines the marginal adaptation of the restoration [18]. These aspects may play an important role in the mechanical properties of preheated composite resin for cementing ceramic restorations compared with a dual-polymerizing resin cement [19,20].

Therefore, the purpose of this *in vitro* study was to evaluate the effect of 2 preheated composite resins with different filler sizes: nano-hybrid and microhybrid versus a dual-polymerizing resin cement for luting lithium disilicate restorations. The null hypothesis was that the type of filler size would not influence the bonding interface, cement

film thickness, Vickers hardness number (VHN), or microtensile bond strength (μ TBS) when used to lute lithium disilicate partial coverage restorations.

Material and Methods

A total of 30 human maxillary premolars and 30 human third molars were collected from patients who visited the specialty clinic of the Orthodontics and Surgery Departments of the Autonomous University of San Luis Potosi, after the acquisition of informed and signed consent. The study was approved by the ethics committee of the Autonomous University of San Luis Potosi (CEI-FE-055-022). The inclusion criteria were teeth without damage to their clinical crown or root and free of carious lesions, fractures, or restorations. The teeth were stored in saline solution, scaled to remove any plaque, calculus, or periodontal ligament, washed with saline solution, and kept in thymol at 4°C until use, but for no longer than 3 months after extraction. A single operator (I.C.P.B.) prepared standardized rectangular-shaped class-II cavities in the premolars (3 mm mesio-occlusal width, 3 mm bucco-palatal width, and 3 mm depth) with enamel margins and axial walls (buccal and palatal) in dentin; the preparations were measured with a periodontal probe, and the cavities were prepared with an electric high-speed handpiece dental motor (NLX nano, NSK; IL, USA) at 20 000 rpm with diamond rotary instruments (Coarse Flat-End Taper Diamond; Brasseler, GA, USA) under constant water cooling. The diamond rotary instrument was replaced after every 5 preparations. Thirteen inlays and 13 \varnothing 6×3 mm ceramic disks were fabricated from a lithium disilicate glass-ceramic (IPS e.max Press, Ivoclar Vivadent AG; Schaan Liechtenstein) (LT-A2). The cavities were scanned with an intraoral scanner (Emerald S, Planmeca USA Inc; IL, USA), and the inlays were designed with a software program (Exocad Dental CAD 3.1; MA, USA), providing a cement space of 40 μ m (Figures 1A & 1B)).

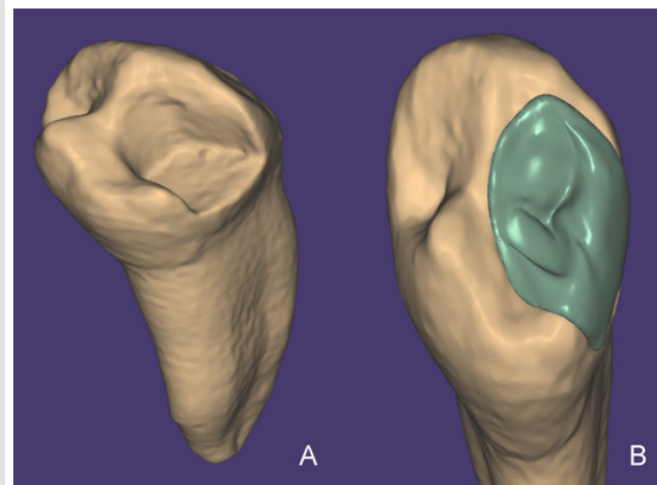


Figure 1:

- A. Class II cavity.
- B. Design of inlay restoration.

