Original Article

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Influence of preparation designs, restorative composite cements, and preheating on cementation force and cement thickness

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Objectives: To evaluate the effects of two ceramic overlay preparation designs and preheating of two restorative composite resins, Clearfil AP-X and Filtek Z250, on cementation force and cement thickness.
Materials and Methods: A total of 112 mandibular first molar resin dies for ceramic overlays were prepared, using either a retentive MOD design (n = 56) or a non-retentive anatomical design (n = 56). Overlays were milled from IPS e.max CAD. Specimens were divided into two groups (n = 28) for cementation force and cement thickness measurements. Each group was further subdivided by luting material (Clearfil AP-X or Filtek Z250), used at room temperature or after preheating to 68 °C for 5 minutes using a Calset device. Restorations were mounted perpendicular to the load axis on a universal testing machine. For thickness measurement, a constant vertical load of 50 N was applied, and the resulting cement thickness was measured. Cementation force was recorded as the load required to achieve a 50 µm cement thickness. Data were analyzed using three-way ANOVA followed by Tukey HSD test at a 0.05 significance level.
Results: The non-retentive design with preheated Clearfil AP-X demonstrated the lowest cement thickness (49.46 ± 12.92 µm), which was statistically significantly lower than those of the other groups ($p<0.05$), while other groups showed cement thicknesses ranging from 79 to 97 µm. Cementation forces ranged from 44 N to 74 N. The preheated Clearfil AP-X group, in both retentive (49.73 ± 7.74 N) and non-retentive (44.45 ± 6.92 N) designs, showed significantly lower cementation forces ($p<0.05$) compared to other groups.
Conclusions: Preparation design, composite type, and preheating significantly influenced cementation. The non-retentive design with preheated Clearfil AP-X showed the lowest cement thickness, approaching the 50 µm ISO threshold, and lower cementation force, highlighting the importance of material and preparation design in achieving thin film thickness and efficient seating for indirect restorations.

Keywords: cementation force, film thickness, overlay, preheated composite resin

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Introduction

The performance of indirect restorations is critically influenced by the characteristics of the luting agents and the cementation procedures. As the weakest component within the bonded complex comprising the restoration, cement, and tooth structure, the luting agent plays a pivotal role in determining the overall durability of the restoration. Current best practices for cementing indirect restorations recommend the use of resin-based cements, combined with adhesive protocols, to ensure a strong and durable bond between the restoration material

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and the tooth structure [1, 2]. In 1995, Besek *et al.* proposed the use of restorative composite resins as luting agents for ceramic inlays [3]. The key difference between resin cements and restorative composite resins lies in the ratio of inorganic fillers to organic matrix, which enhances the mechanical properties of the latter. Thus, restorative composite resins may be considered as luting agents for their improved strength, wider shade options, reduced polymerization shrinkage, and better wear resistance due to higher filler content. However, the higher molecular weight monomers and increased inorganic filler content in restorative composite resins result in higher viscosity compared to resin cements [4]. The high viscosity of restorative composite resins can increase film thickness and hinder proper seating of the restoration, potentially causing issues such as occlusal interference, loss of contact with adjacent teeth, food impaction, secondary caries, and periodontal disease. Thus, alternative methods, such as preheating composite resin before cementation, have been suggested to improve flowability and reduce film thickness [5]. Preheating significantly reduces their viscosity, although they often remain more viscous and exceed the 50 μm thickness limit of the ISO 4049 [6]. Many in vitro studies have recommended specific restorative composite resins for luting indirect restorations. Politano *et al.* [7] suggested Clearfil AP-X for its near-ideal viscosity at room temperature, while Magne *et al.* [8] used Filtek Z100, emphasizing its optimized viscosity for cementation. However, predicting the thickness of a heated composite resin film remains challenging, regardless of brand, filler shape, or filler content. This study aimed to investigate the effect of preheating restorative composite resins, used as luting agents, on cement thickness and cementation force in ceramic overlays with two distinct preparation designs. The first null hypothesis of the study was that the preparation design, type of restorative composite resins, and preheating conditions do not affect cement thickness. The second null hypothesis of the study was that the preparation design, type of restorative composite resins, and preheating conditions do not affect cementation force.

