

RESEARCH ARTICLE

Effect of light-curing techniques and preheated composite types as luting agents on lithium disilicate bond strength

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ABSTRACT

The bond strength between lithium disilicate veneers and tooth structure depends on factors such as curing technique and luting agent. Preheated composite resins have been proposed as alternative luting agents due to their favorable mechanical and polymerization properties. This study aimed to evaluate the effects of conventional, ramp, and pulse light-curing techniques on the shear bond strength of lithium disilicate when preheated microhybrid and nanohybrid composite resins were used as luting agents. Forty-two maxillary premolars were prepared and randomly assigned to six groups ($n = 7$) based on the combination of luting agent and light-curing technique: IA (microhybrid–conventional), IB (microhybrid–ramp), IC (microhybrid–pulse), IIA (nanohybrid–conventional), IIB (nanohybrid–ramp), and IIC (nanohybrid–pulse). Lithium disilicate veneers were cemented accordingly, and shear bond strength was evaluated. Results from a two-way ANOVA indicated a significant effect of the type of preheated composite resin on the shear bond strength of lithium disilicate ($p < 0.05$), while no significant effect of light-curing technique was observed ($p > 0.05$). This study confirmed that preheated microhybrid composite resin produces greater shear bond strength compared to preheated nanohybrid composite resin. Light-curing technique does not significantly affect the shear bond strength of lithium disilicate.

Keywords: cementation; light-curing; preheated composite resin; shear bond strength

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INTRODUCTION

The field of restorative dentistry continues to advance rapidly in response to the increasing patient demand for dental treatments that are not only functional but also highly esthetic. Veneers are a preferred treatment option for restoring anterior teeth due to their minimally invasive nature compared to full-coverage crowns while effectively addressing patients' esthetic concerns. A veneer is a thin layer of tooth-colored restorative material applied to the tooth surface to manage discoloration, fractures, attrition, abnormal morphology, or mild malposition.^{1,2} Based on fabrication methods, veneers are classified as direct or indirect. Indirect veneers are fabricated through a process that involves laboratory procedures, and they offer several advantages, including reduced chairside time for multiple restorations, superior contour and

color outcomes, and greater durability compared to direct veneers.³

Lithium disilicate ceramic is the most commonly used material for indirect veneers due to its excellent mechanical and esthetic properties. Comprising approximately 70% crystalline content, lithium disilicate exhibits a flexural strength of 360–400 MPa, comparable to that of enamel. Additionally, its optical properties allow for natural light reflection, enhancing the esthetic quality of restorations.⁴ The clinical success of lithium disilicate veneers depends significantly on the choice of cementation material. Resin cement is the preferred luting agent due to its strong bonding capability and superior esthetics; however, its low filler content, while beneficial for flowability, also makes it more susceptible to intraoral degradation, microleakage, and reduced hardness.^{5,6,7} Resin

cement luting agents may undergo discoloration over time, compromising the esthetics of ceramic restorations. This discoloration is attributed to the presence of unreacted tertiary amines remaining after polymerization.⁸

Preheated composite resin has emerged as an alternative luting material to overcome the limitations of conventional resin cement. Composite resin is widely used in dentistry due to its excellent mechanical properties and aesthetics. Preheating composite resin before photopolymerization reduces its viscosity by 25–70%, facilitating improved flowability and adaptation during cementation.⁹ This technique enhances the degree of monomer conversion, reduces polymerization shrinkage and microleakage, improves marginal adaptation, and offers greater shade stability and intraoral durability at a lower cost compared to conventional resin cement.^{5,7,10} Composite resin can be classified into four types based on filler particle size: macrofill (0.1–100 µm), microfill (0.01–0.1 µm), nanofill (0.005–0.01 µm), and hybrid. Hybrid composite resins are developed to combine the advantages of various filler sizes, resulting in materials with high mechanical strength and excellent esthetic properties. Microhybrid and nanohybrid composite resins are commonly used for restorative purposes, with microhybrid resins containing filler particles of 0.04–1 µm and nanohybrid resins incorporating a combination of larger fillers (0.4–5 µm) and nanosized fillers.^{3,11}

