

PHYSICAL PROPERTIES OF MULTIPLE PREHEATED RESTORATIVE RESIN COMPOSITES FOR CEMENTATION

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ABSTRACT

This study evaluated the effects of repeated preheating cycles on the surface hardness and flexural strength of two resin composites: Filtek Z350XT and Filtek Z250. Each syringe was subjected to four preheating conditions: 0, 1, 10, and 20 cycles. In each cycle, resin composites were preheated to 54°C for 15 minutes, refrigerated overnight, and allowed to equilibrate at room temperature for 30 minutes before testing. The Vickers microhardness was measured on the top and bottom surfaces after 24 hours of dry storage, and the three-point bending test was evaluated after 24 hours of deionized water storage. Data were analyzed using a two-way mixed ANOVA ($\alpha = 0.05$). Commonly, the microhardness value at the top surface seemed unchanged for both resin composites. However, statistical analysis showed that the microhardness of Z350XT increased significantly after 1 and 10 cycles, while Z250 showed a significant increase only after 10 cycles. At 20 cycles, the top-surface microhardness was not significantly different from the control in either material. For bottom-surface microhardness, Z350XT remained comparable to the control across all cycles, whereas Z250 slightly decreased after 20 cycles compared with the control. The flexural strength increased significantly in Z350XT after one preheating cycle, while Z250 was not significantly affected by preheating cycles. In conclusion, repeated preheating up to 20 cycles did not adversely affect the microhardness or flexural strength of either material, except for the bottom-surface microhardness of Z250, which showed a slight decrease at 20 cycles compared with the control group.

Keywords: Repeated, Preheating, Resin composite, Hardness, Flexural strength

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INTRODUCTION

Resin composite is a popular restorative material in dentistry. In the process of improving their properties, manufacturers often focus on modifying the filler composition to enhance the mechanical properties of the material. However, while resin composites with a higher filler content generally exhibit superior mechanical properties, they also tend to have higher viscosity, which can result in poor adaptation to the cavity. To address these challenges, researchers have explored various techniques to reduce the viscosity of highly filled resin composites without compromising their mechanical properties. One promising approach involves preheating highly filled resin composites before application. Increasing the temperature of resin composites reduces their viscosity, potentially improving handling characteristics and cavity adaptation. Previous studies indicated that preheated resin composites exhibited reduced viscosity, enhanced degree of conversion, allowed for greater polymerization, and resulted in higher microhardness, potentially contributing to enhanced mechanical properties of the material (Rickman et al., 2011; Lopes et al., 2020; Elkaffas et al., 2019). Due to the modified properties of preheated resin composites, their application as luting agents for indirect restorations has gained interest in recent years (Rickman et al., 2011; Barbon et al., 2022). Preheating reduced the viscosity of the resin composite (Al-Ahdal et al., 2014; Lucey et al., 2010), which in turn led to a decrease in film thickness (Sampaio et al., 2017). Preheated resin composites have been used as luting agents for ceramic veneers and partial glass-ceramic posterior restorations (Marcondes et al., 2021; Van den Breemer et al., 2021). Furthermore, preheated resin composite significantly increased the microtensile bond strength of restorations when compared to resin cement (Goulart et al., 2018; Castro-Ramirez et al., 2024). However, the number of studies investigating the effects of repeatedly preheated resin composites remains limited. The effects of repeated heating on properties such as degree of conversion, microhardness, and flexural strength remain controversial. Therefore, these existing gaps and inconsistencies have generated interest in further examining the effects of multiple preheating cycles on resin composite properties.

LITERATURE REVIEWS

Overview of restorative resin composite

Resin composite consists of four key components. The organic matrix provides plasticity and allows for the curing process during polymerization. The inorganic fillers serve as reinforcement agents and enhance mechanical properties. The coupling agent ensures a strong bond between the organic matrix and inorganic fillers. Finally, the photo-initiator system is responsible for initiating the polymerization reaction upon light activation (Shen et al., 2021).