Materials and Methods

Master die preparation

A mandibular right first molar Ivorine™ tooth (Columbia Dentoform Corp, NY, USA) was used for preparation using two distinct designs for ceramic overlay restorations. Each prepared tooth served as an abutment and was embedded in auto-polymerizing acrylic resin (Palavit G, Kulzer GmbH, Hanau, Germany) within a plastic mold, positioned 5 mm below the cementoenamel junction. The two preparation designs included: (1) In the non-retentive (NR) group, tooth preparation involved an anatomical occlusal reduction of 1.5 mm at the central groove and cusp tips. The margins were prepared with internally inclined walls at an angle of approximately 45 degrees toward the tooth center, resulting in a butt joint finish line, as previously described in the literature [7]. (2) In the retentive (R) group, an MOD preparation was performed, featuring proximal boxes at half the intercuspal width, a 3 mm pulpal floor depth, 1.5 mm occlusal reduction, and 1 mm rounded shoulder margins. Representative designs are illustrated in Figure 1.

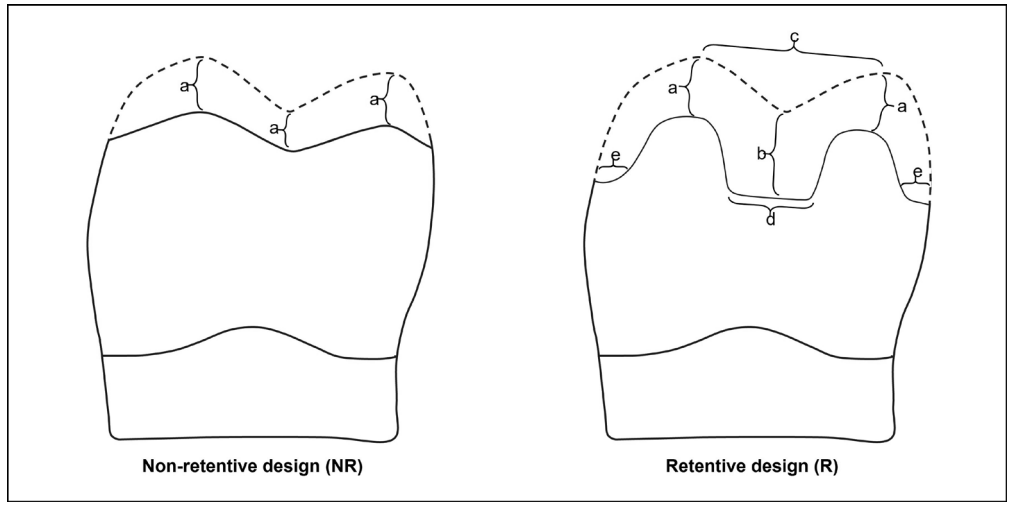


Figure 1 Schematic illustration of the two preparation designs used in this study: non-retentive (NR) and retentive (R) overlays. The dimensions are as follows: a = 1.5 mm, b = 3 mm, c = 7 mm d = ½ c mm, e = 1 mm

Duplication of the master die

Negative replicas of the prepared master die were created using duplicating silicone material (Deguform, DeguDent, Hanau, Germany). These negative replicas were filled with a commercial epoxy resin (EP089) and hardener (HP089) (Concrete Composite Co., Ltd., Bangkok, Thailand). A total of 56 positive replicas of each preparation design were created to ensure standardization. Specimens from each preparation design were divided into four subgroups based on two luting materials at room temperature and preheating conditions (n=14). To minimize duplication errors, a new silicone mold was used for every ten resin dies to prevent distortion or detail loss from repeated use. All dies were examined under ×3 magnification using a dental loupe to ensure replication accuracy. Inclusion criteria included well-defined preparation margins, smooth and continuous internal surfaces, and accurate occlusal anatomy reproduction consistent with the master die. Dies with voids, surface irregularities, marginal discrepancies, or dimensional distortion were excluded from further evaluation.