The appropriate light-curing technique for light-activated luting agents is essential to ensuring adequate polymerization, which enables the resin-based luting agent to achieve optimal physical and mechanical properties, ultimately influencing the final restoration quality. Different light-curing techniques also affect the degree of polymerization shrinkage, where excessive shrinkage can create interfacial gaps within the restoration, leading to microleakage and reduced bond strength at the ceramic-luting agent-tooth substrate interface.^{12,13} The conventional light-curing technique, which maintains a constant light intensity throughout the curing process, is

commonly used for composite resin activation. However, various curing techniques have been developed to enhance the properties of composite resin, including the soft-start technique, which modulates light intensity to reduce shrinkage stress.¹⁴ Compared to conventional curing, the soft-start technique delivers lower energy density, thereby reducing molecular movement and minimizing monomer contraction. This reduction in polymerization shrinkage improves the interfacial adaptation of the restoration, enhancing bond strength.^{15,16} The soft-start curing technique commonly used in light-curing units can be categorized into two types: ramp cure and pulse cure. Ramp cure begins with low light intensity, gradually increasing to a higher intensity over a specific period before maintaining maximum intensity until the curing process is complete. Meanwhile, pulse cure involves multiple on-off cycles within a single curing session, where the light is activated at maximum intensity during the on cycle and turned off or dimmed during the off cycle.¹⁷

Various testing instruments are available to assess the biomechanical and physical properties of dental materials. The shear bond strength test is a simple and widely used method for evaluating the adhesion of restorative materials. Shear bond strength refers to the maximum ability of a material to withstand shear forces before debonding occurs. This test is crucial for understanding the interfacial relationship between two materials.¹¹ According to Ozturk et al, the debonding forces experienced by veneer restorations during intraoral function are best simulated using a shear bond strength test. This method effectively evaluates the bond strength between adhesive materials and enamel or dentin, as well as the restorative material itself. A decrease in shear bond strength may indicate inadequate adhesion quality, which should be carefully considered to ensure optimal clinical outcomes of restorations.¹⁸ Based on this principle, the present study aims to evaluate the effect of different light-curing techniques and preheated composite resin types as luting agents on the shear bond strength of lithium disilicate.

MATERIALS AND METHODS

Ethics approval for this in vitro study was obtained from the Research Ethics Committee of the Faculty of Dentistry-RSGM UGM Prof. Soedomo, Universitas Gadjah Mada, Yogyakarta, Indonesia (number 62/UN1/KEP/FKG-RSGM/EC/2024). A total of 42 human maxillary premolars extracted for orthodontic purposes were selected as research samples. The teeth met the following inclusion criteria: intact crowns, caries-free, absence of vertical or horizontal fractures and developmental abnormalities, a maximum storage period of three months post-extraction, and patient age at the time of extraction ranging from 17 to 25 years. The selected samples were then randomly allocated into six groups, with each group comprising seven teeth. The groups were as follows: Group IA (preheated microhybrid composite resin + conventional light-curing technique), Group IB (preheated microhybrid composite resin + ramp light-curing technique), Group IC (preheated microhybrid composite resin + pulse light-curing technique), Group IIA (preheated nanohybrid composite resin + conventional light-curing

technique), Group IIB (preheated nanohybrid composite resin + ramp light-curing technique), and Group IIC (preheated nanohybrid composite resin + pulse light-curing technique). The composition of the composite resins used in this study is presented in Table 1, while the light-curing technique parameters are detailed in Table 2.

A total of 42 lithium disilicate discs, each with a diameter of 3 mm and a height of 1 mm, were fabricated in a dental laboratory. Forty-two extracted maxillary premolars were disinfected by immersion in a 0.5% chloramine-T solution for 24 hours. Calculus on the teeth was removed using an ultrasonic scaler, followed by cleaning of debris with pumice paste mixed with distilled water. This was performed using a nylon brush attached to a contra-angle low-speed handpiece, rotating at 1000 rpm for 5 seconds at a 90° angle. The teeth were then immersed in distilled water and stored in a refrigerator at 4 °C until the study was conducted, following ISO 29022 guidelines. The distilled water was replaced regularly every two days.

