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Long-term stability of bond strength of preheated composite-based cementing agents. *In vitro*

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Abstract
Preheated composite resin emerged as a response to the degradation of dual resin cement over time, seeking to reduce the percentage of indirect restorations that failed due to restoration detachment.
Sixty Z250 composite resin cylinders (3M, USA) measuring 0.8 mm in diameter and 2 mm in length were attached to 15 Z350 resin sheets (3M, USA) in three experimental groups (n=20). RelyX Ultimate Clicker dual resin cement (3M, USA) was used for the control group, Z350 A2 composite resin (3M, USA) preheated to 60°C for 15 minutes was used for the preheated resin group, and Bulk Fill Flow fluid resin (3M, USA) was used for the fluid resin group. After 24 hours, 10 samples were randomly selected from each group and tested in a universal testing machine at a speed of 0.5 mm/minute. The remaining test specimens were aged in a Vítale class CD autoclave (Cristofoli, Brazil) for 6 cycles at 135°C at 0.23 MPa, to be tested. The data were collected in an Excel spreadsheet. The data were analyzed using ANOVA and T-tests.
The dual resin cement presented the highest absolute values both before and after aging, while the flowable resin showed the lowest values. The preheated resin showed a moderate reduction in bond strength, remaining in an intermediate range between the other materials.
Preheated resin does not have the highest adhesion values compared to other materials, but it does show good stability over time after aging.

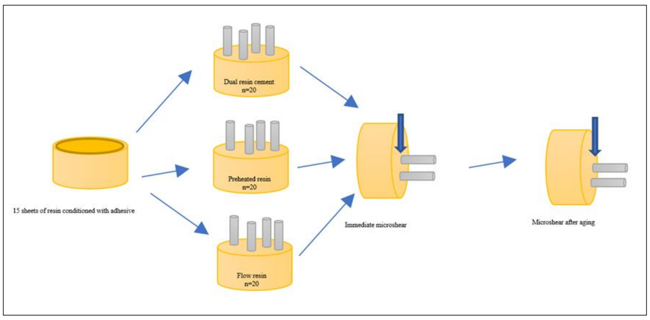
Keywords: Preheated resin, fluid resin, dual resin cement, microshear, aging, *in vitro* study

Introduction

Seeking to mimic the behavior of teeth, dental materials have evolved to acquire mechanical and aesthetic characteristics, with composite resin being the restorative element of first choice (Pereira *et al*., 2025; Poubel *et al*., 2024) [20, 22]. The filler particles aim to maximize aesthetics and flexibility while increasing fatigue resistance to better withstand masticatory forces. It offers faster fabrication compared to other materials since it does not require additional procedures such as firing, glazing, or crystallization, making it a durable and affordable option for indirect restorations (Hassanien & Tolba, 2024; Patussi *et al*., 2023) [10, 19].
On the other hand, the material most commonly used for cementing indirect restorations is dual-cure resin cement, since it does not need to be in the presence of light to continue the polymerization reaction; however, it has been observed that they tend to degrade and change color over time ("Influence of curing modes on thermal stability, hardness development and network integrity of dual-cure resin cements", 2021; Mazzitelli *et al*., 2022) [11, 17]. Therefore, other alternatives that offer better stability over time are being sought.
The use of flowable composites has expanded to other applications, such as the cementation of restorations. They offer advantages such as high filler content, shade variability, lower cost, as well as greater flowability and low viscosity, making them an affordable and easy-to-use material (Ashraf *et al*., 2025; Dikici *et al*., 2025; Hassanien & Tolba, 2024) [3, 4, 10].
Several studies suggest that using composite resins as a cementing agent has advantages due to their high inorganic filler content. To reduce their viscosity, they must undergo a heat treatment known as preheating, which helps the resin flow between the restoration and the tooth (Hassanien & Tolba, 2024; Patussi *et al*., 2023) [10, 19]. Furthermore, higher thermal energy results in greater movement of radicals and monomers, achieving better conversion and a higher polymerization rate (Elkaffas *et al*., 2019; Erhardt *et al*., 2020) [5, 6].
The literature has documented that the preheating range for composites is from 54° to 68°C; however, several studies agree that this is a safe temperature to avoid damage to the pulp tissue, considering it safe. (Kocha *et al*., 2024).