The specimens were invested (IPS PressVEST, Ivoclar Vivadent AG; Schaan Liechtenstein) and pressed (Programat EP 5000; Ivoclar Vivadent AG, Schaan Liechtenstein) according to the manufacturer’s instructions. The surfaces of the lithium disilicate disks were finished with waterproof abrasive paper (Wetordry Sanding Sheets, 3M ESPE; MS, USA). The inlays were cemented according to the manufacturer’s instructions (Table 1) and divided into 3 experimental groups (n=10). The composite resins were preheated for 60 minutes at 54°C and immediately used after being removed from the heating device (Ena Heat Composite Heater, Micerium S.p.A; GE, Italy). The temperature of the composite resin was verified with a thermometer (Mercury-Filler Armored Thermometer, Gilson Co, Inc; OH, USA). Before luting, the

composite resin was placed on a glass slide and then quickly measured with a precision balance (Electronic Balance, Thermo Fisher Scientific, Inc; MA, USA) to standardize the quantity of material for luting. The median time between removing the composite resin from the heating device and light-polymerization was approximately 40 seconds for all tests. The surfaces of the restorations were treated similarly before cementation in all the groups (Table 2). Gradual finger pressure was used until the restorations were completely seated, and excess material was removed with a microbrush. The resin was polymerized with a light-polymerizing unit (VALO, Ultradent Products Inc; UT, USA) at 1200 mW/cm² for 20 seconds. The tests were conducted after storing the teeth in distilled water for 24 hours.

Table 1: Tested materials, type, and composition according to manufacturer’s data.

Product name manufacturer (Batch no.)	Type	Manufacturer	Average size	Shade	Composition
Ena HRi (2022003487)	Nanohybrid resin composite	Micerium S.p.A.	1.0 µm- 20 nm	A2	Tetramethylenedimethacrylate (2.5–10%). Content of fillers: 74% by weight (60% by volume); particle size of highly dispersed silicon dioxide is 0.005–0.05 µm.
Filtek Z250 (NC96033)	Microhybrid resin composite	3M ESPE	0.01–3.5 µm (average: 0.6)	A2	Matrix: Bis-GMA, UDMA, Bis- EMA, TEGMA. Fillers: Zirconia, silica 10–3500 nm (0.01–3.5 µm). Filler loading: 75–85 wt%, 60 vol%.
Duo link Universal (2200002815)	Dual- polymerized resin cement	Bisco Inc.			Bisphenol A diglycidyl methacrylate (5-30%) Triethyleneglycol dimethacrylate (5-20%) Glass filler (50-80%) Urethane dimethacrylate (5-15%).

Note: Bis-EMA, bisphenol A ethoxylated dimethacrylate; Bis-GMA, bisphenol A-glycidyl dimethacrylate; TEGDMA, triethyleneglycol dimethacrylate; UDMA, urethane dimethacrylate.

Table 2: Description of procedures for placement of resin cement.

Treatment surface	Procedure
Dentin	Etching with 35% phosphoric acid (Ultra-Etch; Ultradent Products, Inc) for 15 seconds on enamel along finish line. Rising with air/ distilled water spray for 1 minute, removing excess water with high-speed suction, and gently airdried without desiccation. Two layers of adhesive system (All Bond Universal; Bisco) applied actively and excess removed with microbrush, followed by evaporation of solvent for 5 seconds.
Intaglio surface of restoration	Etching with 9% hydrofluoric acid (Porcelain etch; Ultradent Products, Inc) for 20 seconds, followed by air/ distilled water spray for 1 minute. 35% phosphoric acid applied for 30 seconds, followed by air/ distilled water spray for 1 minute. Ultrasonic bath with distilled water for 10 minutes and dried in oil-free air. Silane agent (Ceramic silane; Ultradent Products, Inc) applied, and after 5 minutes surface dried with air spray for 20 seconds.

The VHN values (N/mm²) were obtained with a Vickers hardness tester (Digital Hardness Tester; Guangdong, Sinowon) under 100 g for 10 seconds in the disk-shaped specimens of the DPRC and PCR (n=10). Median value differences in VHN values were calculated. The occlusal surface was sectioned transversely to evaluate the bonding interface and the cement film thickness, followed by the wall of the proximal restoration, and an internal longitudinal section was made

in the central part of the restoration. Each premolar was sectioned in half through the center of the restoration with a diamond disk guided by 2 lights in a cutting machine (IsoMet Wafering Blades 15 LC, Buehler; IL, USA), resulting in 2 sections with a 1.5 mm width, exposing the adhesive interface. The disks were finished with 600-, 800-, and 1,000-grit abrasive paper (Sandpaper, 3M ESPE; MS, USA) and then desiccated in 25% to 100% alcohol for 30 minutes and coated with 15

to 20 nm gold particles to be observed with a scanning electron microscope (SEM) (JSM-6510, JEOL; CA, USA). Before the cementation protocol, rhodamine B (Sigma-Aldrich 1%) was added to the dual-polymerized resin cement and the preheated composite resins to evaluate the cement film thickness by confocal laser scanning microscopy (SP5 II, Leica; IL, USA). The images were obtained (LAS AF, Leica; IL, USA) under $\times 400$ magnification. The cement film thickness layer was measured on each side of the cavity (buccal, palatal walls and cavity floor) and the median values were obtained.