The teeth were embedded in cold-cure acrylic resin, prepared by mixing powder and liquid in a 3

Table 1. Composition of composite resins used in the study

Brand	Filtek Z250	Filtek Z250 XT
Manufacturer	3M ESPE, USA	3M ESPE, USA
Type	Microhybrid	Nanohybrid
Matrix	Bis GMA, UDMA, Bis-EMA	Bis GMA, UDMA, Bis-EMA, TEGDMA
Matrix loading	39 vol%	31 vol%
Filler	zirconia/silica	zirconia/silica, nano-sized silica
Filler loading	60 vol%	68 vol%
Filler size	0.01-3,5 µm	20 nm - 3 µm

Table 2. Light-curing technique parameters used in the study

Light-Curing technique	Light-Curing method	Curing duration
Conventional	Constant intensity of 850 mW/cm ² for 20 seconds, repeated twice	40 s
Ramp Cure	Gradual intensity increase from 0 mW/cm ² to 850 mW/cm ² during the first 5 seconds, followed by 15 seconds of constant 850 mW/cm ² intensity, repeated twice	40 s
Pulse Cure	Consisting of 25 cycles in a single 20-second curing session, repeated twice. Each cycle has a total duration of 0.8 seconds, comprising an "on" period of 0.6 seconds at 850 mW/cm ² intensity and an "off" period of 0.2 seconds.	40 s