Design and manufacturing of the restorations

All ceramic restorations were fabricated using a CAD/CAM system. A plastic mandibular first molar was scanned with an intraoral scanner (CEREC Omnicam, Dentsply Sirona, Bensheim, Germany) to generate a biogeneric reference, ensuring standardized morphology resembling the original tooth prior to preparation. Each prepared master die was scanned, and restorations were designed using CAD software (inLab SW 4.2, Dentsply Sirona). A uniform cement space of 50 μm was set for the internal axial and pulpal surfaces, with the marginal region excluded and set to zero to ensure optimal marginal adaptation. Restorations were milled from lithium disilicate blocks (IPS e.max CAD, Ivoclar Vivadent AG, Schaan, Liechtenstein) using a milling machine (inLab MC X5, Dentsply Sirona). To assess marginal adaptation, restorations were tried in on their corresponding resin dies without any luting agent. The fit at the restoration–die interface was inspected circumferentially under ×3 magnification using a dental loupe. Specimens exhibiting visible gaps, rocking, or incomplete seating were excluded from further analysis.

Luting agents and cementation procedures

This study evaluated two restorative composite resins: Clearfil AP-X, and Filtek Z250. All procedures were conducted in a laboratory under controlled conditions at a room temperature of 25 °C. The internal surfaces of the restorations were treated with a ceramic primer (Clearfil Ceramic Primer Plus, Kuraray Co. Ltd, Tokyo, Japan) and air-dried after 60 seconds. An adhesive resin (Clearfil SE Bond X, Bond, Kuraray Co. Ltd, Tokyo, Japan) was then applied without light curing. For the preheating group, the resin composite was heated for 5 minutes at 68°C using a Calset device (AdDent, Danbury, Connecticut, USA). A standardized 150 mg portion of the composite resin was dispensed from the syringe prior to preheating, placed on the heating unit using a spatula, and covered with aluminum foil to prevent premature curing. The preheated composite was applied to the intaglio surface of the restoration within 1 minute. In the room temperature group, the composite was applied without preheating. The composition and properties of the adhesive, luting materials, and surface treatment agents used in the study are detailed in Tables 1.

Table 1 Materials used in the present study are based on manufacturers' specifications.

Material	Manufacturer	Composition	Filler load wt% (vol%)
Clearfil AP-X, shade A2 (Microhybrid composite)	Kuraray Co. Ltd, Tokyo, Japan	Matrix: Bis-GMA, TEGDMA Filler: Barium glass, Silica, Colloidal filler (0.04-17 μm)	86 (70)
Filtek Z250, shade A2 (Microhybrid composite)	3M ESPE, St.Paul, MN, USA	Matrix: Bis-GMA, UDMA, Bis-EMA, and TEGDMA Filler: Zirconia, Silica (0.01 - 3.5 μm)	78 (60)
Clearfil SE Bond X Bond (Light-curing adhesive)	Kuraray Co. Ltd, Tokyo, Japan	10-MDP, Bis-GMA, HEMA, Hydrophobic aliphatic dimethacrylate, dl-Camphorquinone, N,N-Diethanol-p-toluidine, Colloidal silica (1 - 100 nm)	n/a
Clearfil ceramic primer plus (Universal prosthetic primer)	Kuraray Co. Ltd, Tokyo, Japan	3-Methacryloxypropyl trimethoxysilane, 10-MDP, Ethanol	n/a

*n/a not available

Cement thickness measurement

Seven specimens from each group were allocated for cement thickness evaluation. Prior to cementation, each restoration was positioned on a universal testing machine (ElectroPuls E3000, Instron, Norwood, MA, USA) using a 3 mm stainless steel spherical tip aligned vertically with the center of the occlusal concavity. The resin die was rigidly secured to the lower base of the machine, while the ceramic restoration was attached to the spherical tip connected to the upper crosshead. A small amount of adhesive was applied between the tip and the occlusal surface to stabilize the restoration during loading (Figure 2a). While full internal adaptation was verified under 3× magnification dental loupe, localized discrepancies at the intaglio surface could not be quantitatively assessed due to the non-destructive nature of the method. Nevertheless, consistent seating under controlled conditions reduces variability and allows for reliable group comparison.