μ TBS was assessed on the 30 molars (Figure 2A). A single operator (I.C.P.B.) sectioned the clinical crown at the cementum-enamel junction with a double-sided diamond disk (NTI Serrated Diamond Disc, Kerr Corp; CA, USA) and smoothed it with a wheel-shaped diamond rotary instrument (909.31.055 FG Medium Wheel Diamond, Brasseler; GA, USA) (Figure 2B). Two layers of adhesive (All Bond Universal, BISCO Inc; IL, USA) were placed on the sectioned dentin, and the solvent was evaporated with a 5-second air stream. The lithium disilicate disks were luted as previously described for the restorations (Figure 2C). Longitudinal sections (Figure 2D) were made

to obtain slices (Figure 2E) and cross-sectional sections to obtain $1 \times 1 \times 6$ mm specimens (Figure 2F). The specimens were fixed to a notched gripping device (TA. XT Plus C, Stable Micro System; United Kingdom) and tested under tensile stress at a crosshead speed of 1 mm/min until failure. The failure type was determined by visual inspection with a stereomicroscope (EZ4W, Leica; IL, USA) under $\times 40$ magnification and was classified as adhesive if the fracture occurred at the dentin-ceramic and luting agent interface, cohesive if in the luting agent, or mixed if a combination of adhesive and cohesive failure. One calibrated examiner, who was unaware of the group allocation, evaluated the specimens. The data were analyzed with a statistical software program (SigmaPlot version 11.0, Inpixon; CA, USA). The normal distribution and homogeneity of variance were checked using the Shapiro-Wilk test, and the homogeneity of variances with the Levene test. μ TBS, VHN, and cement film thickness data were individually analyzed by 1-way analysis of variance (ANOVA). Tukey post hoc tests were used to determine differences among groups. The Pearson chi-squared test was used for the failure mode data ($\alpha=.05$ for all tests). Descriptive analysis was used to determine the failure mode.

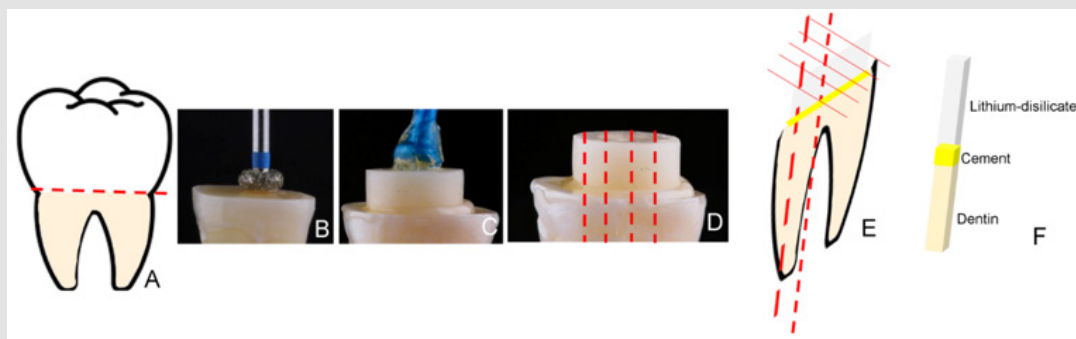


Figure 2: Schematic of specimen preparation for microtensile bond strength test.

- A. Third molar tooth.
- B. Clinical crown sectioned at cementum-enamel junction.
- C. Luting of lithium disilicate disk.
- D. Specimens cut perpendicularly with a low-speed diamond saw to obtain slices.
- E. Rectangular cross-sectional cuts.
- F. Stick-shaped specimens.