Figure 1. Prepared tooth surface



Figure 2. Preheating of composite resin

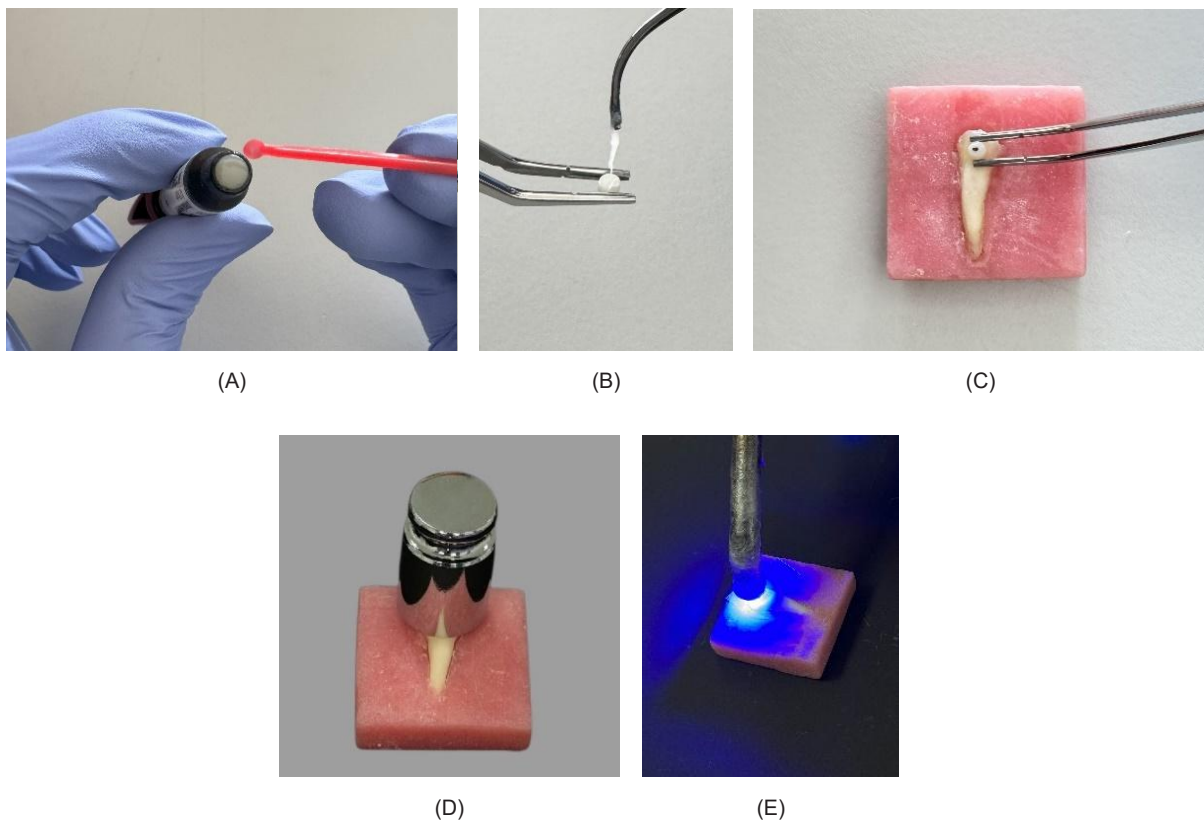


Figure 3. (A) Collection of luting material using a 1 mg micro scoop. (B) Application of luting material using a plastic instrument. (C) Placement of lithium disilicate on the prepared tooth surface. (D) Load application to standardize the luting material thickness. (E) Light curing of the preheated composite resin luting agent

: 1 ratio inside a stellan pot using a metal spatula. Once the resin reached the dough phase, it was placed into a metal mold measuring 3.5 cm × 3.5 cm × 1 cm, which had been coated with petroleum jelly. The teeth were carefully positioned before the acrylic resin hardened, ensuring that the buccal surface remained visible in a horizontal orientation (Figure 1). After complete polymerization, the

acrylic resin blocks were removed from the mold. Each group was assigned a numerical code to facilitate subject identification during shear bond strength testing.

The buccal enamel was reduced by 1.4 mm using a depth-cutting bur to expose the underlying dentin, followed by surface flattening with a separating disc and further smoothed with

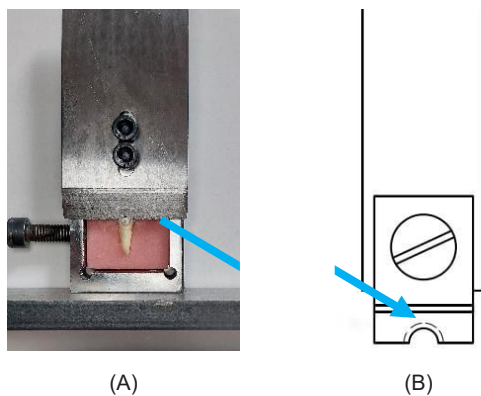


Figure 4. (A) Shear bond strength measurement using a universal testing machine (B) Illustration of the semicircular shear blade design (ISO 29022)

600-grit abrasive paper under running water, with 25 strokes applied over 30 seconds to achieve a uniform dentin surface. The prepared dentin was then etched with 35% phosphoric acid for 10 seconds, thoroughly rinsed, and gently dried. An adhesive system (Adper Single Bond 2 Adhesive, 3M ESPE) was applied to the treated dentin surface for 15 seconds, then gently air-dried using an air syringe for 5 seconds from a distance of 10 cm at a 45° angle to the prepared tooth surface. This was followed by light curing for 10 seconds using a light-curing unit, maintaining a separation of one cellulose strip thickness from the surface. The lithium disilicate discs were conditioned by etching with 9% buffered hydrofluoric acid (Porcelain Etch, Ultradent) for 90 seconds, then rinsed and dried. A silane (Silane, Ultradent) was applied to the etched lithium disilicate surface and left to air dry for 60 seconds.

The composite heater (Ena Heat, Micerium) was set to a temperature of 55 °C. Once the desired temperature was reached, syringes containing microhybrid composite resin (Filtek™ Z250 Universal Restorative, 3M) and nanohybrid composite resin (Filtek™ Z250 XT Universal Restorative, 3M) were placed into the designated slots of the device for 20 minutes (Figure 2).