A baseline measurement was obtained by seating each restoration without any luting agent, under no applied force, using the universal testing machine to ensure precise and reproducible vertical positioning. The crosshead was gradually lowered until the restoration made initial passive contact with the die. This contact point—just before any measurable force was detected on the machine interface—was considered the baseline position. Passive seating was verified under 3× magnification dental loupe to confirm full adaptation without visible gaps. It should be noted that when the restoration was further lowered beyond this point, the machine began detecting positive force values, indicating the onset of active loading. Therefore, the baseline was defined as the closest vertical position where full seating occurred under zero load (Figure 2b).

The restoration was then elevated 2 cm above this reference point. After surface treatment, either preheated or non-preheated composite resin was applied to the internal surface (Figure 2c). Cementation was performed under a constant vertical load of 50 N at a speed of 1 mm/min for 2 minutes. The vertical seating displacement was automatically recorded by the testing machine, defined as the difference between the dry-fit reference and the final seated position (Figure 2d). This value, expressed in micrometers, was used to estimate overall cement thickness. The same loading protocol was applied consistently to all specimens to ensure reproducibility. Alignment, seating force, and speed were standardized across all groups to minimize variability.

Although this approach does not provide localized cement thickness measurements, it may serve as a non-destructive means of approximating overall cement space under standardized in vitro conditions

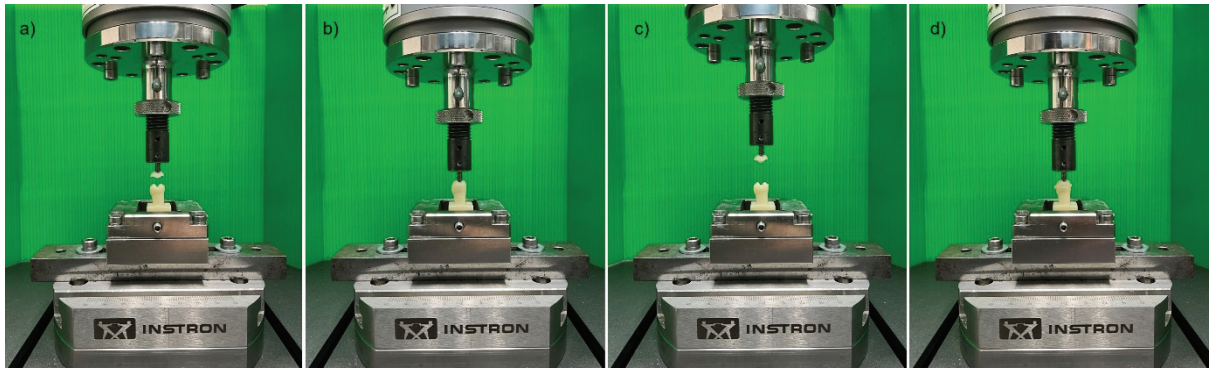


Figure 2 Cement thickness measurement protocol using a universal testing machine.
a) Specimen setup and stabilization. b) Baseline measurement. c) Restoration elevation, followed by surface treatment and luting agent application. d) Seating under 50 N vertical load.

Cementation force measurement

The remaining specimens (n = 7 per group) were used to evaluate cementation force. The same preparation and cementation protocols as in the cement thickness measurement were followed. Each restoration was mounted on a universal testing machine and luted with either preheated or non-preheated composite resin. A vertical load was applied using a universal testing machine (ElectroPuls E3000, Instron, Norwood, MA, USA) in displacement-controlled mode at a crosshead speed of 1 mm/min. The machine was programmed to stop once the restoration reached a vertical displacement of 50 µm from the zero-load (dry-fit) position. The force required to achieve this displacement was automatically recorded in newtons (N) and defined as the cementation force.