Methodology

A case study was conducted, of an experimental, cross-sectional, comparative, prospective, and *in vitro* nature. For the research, 60 resin cylinders were manufactured distributed on 15 resin sheets, using preheated resin, flowable resin, and dual resin cement as cementing agents. The sample size will be non-probabilistic and selected by convenience.



Graphic 1: Methodology diagram

The exclusion criteria included:

- Resin cylinders measuring 2mm in length
- Resin cylinders with bubbles
- Inclined resin cylinders
- Resin sheets with a rough surface

Sample Manufacturing

For the preparation of the 60 resin cylinders, a 2m long Tygon tube with an internal light of 0.8mm was used. With the help of a new #11 scalpel, the Tygon tube was cut to a length of 2 mm and stored in a sterile container.

Using a dam drill, the Tygon cutouts will be fixed onto a tile, and Z250 A3 composite resin (3M, USA) was packed with a North Carolina probe.

Each resin cylinder was light-cured using a Gran Valo halogen lamp for 20 seconds at an intensity of 1000 mW/cm² per cylinder, positioned 2.5 mm above the slab. Using a new scalpel, the plastic matrix was cut to extract the resin cylinders that met the exclusion criteria, and these were stored in a sterile jar.

A cylindrical aluminum mold, made to specific dimensions (5 mm internal diameter by 2 mm high), was used to manufacture the resin sheets. Z350 A2 composite resin (3M, USA) was packed into the mold, which had been previously insulated with glycerin and placed on a glass slab, using a condenser. Before light-curing, a 1 mm thick glass slide was placed on the mold. Each sheet was light-cured with a Gran Valo light (Ultradent, USA) for 20 seconds at an intensity of 1000 mW/cm². The 15 sheets that met the exclusion criteria were stored and randomly divided into 3 sterile containers.

All sheets were cleaned with 5% NaClO, rubbing for 10 seconds, then washed for 10 seconds and dried with oil-free air. Universal adhesive (3M, USA) was applied by actively rubbing for 20 seconds and airing for 5 seconds.

For cementation of the control group, RelyX Ultimate Clicker dual-cure resin cement (3M, USA) was used. Using the tip of an OMS probe, the cement was applied to the end of the resin cylinder, and then, with tweezers, it was placed onto the treated surface of the resin sheet. A Gran Valo lamp was used for 20 seconds at an intensity of 1000 mW/cm² per cylinder at a height of 2.5 mm. The samples were then immersed in distilled water pending testing.

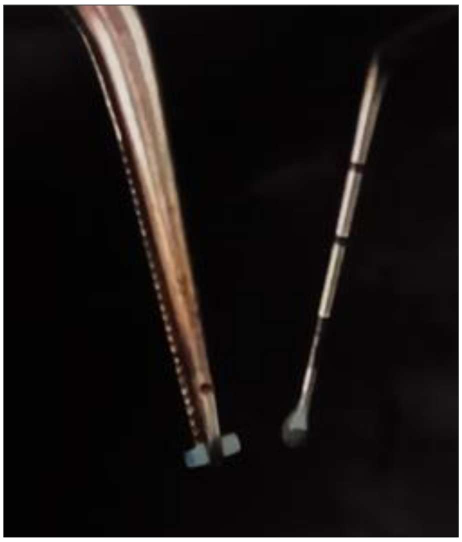


Image 1: Cementation with dual cement

For cementation of the RP, Z350 A2 composite resin (3m, USA) was preheated to 60°C for 15 seconds in a device. Using the tip of an OMS probe, the resin was distributed over the end of the resin cylinder and placed onto the treated surface of the resin sheet with tweezers. A Gran Valo lamp was used for 20 seconds at an intensity of 1000mW/cm2 per cylinder at a height of 2.5mm. The samples were then immersed in distilled water pending testing.

Image 2: Cementation with preheated resin

For cementation of the Bulk Fill Flow (3M, USA) fluidized resin group, the resin was applied to the end of the resin cylinder using the tip of an OMS probe. The cylinder was then placed on the treated surface of the resin sheet with tweezers and cured with a Gran Valo lamp for 20 seconds at an intensity of 1000 mW/cm² per cylinder at a height of 2.5 mm. The samples were then immersed in distilled water pending testing.