Results

The overall cement film thickness, VHN, and μ TBS median values of the luting agents are shown in Table 3. The median values of the cement film thickness were higher than those of the dual-polymerized resin cement at the axial walls and floor cavity compared with the preheated nano- and microhybrid cements. The 1-way ANOVA showed a significant difference in the cement film thickness between the axial walls compared with the dual-polymerized resin cement versus the preheated composite resins ($P < .05$). In contrast, no signif-

icant difference in the cement film thickness was found between the preheated composite resins at the axial walls and floor cavity ($P > .05$). The cement film thickness in the floor cavity showed no significant difference between the dual-polymerized resin cement and the preheated composite resins. The preheated composite resins had a thicker film than the dual-polymerized resin cement at the axial walls ($P < .05$), and a higher number of voids were found in the floor cavity of the dual-polymerized resin cement than in the preheated nano- and microhybrid cements.

Table 3: Comparison of median values ±standard deviations of VHN in N/mm², μTBS in MPa, and cement film thickness in μm.

	Dual- Ploymerized Resin Cement	Microhybrid	Nanohybrid
Film Cement VHN (N/mm² ±SD) (P)			
Buccal	67.8 ±12.1	125.3 ±14.7* (.001)	132.8 ±16.5* (.011)
Floor of cavity	113.0 ±21.2	152.3 ±21.2* (.001)	137.0 ±15.3* (.001)
Palatal	68.1 ±9.4	117.7 ±9.8* (.003)	136.2 ±21.7* (.003)
Ceramic Disk- Shaped Specimens	57.0 ±3.4	95.3 ±17.4* (.002)	135.5 ±16.6* (.001)
Microtensile Bond Strength (MPa ±SD)			
	14.82 ±2.9	16.26 ±1.2	18.06 ±3.6
Cement Film Thickness (μm ±SD) (P)			
Buccal	197.8 ±38.4 ^a	115.3 ±52.3 ^{ac} (.002)	82.7 ±31.7 ^{ac} (.003)
Floor of cavity	193.0 ±37.9 ^a	187.3 ±57.8 ^b	182.0 ±50.2 ^b
Palatal	178.0 ±21.9 ^a	77.7 ±33.6 ^{ck} (.021)	76.1 ±34.0 ^{ck} (.010)

*Indicates statistically significant (P<.05) versus control group (Dual-Polymerized Resin Cement). Median followed by same lowercase letter within column not statistically different (P>.05). μTBS, Microtensile Bond Strength; SD, Standard Deviation; VHN, Vickers Hardness Number.

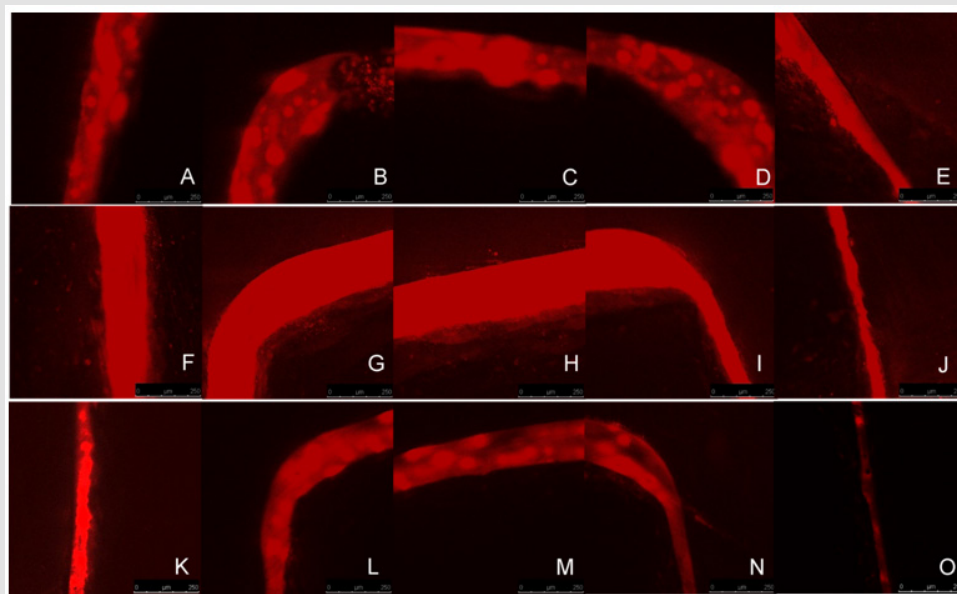


Figure 3: Representative confocal fluorescence image showing configuration of cement film thickness with Rhodamine from buccal wall, floor cavity to palatal wall. (A-E) Dual-polymerized resin cement, (F-J) Preheated microhybrid composite resin, (K-O) Preheated nanohybrid composite resin.