Statistical analysis

Statistical analysis was performed using IBM® SPSS® Statistics 23 (IBM SPSS Inc., Illinois, USA). The Shapiro–Wilk test was used to verify the normality of data distribution, while Levene's test confirmed the homogeneity of variances. A three-way ANOVA was conducted to evaluate the effects of preparation design, resin composite type, and preheating on cement thickness and cementation force. Post hoc pairwise comparisons were performed using the Tukey HSD test. A significance level of $\alpha = 0.05$ was applied for all statistical tests.

Results

The means and standard deviations for cement thickness and cementation force are presented in Figures 3 and 4. Three-way ANOVA revealed statistically significant differences in both cement thickness and cementation force among the groups ($p<0.05$), with a significant interaction between preparation design, resin composite type, and preheating condition ($p<0.05$). Tukey's HSD post hoc test was applied for pairwise comparisons, with statistically significant differences indicated by different letters in Figures 3 and 4. The non-retentive design cemented with preheated Clearfil AP-X demonstrated the lowest cement thickness, which was significantly lower than that observed in the other groups, whereas the remaining groups exhibited values ranging from 79 to 97 µm. Within the retentive design, no significant differences in cement thickness were found across different composite types or temperatures. However, for the non-retentive design, only the preheated Clearfil AP-X group showed a significant reduction in cement thickness. As a result, the first null hypothesis is rejected. Cementation force results indicated that preheated Clearfil AP-X required the lowest force in both preparation designs, with significant differences compared to other groups ($p<0.05$). The remaining groups used seating forces ranging from 61 to 74 N, with no significant differences observed between design, material, and temperature. Consequently, the second null hypothesis of the study is rejected.

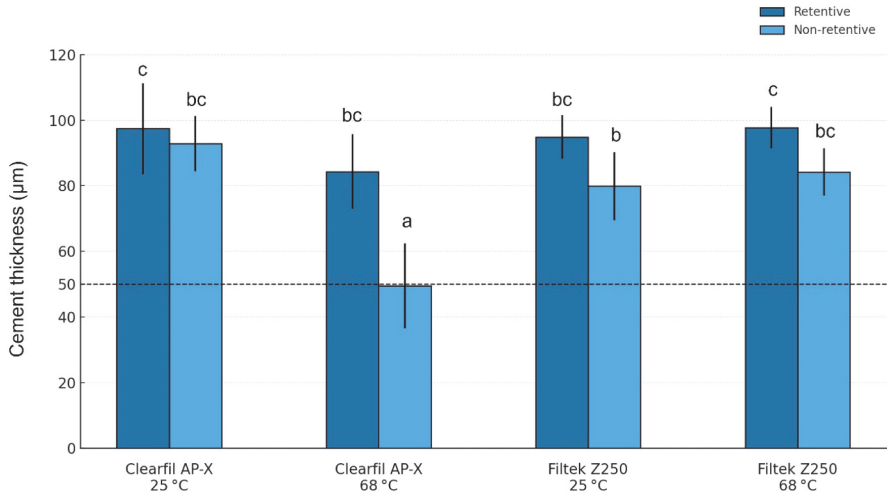


Figure 3 Mean cement thickness (µm) and standard deviations according to preparation design, resin composite, and preheating condition. Identical letters indicate no statistically significant difference (p>0.05). The dashed line indicates the ISO 4049 limit of 50 µm.