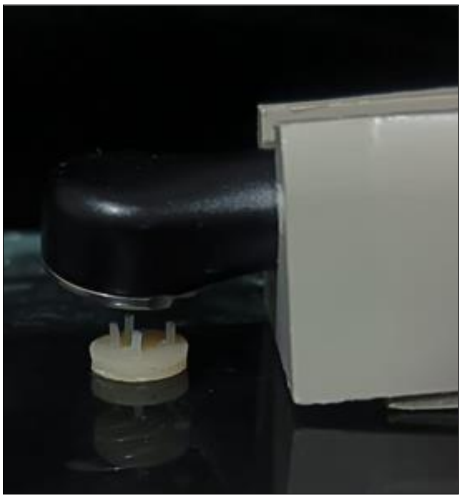


Image 3: Flowable resin cementation

After 24 hours, 10 random samples were selected from each group and tested on a universal testing machine at a speed of 0.5 mm/minute. The data were collected in an Excel spreadsheet (Microsoft, USA).

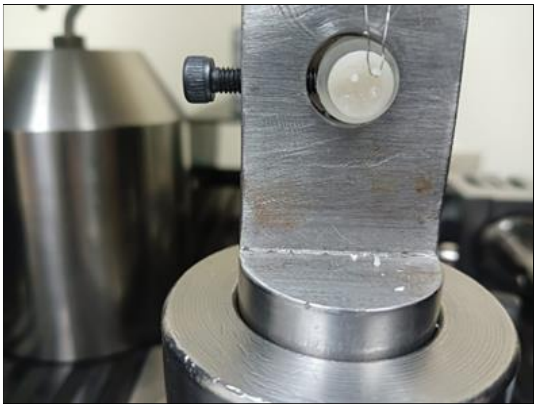


Image 4: Sample testing

The remaining test specimens were aged in a Vítale class CD autoclave (Cristofoli, Brazil) for 6 cycles at 135°C and 0.23 MPa, and then tested on a universal testing machine at a speed of 0.5 mm/minute. The data were collected in an Excel spreadsheet and analyzed using ANOVA.

Results

Three groups of n=20 test bodies were analyzed, of which 10 were tested immediately and 10 were tested after simulated aging.

Table 1 shows the mean bond strength values (MPa) obtained before aging for the three cementing agents evaluated. The dual-cure resin cement exhibited the highest bond strength (35.33 ± 4.32 MPa), followed by the flowable resin (26.7 ± 3.2 MPa), while the preheated resin registered the lowest values (15.94 ± 1.4 MPa). These results demonstrate differences in the adhesive performance of the materials, associated with their composition and polymerization mechanism.

Table 1: Bonding strength before aging

Material	Samples (n)	Mean ± SD (MPa)
Dual resin cement	10	35.33±4.32
Preheated resin	10	15.94±1.4
Flowable resin	10	26.7±3.2

Table 2 presents the bond strength values after artificial aging. A general decrease was observed in all three materials evaluated. Dual-cure resin cement maintained the highest values (26.25 ± 4.18 MPa), followed by flowable resin (21.63 ± 1.21 MPa), while preheated resin continued to show the lowest values (14.58 ± 1.67 MPa).

Table 2: Bonding strength after aging

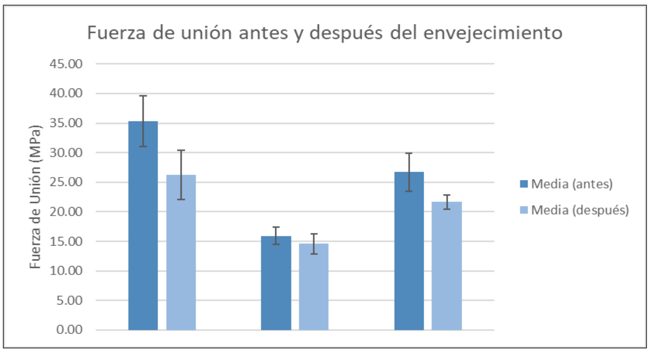
Material	Samples (n)	Mean ± SD (MPa)
Dual resin cement	10	26.25±4.18
Preheated resin	10	14.58±1.67
Flowable resin	10	21.63±1.21

The comparison of materials before and after aging, using Student's t-test, showed a statistically significant decrease in bond strength in the flowable resin ($p < 0.05$) and in the dual-cure resin cement ($p < 0.001$). In contrast, the preheated resin showed no statistically significant differences after aging ($p > 0.05$).