The preheated composite resin was collected using a 1 mg micro scoop and applied to the fitting surface of lithium disilicate disc (Figures 3A and 3B). In Groups IA, IB, and IC, cementation was performed

using preheated microhybrid composite resin, while in Groups IIA, IIB, and IIC, cementation was performed using preheated nanohybrid composite resin. The lithium disilicate disc was placed onto the prepared tooth surface with preheated composite resin using tweezers (Figure 3C). A load of 200 grams was applied and maintained for 15 seconds to standardize the compressive force and luting agent thickness (Figure 3D). Excess luting material was then removed using a sharp plastic instrument. The working time for the luting agent application was 40 seconds.

The luting agents was immediately light-cured using a light-curing unit (LED-H, Woodpecker) perpendicularly to the lithium disilicate surface, with a curing distance of one layer of celluloid strip (Figure 3E). Groups IA and IIA were cured using the conventional light-curing technique. Groups IB and IIB were cured using the ramp light-curing technique. Groups IC and IIC were cured using the pulse light-curing technique. Light curing for all techniques was performed twice, resulting in a total curing duration of 40 seconds for each group. The light intensity of the curing unit for each light-curing technique was controlled using a radiometer (LM-1, Woodpecker).

All research samples were immersed in a container filled with artificial saliva at pH 6.8 for 24 hours at a temperature of 36.5°C. After 24 hours, all samples were removed, dried, and subjected to shear bond strength testing using a universal testing machine (Tensilon RTF-2350, A&D) at a crosshead speed of 0.5 mm/min (Figures 4A and 4B). The recorded shear bond force was measured in Newtons (N). This force was then divided by the bonded surface area of the lithium disilicate on the tooth to obtain the shear bond strength in megapascals (MPa). The obtained data were entered into SPSS version 26.0 and analyzed using a two-way ANOVA parametric test with a 95% confidence level ($\alpha = 0.05$).

RESULTS

The mean shear bond strength of lithium disilicate is presented in Table 3. The obtained data were

Table 3. Mean and standard deviation of shear bond strength (MPa) of lithium disilicate under different light-curing techniques and preheated composite resin luting agents

Type of preheated composite resin luting agent	Light-Curing technique		
	Conventional (A)	Ramp cure (B)	Pulse cure (C)
	Mean \pm SD	Mean \pm SD	Mean \pm SD
Microhybrid (I)	13.94 \pm 0.76	14.26 \pm 0.80	14.72 \pm 1.29
Nanohybrid (II)	11.23 \pm 0.56	11.57 \pm 0.94	11.95 \pm 0.74

Table 4. Results from the Shapiro-Wilk test and Levene's test

Type of preheated composite resin luting agent	Light-Curing technique		
	Conventional (A)	Ramp cure (B)	Pulse cure (C)
	Mean \pm SD	Mean \pm SD	Mean \pm SD
Microhybrid (I)	13.94 \pm 0.76	14.26 \pm 0.80	14.72 \pm 1.29
Nanohybrid (II)	11.23 \pm 0.56	11.57 \pm 0.94	11.95 \pm 0.74

Table 5. Results of the two-way ANOVA for the shear bond strength of lithium disilicate under different light-curing techniques and preheated composite resin luting agents

Independent Variable	df	F	p-value
Light-curing techniques	2	2.523	0.094
Type of preheated composite resin luting agent	1	100.210	0.000*
Interaction light-curing techniques and type of preheated composite resin luting agent	2	.008	0.992

subsequently subjected to a normality test using the Shapiro-Wilk test ($\alpha = 0.05$) and a homogeneity of variance test using Levene's test ($\alpha = 0.05$). The results of the normality and homogeneity tests are presented in Table 4. The normality and homogeneity test results indicated p-values > 0.05 , signifying that the data were normally distributed and homogeneous. Based on these findings, the data met the requirements for parametric analysis using a two-way analysis of variance (ANOVA). The results of the two-way ANOVA test are presented in Table 5.