Note: N/mm², μTBS in MPa, type of failure. and cement film thickness in μm.

*Indicates statistically significant (P<.05) versus control group (dual-polymerized resin cement). Median followed by same lowercase letter within column not statistically different (P>.05). μTBS, microtensile bond strength; SD, standard deviation; VHN, Vickers hardness number.

Figure 3 shows the configuration sequence of the cement film thickness with the dual-polymerized resin cement (Figures 3A-3E)), with the preheated micro- (Figures 3F-3J)), and nanohybrid cements (Figures 3K-3O)). Representative micrographs at ×400 magnification of the bonding interface with dual-polymerized resin cement (Figures 4A-4C)) and with preheated microhybrid cement (Figures 4D-4F)) and using preheated nanohybrid cement (Figures 4G-4I)), no marginal discrepancy in the bonded interface between dentin-film cement

and the ceramic restoration was observed with either preheated composite resin. The median values of VHN on the disk-shaped specimens of preheated composite resins were lower than those of the film cement at the axial walls and floor cavity of the preheated composite resins. The VHN results revealed statistically significant differences between the dual-polymerized resin cement and the preheated microhybrid and nanohybrid cements ($P > .05$). For the measurements of the film cement at the axial walls and the floor cavity, the VHN median values showed statistically significant differences in the dual-polymerized resin cement and the preheated microhybrid cement ($P < .05$) but not for the preheated nanohybrid cement ($P > .05$). The median

values of μTBS not showed statistically significant differences ($P > .05$) for the dual-polymerized resin cement and preheated composite resins. The distribution of failure modes among the luting agents is shown in Figure 5A. The failures were predominantly cohesive for the dual-polymerized resin (65%), preheated microhybrid (55%), and preheated nanohybrid (40%) cements (Figure 5B), followed by adhesive failure for dual-polymerized resin (35%), preheated nanohybrid (25%), and preheated microhybrid (20%) cements (Figure 5C). Mixed failure was present only in the preheated microhybrid (25%) and preheated nanohybrid (15%) cements (Figures 5D).