Table 3: Before and after comparison (t-test)

Material	Before (Mean ± SD)	After (Mean ± SD)	P
Dual resin cement	35.33±4.32	26.25±4.18	0.000035836
Preheated resin	15.94±1.4	14.58±1.67	0.043081800
Flowable resin	26.7±3.2	21.63±1.21	2,17881283

Figure 1 shows a decrease in bond strength for all three materials after aging. Dual-cure resin cement exhibited the highest absolute values both before and after aging, while preheated resin showed the lowest. Flowable resin showed a moderate reduction in bond strength, remaining in an intermediate range between the other materials.



Graphic 2: Bond strength (MPa) of the cementing agents evaluated before and after artificial aging

The two-way analysis of variance (Table 4) showed that both material type and aging significantly influenced bond strength ($p < 0.001$). Furthermore, a significant interaction was observed between material and aging ($p < 0.01$), indicating that the effect of aging was not uniform among the different cementing agents evaluated.

Table 4: Two-way ANOVA analysis

Factor	p
Material	0.000009136
Aging	0.000000000
Material interaction x aging	0.000501973

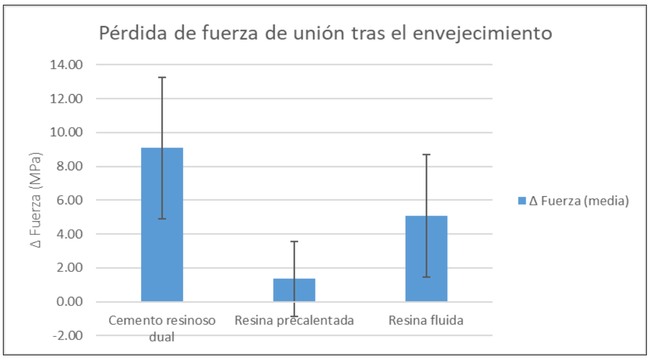
Table 5 summarizes the bond strength loss (Δ MPa) after aging. Dual-cure resin cement showed the greatest bond strength loss (9.08 ± 4.16 MPa), followed by flowable resin (5.07 ± 3.63 MPa). Preheated resin showed the least bond strength loss (1.35 ± 2.21 MPa), suggesting greater relative stability, although with lower absolute bond strength values.

Table 5: Loss of bond strength

Material	Δ Force (MPa) ± DE
Dual resin cement	9.08±4.16
Preheated resin	1.35±2.21
Flowable resin	5.07±3.63

It should be noted that, in some samples, isolated increases in bond strength were observed after aging. This behavior could be attributed to post-polymerization phenomena, water absorption with an initial plasticizing effect, or inherent variability in *in vitro* tests, without compromising the overall trend observed.

Figure 2 shows the magnitude of bond strength loss after aging. Dual-cure resin cement exhibited the greatest strength loss, followed by flowable resin. Preheated resin showed the least strength loss, indicating greater relative stability over time, although with lower absolute bond strength values.



Graphic 3: Loss of strength (MPa) of cementing agents after artificial aging

Overall, the findings of this study indicate that aging significantly affects the bonding strength of the cementing agents evaluated, with behavior dependent on the type of material.

Discussion

The present study was designed to evaluate the stability over time of the microshear bond strength of µSBS, which the Z350 composite presented preheated to 55°C, compared to the Bulk Fill Flow resin and the RelyX Ultimate Clicker dual cement, materials used as cementing agents between two resinous bodies, after being artificially aged for 4 years.

Once the samples were tested, it was observed that the highest bond strength values were found in the dual-cure resin cement group, with the lowest values for the flowable resin group followed by the preheated resin group. However, after aging, the preheated resin group showed the best stability over time, as there was no significant variance between the immediate and aged groups. The flowable resin group followed, and the dual-cure cement group showed the least stability, as its values showed the greatest discrepancy between the immediate and aged groups.