The results of the two-way ANOVA for the light-curing technique variable showed a p-value of 0.094, indicating no significant effect of conventional, ramp cure, and pulse cure light-curing techniques on the shear bond strength of lithium disilicate ($p > 0.05$). For the variable of preheated composite resin luting material type, the p-value was 0.000, demonstrating a significant

effect of preheated microhybrid and nanohybrid composite resin luting agents on the shear bond strength of lithium disilicate ($p < 0.05$). No interaction was observed between the light-curing technique and the type of preheated composite resin luting material on the shear bond strength of lithium disilicate, with a p-value of 0.992 ($p > 0.05$).

DISCUSSION

The results of the two-way ANOVA test showed that conventional, ramp cure, and pulse cure light-curing techniques had no significant effect on the shear bond strength of lithium disilicate ($p > 0.05$). The variation in light-curing techniques in this study may not have been sufficiently different in terms of total light energy density to produce a significant change in the shear bond strength of lithium disilicate. The total light energy density is the product of light intensity and exposure time, expressed in joules per

square centimeter (J/cm^2).¹⁹ The total light energy density generated by the conventional light-curing technique was $34 \text{ J}/\text{cm}^2$, whereas the ramp cure technique produced $29.75 \text{ J}/\text{cm}^2$, and the pulse cure technique yielded $25.5 \text{ J}/\text{cm}^2$.

Minimum light energy density required to achieve optimal polymerization is $17 \text{ J}/\text{cm}^2$. If the applied energy density is less than $17 \text{ J}/\text{cm}^2$, the polymerization process will be incomplete, leading to an inadequate degree of monomer conversion. Consequently, some monomers will not be optimally incorporated into the polymer network. An insufficient degree of monomer conversion can compromise the mechanical properties of the composite resin.²⁰ In this study, the total light energy density for all light-curing techniques exceeded $17 \text{ J}/\text{cm}^2$. This indicates that although the three light-curing techniques (conventional, ramp cure, and pulse cure) employ different methods for modulating light intensity, if the total light energy density is sufficient and appropriately delivered at the correct wavelength, the degree of monomer conversion in the preheated composite resin adhesive will remain optimal. This ensures the formation of a polymer network with favorable mechanical properties, including maximal shear bond strength.

The findings of this study, however, do not align with those of Simamora et al, who reported that a light-curing technique with lower total light energy density can reduce polymerization shrinkage.²¹ A lower total light energy density slows down the polymerization process and extends the polymerization time interval, thereby prolonging the pre-gel phase. During this phase, composite resin exhibits viscoelastic properties, allowing greater mobility of monomers. A prolonged pre-gel phase facilitates polymer chain rearrangement, which helps compensate for contraction stress. Consequently, longer polymer chains are formed with minimal reduction in the intermolecular distance between monomers. This minimal decrease in intermolecular distance contributes to reduced polymerization shrinkage. Lower polymerization shrinkage enhances interfacial adaptation of the restoration, ultimately improving shear bond strength.^{15,16}

The results of the two-way ANOVA revealed a significant effect of the luting agent type on the shear bond strength of lithium disilicate ($p < 0.05$). The group luted with preheated microhybrid composite resin demonstrated a higher mean shear bond strength than the group luted with preheated nanohybrid composite resin. This difference may be attributed to compositional variations in each luting agent, particularly the type and quantity of the resin matrix and filler content.

Resin matrix contains functional groups of composite resin monomer molecules that are interconnected through hydrogen bonds. These hydrogen bonds are relatively weak and serve as linking agents that hold the functional groups of monomer molecules in place, thereby limiting the material's ability to flow. When composite resin is heated, it absorbs sufficient thermal energy to break the hydrogen bonds within the resin matrix. Heating the composite resin increases the kinetic energy of monomer molecules, allowing them greater freedom of movement. This enhanced molecular mobility facilitates material flow, ultimately improving its flowability.^{22,23} In this study, preheated microhybrid composite resin contained a higher proportion of resin matrix compared to the preheated nanohybrid composite resin. As a result, the number of hydrogen bonds disrupted due to heating is greater in the preheated microhybrid composite resin, leading to a more pronounced increase in flowability after heating. The enhanced flowability after heating can minimize the risk of gaps or discrepancies between the material and its substrate, thereby enhancing the interfacial adaptation of the restoration, which contributes to an increase in shear bond strength.