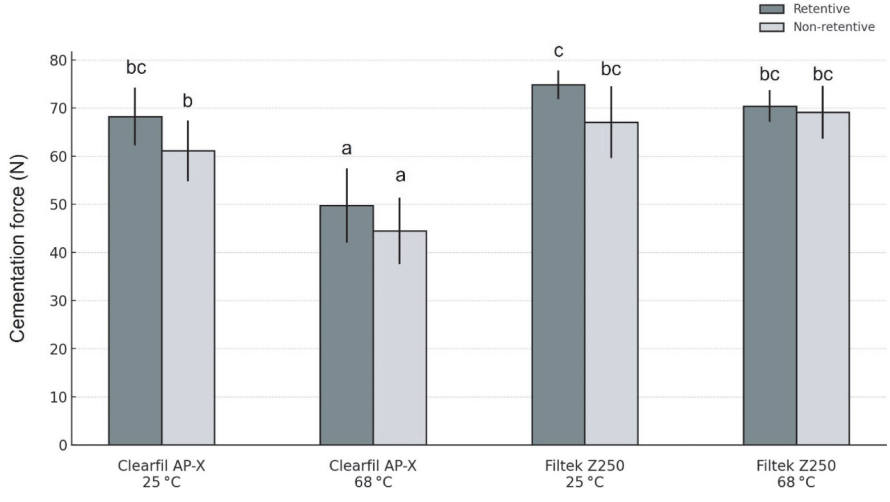


Figure 4 Mean cementation force (N) and standard deviations according to preparation design, resin composite, and preheating condition. Identical letters indicate no statistically significant difference (p>0.05).

Discussion

This study aimed to investigate the influence of preheating on the performance of restorative composite resins used as luting agents, with a focus on cement thickness and cementation force in ceramic overlays with varying preparation designs. Various methods have been used to measure cement thickness. Sectioning combined with optical or electron microscopy provides high-resolution measurements but is destructive and limited to selected sections. Micro-computed tomography (micro-CT) offers accurate, non-destructive three-dimensional analysis; however, it requires specialized equipment, is associated with high costs, and may introduce artifacts—particularly in the presence of radiopaque materials [9].

The present study adopted a universal testing machine to measure both cementation force and cement thickness. Notably, Magne *et al*. reported a similar approach, employing an electrodynamic testing system (Acumen III, MTS Systems) during the cementation of indirect restorations to achieve precise control and measurement of vertical seating [8]. Although this method does not yield detailed information on cement thickness at specific locations, thereby limiting a comprehensive evaluation of its distribution, it provides a practical, reproducible, and non-destructive means of assessment using a single device. It should be noted that cement thickness in this study was derived from overall vertical seating displacement, reflecting the integrated response of the restoration–die assembly rather than site-specific measurements. This inherent limitation should be considered when interpreting the findings.

In line with this simplified and focused methodology, surface pretreatment protocols were adjusted to minimize procedural variability and to isolate the effects of resin type, preheating condition, and preparation design on cement thickness. Although hydrofluoric acid (HF) etching is widely employed to enhance micromechanical retention by selectively dissolving the glassy matrix and creating surface irregularities [10], it was intentionally omitted in this study. The objective was not to evaluate bond strength, but to quantify cement thickness and cementation force under standardized surface conditions. Additionally, the use of HF presents substantial safety concerns due to its caustic and systemically toxic nature [11], and was impractical in this experimental setup, where specimens were mounted on a universal testing machine (UTM), limiting the ability to apply and rinse HF safely. To standardize chemical pretreatment under non-etched conditions, a ceramic primer and bonding agent were uniformly applied to all specimens to enhance surface wettability [12]. Nevertheless, omitting HF etching constitutes a methodological limitation. HF is widely recognized for its ability to increase the surface energy of silica-based ceramics, thereby improving wettability and promoting resin infiltration [13, 14]. In its absence, reduced surface energy may compromise the resin's ability to spread and adapt uniformly, potentially affecting the thickness and integrity of the cement layer. Although this approach minimized procedural variability and aligned with the primary aim of the study, it may not fully replicate clinical conditions where HF etching is routinely performed to optimize bonding performance. Furthermore, the use of HF in the cementation of resin cement to all-ceramic restorations under safety precautions due to its corrosive nature has been proposed as it has clinically relevant for achieving strong, durable bonds, which is critical to the success of these restorations [15].