Previous similar investigations showed that the preheated Enamel Plus HRI resin (Micerium) had higher values compared to flowable resin and dual resin cement; all samples were aged before being tested on SBS, and the adhesion was performed on dentin (Akyle *et al*., 2024) [1]. When the bonding surface was changed to Lithium Disilicate, the highest results were obtained by the preheated Z350 composite group compared to the RelyXU200 cement and the preheated Proclinc composite (Hajjaj *et al*., 2025) [9]. Similarly, when comparing the bond strength of these materials to enamel, the highest values were observed when using a preheated Viscalor Bulk composite (Viscalor) as a cementing agent, compared to a RelyX Veneer dual resin cement (RelyX). Furthermore, in adhesion to feldspathic ceramics, both preheated Viscalor Bulk (Viscalor) and Z100 performed better than the dual resin cement (Raposo *et al*., 2025) [23]. Although there are countless resins that can be used in the preheating technique, it has been observed that high-filler resins tend to produce better results; according to a meta-analysis, the most commonly used in research is Z350 (Patussi *et al*., 2023) [19].

The microleakage when using a preheated resin is less than when using a resin cement; however, marginal sealing and adaptation are better when using resin cement due to its fluidity. It is known that after 2 minutes of heating, the resin loses 50% of its temperature, affecting its consistency and thickness (Alvarado *et al*., 2020) [2]. Ultrasonic vibration reduces the thickness of the preheated resin film used as a cementing agent (Falacho *et al*., 2022) [7]. reducing its thickness by between 21% and 49% (Marcondes *et al*., 2020) [16]. While another study observed that ultrasound did not significantly reduce the preheated resin film, and that subjecting the Z350 composite to this step decreased its mechanical properties, temperature was found to be a more effective variable (Porto *et al*., 2022) [21]. Although this step presents great relevance, the present study omitted it, choosing a small measurement for the study bodies (0.8 mm in diameter), given that microshear tests are more sensitive than macroshear tests and therefore present more reliable values (Ismail *et al*., 2021) [12].

Unlike our study, in the one by (Hassanien & Tolba, 2024) [10]. The flowable resin group showed lower bond strength values after aging compared to the resin cement group. When comparing SBS between flowable resin and self-etching cements, the self-etching cement exhibited the highest results (Omidi *et al*., 2025) [18]. The high-load flowable resin-based materials G-ænia Universal Flo performed excellently, with no significant difference when compared to dual-cure resin cement (Dikici *et al*., 2025) [4].

The choice of material for an indirect restoration depends not only on cost but also on the material's characteristics. Over a nine-year period, ceramic restorations had a higher survival rate compared to composite restorations (Spyropoulou *et al*., 2025) [24]. When comparing these materials, after aging the ceramic restorations showed better aesthetic results (Kalpana *et al*., 2024) [14]. However, it should be emphasized that composite resins have undergone significant changes, resulting in increasingly better properties. Their high percentage of filler particles reduces shrinkage during polymerization, providing greater wear resistance and enhancing their mechanical and aesthetic properties (Patussi *et al*., 2023) [19].

Previous studies observed that the stability of indirect resin-based restorations failed due to detachment of the restoration, followed by fracture of the restoration which led to secondary caries and finally tooth fracture (Josic *et al*., 2023) [13]. Therefore, it is presumed that the resin cement is the weakest segment in this type of technique, given that it is susceptible to greater wear and degradation (Galiatsatos *et al*., 2022) [8]. Showing similarity with the results of our study, indicating that the stability of the bond strength of this material tends to decrease over time.

Conclusions

Despite having low adhesion values, the preheated resin showed less degradation after aging, exhibiting the best long-term stability. The dual-cure resin cement proved to be a reliable material, although it showed greater degradation during the study period and achieved the highest adhesion values. The flowable resin demonstrated considerable long-term stability, making it a material that should be considered for further research.

Preheated resin does not have the highest adhesion values compared to other materials; however, it exhibits good stability over time after being subjected to aging.

Recommendations

It is recommended to conduct similar studies with a longer aging time in order to document the performance of the materials.

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