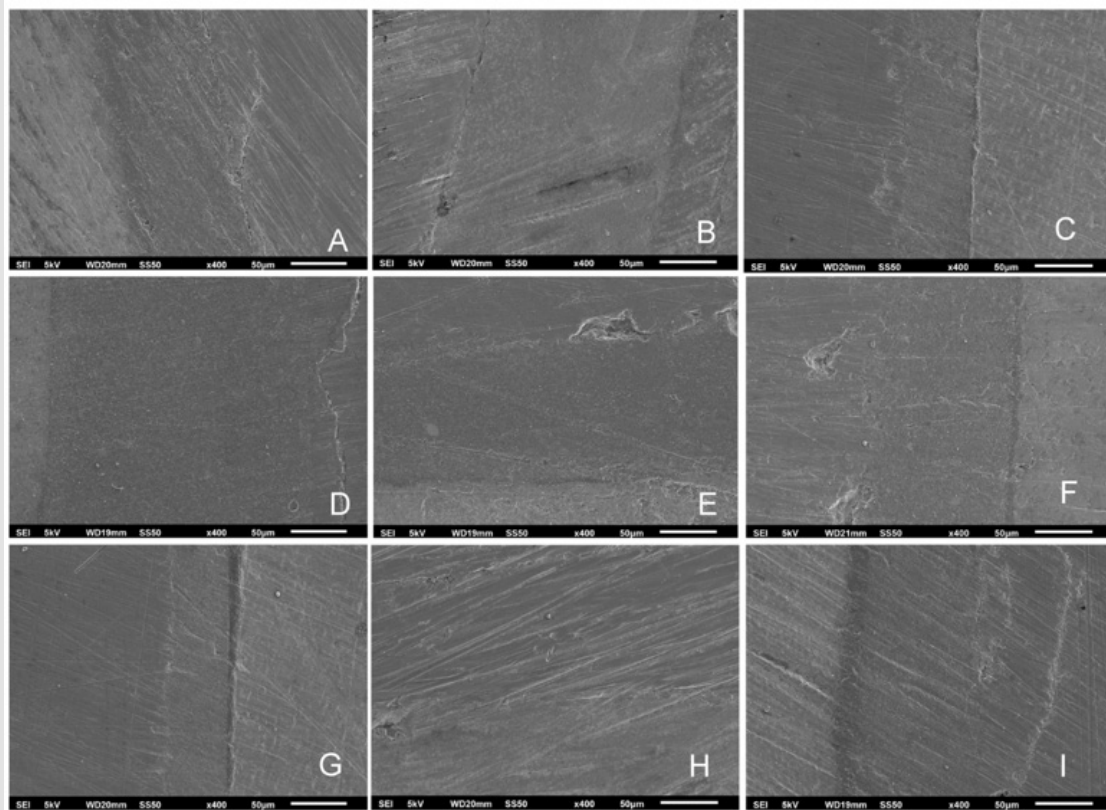


Figure 4: Scanning electron microscope images of adaptation to cavity walls and bonding interface of inlay restorations with dual-polymerized resin cement:

- A. Buccal dual-polymerized resin cement.
- B. Floor of cavity dual-polymerized resin cement.
- C. Palatal dual-polymerized resin cement.
- D. Buccal preheated microhybrid composite resin.
- E. Floor of cavity preheated microhybrid composite resin.
- F. Palatal preheated microhybrid composite resin.
- G. Buccal preheated nanohybrid composite resin.
- H. Floor of cavity preheated nanohybrid composite resin.
- I. Palatal preheated nanohybrid composite resin.

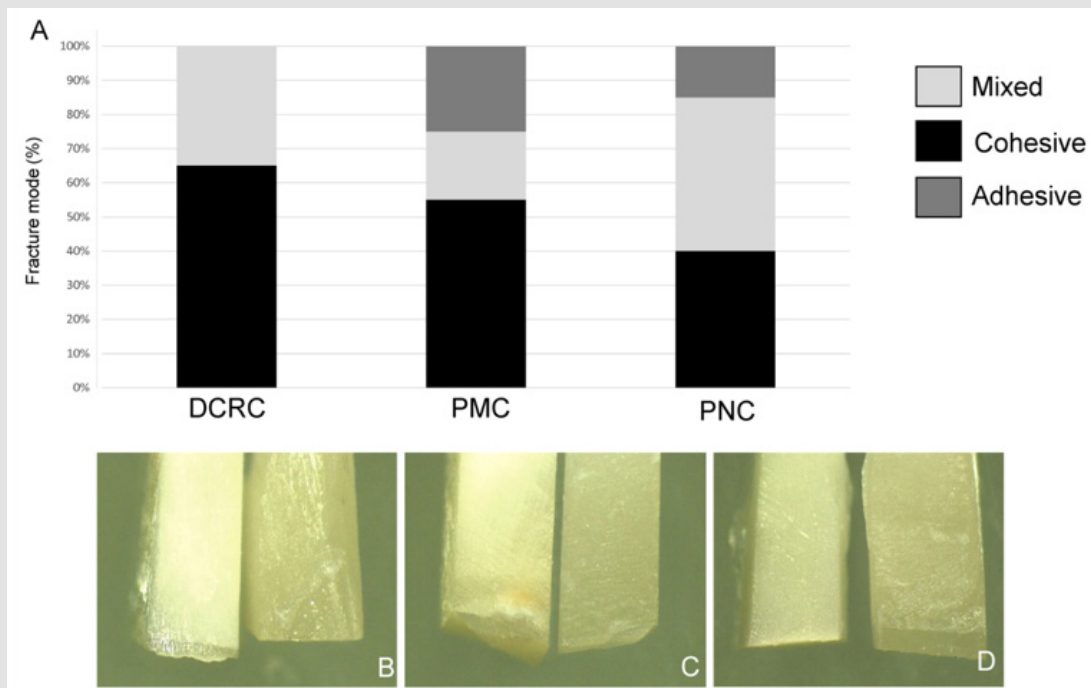


Figure 5:
 A. Percentages of failure mode.
 B. Cohesive failure.
 C. Adhesive failure.
 D. Mixed failure.