The results of this study highlight the significant influence of preparation design, resin composite type, and preheating on both cement thickness and cementation force. Two restorative composite resins, Clearfil AP-X and Filtek Z250, both suggested for cementation of indirect restorations, were selected as luting agents in this study [7, 8]. The findings reveal that preheating the Clearfil AP-X composite significantly reduced cement thickness in the non-retentive design, achieving a value within the ISO 4049 threshold of 50 μm [16]. This effect was not observed in the retentive design or with the other tested groups (Figure 3). Preheating significantly enhanced the flowability of Clearfil AP-X, leading to lower cementation forces across both preparation designs. The cementation force for preheated Clearfil AP-X was notably lower than in the other groups (Figure 4). This reduction in cement thickness and force can be attributed to the increased flowability of the preheated composite, which facilitates seating and promotes a more

uniform cement layer [17]. High molecular weight, extensive hydrogen bonding, and increased chain branching in restorative composite resins typically hinder flow by causing polymer chain entanglement [18]. Preheating, however, provides sufficient thermal energy to disrupt these intermolecular interactions, improving molecular mobility and enhancing the flow during the cementation [19].

The lack of significant differences in cement thickness and cementation force among the groups using Filtek Z250, regardless of preheating, suggests that the material's inherent viscosity is less affected by temperature changes compared to Clearfil AP-X. This observation is consistent with previous studies reporting variability in the viscosity and flowability of composite resins upon preheating, with certain materials exhibiting greater sensitivity to temperature fluctuations [6, 17]. According to manufacturer data, Clearfil AP-X contains filler particles ranging from 0.04 to 17 μm, whereas Filtek Z250 incorporates smaller fillers ranging from 0.01 to 3.5 μm (Table 1). However, the relationship between filler characteristics and cementation outcomes remains inconclusive. Magne *et al*. reported that materials such as Simplishade, despite containing ultrafine filler particles (0.005–0.4 μm), exhibited relatively thick cement films (102.4 μm), suggesting that filler size alone is not a reliable predictor of film thickness [20]. Furthermore, the extent of film thickness reduction did not appear to depend on composite classification but varied among brands [19]. Nonetheless, conflicting findings have also been reported, suggesting that preheated Clearfil AP-X and Filtek Z250 may result in similarly minimal cement thicknesses and comparable spreading behavior under standardized loading conditions [20].

The materials exhibited distinct responses to preheating, highlighting differences in thermal sensitivity and handling characteristics. Preheating Clearfil AP-X resulted in significant reductions in cement thickness and cementation force, most notably in non-retentive preparations, whereas Filtek Z250 showed minimal changes under similar conditions. This contrast may be attributed to differences in composition and rheological behavior, as Clearfil AP-X appeared more responsive to thermal modulation [21, 22]. Although Filtek Z250 contains finer filler particles, typically associated with enhanced flow, its viscosity remained relatively stable, suggesting lower sensitivity to thermal changes [22]. These findings underscore that restorative composite resin can vary significantly in their clinical handling when used as luting agents, and such differences should be carefully considered during material selection and protocol planning [6, 17].

The divergent behavior between materials may reflect underlying variations in resin matrix composition and filler characteristics. According to Lee *et al*. [23], viscosity can differ substantially even among composites of the same class, influenced not only by filler content but also by filler–matrix interaction. These factors affect how the material responds to heat; preheating enhances polymer chain mobility within the matrix and reduces filler–matrix interlocking, leading to lower viscosity and improved flow under pressure. However, due to the proprietary nature of commercial products, detailed formulation data are often unavailable. Future studies using experimental composites with defined chemical profiles would help clarify the specific roles of monomer type and filler properties in thermal responsiveness and flow behavior.