The results of this study are consistent with the findings of Kramer et al. (2016), which demonstrated that preheated nanohybrid composite resin reduces film thickness and viscosity by 25%, whereas preheated microhybrid composite resin exhibits a reduction of up to 70%.⁹ Goulart et al also reported that preheated microhybrid composite resin has a thinner film thickness compared to preheated nanohybrid composite resin.²⁴ A thinner film thickness

generally results in a stronger bond between the veneer and the tooth surface. This is because a thinner film thickness minimizes the formation of voids that could compromise bond strength. Additionally, occlusal forces from mastication are more evenly distributed from the veneer to the tooth. In contrast, a thicker film thickness tends to create variations in stress distribution, potentially leading to stress concentration at specific points, which may ultimately increase the risk of restoration debonding.²⁵

Nanohybrid composite resin used in this study contains TEGDMA monomers, which are added to the resin matrix to enhance flowability and facilitate clinical handling. However, the main drawback to it is an increase in the risk of polymerization shrinkage. The polymerization shrinkage of TEGDMA can reach 12.5%, which is higher than that of Bis-GMA at 5.2%.²⁶ Low-molecular-weight monomers such as TEGDMA can cause significant volumetric contraction during polymerization due to the formation of shorter intermolecular distances between monomers. High polymerization shrinkage poses clinical challenges, including microleakage. Microleakage can compromise shear bond strength by weakening the integrity of the adhesive bond.^{27,28,29}

Nanohybrid composite resin contains a higher filler content compared to the microhybrid composite resin. According to Yalcin et al. (2016), the filler content in composite resin influences how heat is transferred through the material. Fillers play a role in reducing thermal conductivity. Composite resin with a higher filler content loses its heating temperature more quickly, causing its viscosity to return to its original state more rapidly.³⁰ This condition leads to suboptimal viscosity of the luting agent, which compromises the interfacial adaptation of the restoration and subsequently reduces shear bond strength. These findings align with those of the study by Marcondes et al. (2020), which stated that the viscosity of preheated composite resin affects how long the material retains heat after heating. High-viscosity materials tend to have lower thermal conductivity and are unable to retain heat effectively, causing them to

lose temperature more quickly after heating. In contrast, lower-viscosity materials exhibit better flowability and higher thermal capacity, allowing them to maintain elevated temperatures for a longer period before gradually cooling down.³¹

Type of luting agent used in this study played a predominant role in enhancing the shear bond strength of lithium disilicate. This finding is consistent with that of Miljkovic et al. (2022), which stated that differences in composite resin composition are a more significant factor in determining shear bond strength than variations in light-curing techniques.¹⁴ The results of this study also reinforce that light-curing techniques are not always the primary determining factor in clinical outcomes.

A limitation of this study lies in the application procedure of preheated composite resin, which requires a rapid working time of 40 seconds. The working time of preheated composite resin varies between 30 seconds and 1 minute, depending on its composition. Once removed from the heating device, approximately 50% of the acquired temperature is lost within 120 seconds, and about 90% is lost within 300 seconds. Therefore, preheated composite resin must be applied immediately after removal from the heating device to maintain its optimal temperature.^{23,24,28} An alternative composite resin heating device has been developed to enhance material handling and extend the working time, utilizing a specialized gun-shaped heater. This device allows for rapid heating of the material without additional time needed to dispense the composite resin from the syringe. It enables a longer working time of up to 3 minutes, providing the operator with more time to accurately apply the material.³² Further research using this device is necessary to evaluate its effectiveness and reliability, ensuring greater clinical benefits.

CONCLUSION

Notwithstanding the limitation, this study has shown that conventional, ramp, and pulse light-curing techniques applied to both preheated microhybrid and nanohybrid composite resin luting

agents does not significantly affect the shear bond strength of lithium disilicate. Preheated microhybrid composite resin luting agents exhibits higher shear bond strength compared to preheated nanohybrid composite resin luting agents.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

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