Discussion

The filler size of preheated conventional composite resin was not found to significantly influence the μ TBS, VHN, or cement film thickness when used as a cement for luted lithium disilicate inlays. Therefore, the null hypothesis was rejected. However, the dual-polymerized resin cement showed different values. Different luting agents are available for the cementation of restorations but can affect their clinical behavior and different preheating techniques for conventional composite resins used for cementation have been described [20]. Controlling for the preheating device, preheating temperature and time, particle size of the composite resin, and bonding procedure to prevent dissipation of heat has not allowed a general conclusion on the clinical cementation procedure [2]. Similar μ TBS values were found with the preheated microhybrid and nanohybrid composite resins, both showing an improved bonding interface, cement film thickness, and VHN when used as luting agents compared with the dual-polymerized resin cement. The cement film thicknesses were statistically similar for the microhybrid and nanohybrid composite resins, both being lower than the dual-polymerized resin cement. The cement film was thinner in the axial walls compared with the floor of the cavity.

The studies are consistent in that preheating composite resin for luting procedures did not improve μ TBS, possibly because of the loss

of temperature during the bonding procedure. Moreover, the effect of temperature on the extent of polymerization has been demonstrated to depend on the photo-initiator system of the composite resin and other properties that may vary from brand to brand [9]. Previous studies have shown that preheating increased flowability. This change in viscosity depends on the resin composition and the filler content, and composite resins with a high percentage of inorganic filler particles are highly viscous [10,11]. Flowability enhances the adaptation of the preheated composite resin to cavity walls, especially into internal angles [12] and results in thinner luting interfaces, which should improve restoration longevity. In the present study, the SEM images showed a complete seal without the presence of cracks or irregularities in the cement film or at the bonded interface. In the case of the dual-polymerized resin cement, the seal was also complete, but the confocal laser scanning microscopy images showed voids in the floor cavity. The microhybrid composite resin had fewer voids, and no voids were observed with the preheated nanohybrid composite resin; therefore, the use of preheated composite resins reduced the risks of incorporating interfacial voids between the cement and the restoration. The VHN values in the film cement were statistically like those of the preheated nanohybrid and microhybrid composite resins.

Preheating has been reported to maximize polymerization and improve the microhardness of composite resins, although the micro-

hardness may depend on the composition of the composite resin. Blalock, et al. [6]) reported that cement film thickness was not a function of filler content or particle shape. In another study by (Ayub, et al. [21]) compared 3 microhybrid composite resins with different filler content and average sizes compared with a nanofiller composite resin. They reported that the highest microhardness values were found with the preheated nanocomposite, characterized by the lowest filler particle size and the highest filler content. The present results were consistent with those of other studies concluding that preheating composite resins provided clinical advantages such as improved adaptation to the cavity walls and unchanged mechanical properties [2-22]. The present result suggested that preheated composite resin used in cementation resulted in the better seating of indirect restorations than a dual-polymerized resin cement. Limitations of this study included the in vitro design that did not necessarily reflect the clinical environment of the oral cavity. Further studies are required to study the longevity of restorations with different ceramic restorative materials and different luting agents. Clinical studies are necessary to evaluate the effectiveness of preheated composite resin as luting agents of partial and complete-coverage ceramic restorations.

Conclusions

The filler size of preheated composite resin did not significantly affect the bonded interface, cement film thickness, surface microhardness, or microtensile bond strength. Preheating nanohybrid and microhybrid composite resin provided better film cement parameters than a dual-polymerized resin cement. Preheated nanohybrid or microhybrid composite resin is a suitable luting agent for lithium disilicate inlays.

Authors Conflicts

There are no conflicts of interest.

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