Although conventional resin cements were not included in the present study, their clinical relevance warrants contextual comparison. Traditional resin cements generally exhibit lower viscosity and produce thinner film thicknesses than restorative composites, even when the latter are preheated. Marcondes *et al*. [6] found that all preheated restorative composites exceeded the ISO 4049 threshold of

50 µm, while flowable materials and resin cements typically remained below this limit. Another study reported that preheated composites generated luting films approximately 4.5 times thicker than those produced by dual-cure resin cements under identical conditions [24]. Similarly, Falacho *et al*. [5] demonstrated that resin cement consistently resulted in significantly thinner films than all tested composite resins, regardless of preheating or ultrasonic vibration. Only one group, which used a preheated and ultrasonically vibrated resin composite, produced a film thickness close to that of the resin cement. These findings underscore the differences in viscosity and film thickness between restorative composites and conventional resin cements, highlighting the need to adapt luting protocols accordingly. Additionally, it has been shown that achieving an optimal film thickness using preheated composites may be more technique-sensitive than when using conventional resin cements. A recent study demonstrated that clinically acceptable results with preheated composites required a higher level of operator experience, while dual-cure resin cements showed less variation between experienced and novice users [25]. This suggests that, although preheated composites can be effective, their clinical reliability may depend more heavily on the practitioner's skill, which further differentiates them from traditional resin cements in routine practice.

From a clinical perspective, cementation force influences film thickness, seating accuracy, and marginal adaptation. Finger pressure applied during manual cementation typically ranges from 8 to 88 N, with high variability among operators [26, 27]. In this study, a standardized load of 50 N, which falls within the clinically relevant range, was applied using a universal testing machine, consistent with previous in vitro protocols [28, 29]. The recorded forces across all groups ranged from 44 to 74 N, indicating that none exceeded typical manual application. Specimens cemented with preheated composite resin generally required lower seating forces, with some groups achieving values below the 50 N threshold. Although these differences were not statistically significant, non-retentive preparations exhibited a trend toward lower seating forces. This may be attributed to their simplified geometry, which facilitates smoother insertion and material flow. Previous studies have also demonstrated that less complex preparation designs are associated with improved marginal adaptation, reduced cement thickness, and better seating outcomes [8, 30]. Therefore, the present findings support the notion that both preparation design and material flowability, particularly when improved through preheating, may influence seating resistance and marginal fit during cementation.

Nevertheless, the literature lacks clear evidence of a definitive threshold for luting film thickness in indirect restorations, and no universal consensus has been reached regarding clinically acceptable values [31, 32]. Although ISO 4049 specifies 50 µm as a laboratory reference limit for resin-based luting agents, this criterion is primarily used for screening purposes and its clinical relevance remains debated. In this study, only the non-retentive group cemented with preheated Clearfil AP-X achieved a mean value approaching the ISO threshold. However, due to the observed standard deviation, it is likely that not all specimens within this group met the 50 µm limit. Indeed, 71.4% (5 out of 7) of the specimens in this group exhibited values at or below the threshold. Therefore, the clinical relevance of this finding should be interpreted with caution, as high variability may affect the consistency of seating and marginal adaptation. However, previous studies have suggested that cement thicknesses up to 100 µm may still be considered clinically acceptable [2, 31]. In this context, all experimental groups in the current study exhibited film thicknesses within

this broader range, suggesting potential clinical acceptability despite exceeding the ISO-defined standard. These findings indicate that preheated restorative composite resins, particularly Clearfil AP-X, may be beneficial in minimally invasive non-retentive preparations, where achieving a thin cement layer is advantageous. Although slight deviations from the ISO threshold may be acceptable in clinical settings, clinicians should strive to minimize cement thickness whenever possible, as thinner layers are generally associated with improved adaptation and mechanical performance [20].

Conclusion

This study demonstrated that preheating restorative composite resins can reduce cement thickness and cementation force in ceramic overlays, with the most pronounced effects observed in non-retentive preparations. These findings suggest that preheating may enhance material flow and improve seating. However, due to differences in composition and thermal responsiveness, careful selection of composite resins remains essential when using them as luting agents.

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