Overview of preheated restorative resin composite

The use of preheated resin composites for luting ceramic veneers and posterior partial ceramic crowns was reported in several studies due to their multiple advantages (Marcondes et al., 2021; Van den Breemer et al., 2021). During the preheating process for cementation, the resin composite was preheated either in its original syringe or in individual compules, depending on the clinical protocol and handling preference. A systematic review (Lopes et al., 2020) found that the average preheating temperature ranged from 54°C to 68°C. A preheating time of 15 minutes was the most recommended duration across multiple publications. Several studies have demonstrated that increasing temperature can enhance the degree of conversion of resin composites (Trujillo et al., 2004; Daronch et al., 2005). This may be explained by the increased thermal energy, which enhances the mobility of both monomers and free radicals, resulting in a faster polymerization rate and improved overall conversion efficiency.

Preheated resin composites have also been reported to exhibit higher microhardness than non-preheated resin composites (Elkaffas et al., 2019; Lucey et al., 2010). The increase in microhardness observed in preheated resin composites is attributed to enhanced polymerization

induced by the higher temperature. Previous research (Daronch et al., 2006) reported that microhardness was directly correlated with the degree of polymerization, further supporting the idea that preheating improves polymer conversion efficiency, leading to superior mechanical properties.

In addition, the effect of preheating on the flexural strength of resin composites appears to be material dependent. Some resin composites exhibited a reduction in flexural strength after preheating, whereas others showed an increase or no significant change (Bueno et al., 2025). The increase in flexural strength observed in certain preheated resin composites was attributed to the higher degree of conversion achieved at elevated temperatures (Trujillo et al., 2004).

Physical properties of repeatedly preheated resin composites: Degree of conversion

The degree of conversion of three types of resin composites after multiple preheating cycles was analyzed. Some resin composites showed a stable or increased degree of conversion in certain cycles, while others exhibited a decrease in degree of conversion. However, there was no statistically significant correlation between the number of heating cycles and the degree of conversion across all tested materials (Gebril et al., 2021).

Another study indicated that the degree of conversion slightly decreased with an increasing number of preheating cycles, possibly due to repeated heating affecting the photoinitiator system. However, preheating did not significantly impact the degree of conversion across different cycles (Safwat Mustafa, 2024).

Physical properties of repeatedly preheated resin composites: Microhardness

Vicker microhardness was evaluated after multiple preheated resin composites. The result found that repeated heating and cooling had a minimal effect on the microhardness of all materials tested. No correlation was found between the number of heating cycles and top surface hardness for any material. However, a significant positive correlation was observed between bottom surface hardness and the number of heating cycles for some materials (Gebril et al., 2021). Another study revealed that repeated preheating cycles did not significantly affect microhardness. But the type of composite resin significantly influenced these mechanical properties (D'Amario et al., 2015).

Physical properties of repeatedly preheated resin composites: Flexural strength

The three-point bending test was conducted to evaluate the flexural strength after repeated preheated nanohybrid resin composites. Repeated preheating cycles resulted in a slight reduction in flexural strength, which was not statistically significant, suggesting that the material retained mechanical integrity despite multiple heating cycles (Safwat Mustafa, 2024). Another study found that repeated preheating cycles did not significantly affect flexural strength, but the type of resin composite significantly influenced these mechanical properties, indicating that mechanical performance is more dependent on the composite formulation rather than preheating cycles (D'Amario et al., 2015).

Although some studies have reported a correlation between the degree of conversion and microhardness, most findings indicated that repeated preheating did not significantly affect the microhardness at the top surface, but had a greater influence on the bottom surface microhardness. Moreover, the effect of repeated preheating on the flexural strength also varied across different types of resin composites. These inconsistencies and gaps in current knowledge form the basis for the objectives of the present study.

The aim of this study was to investigate the effects of multiple preheating on the microhardness and flexural strength of two resin composites.

RESEARCH METHODOLOGY

Preheating resin composite

Two resin composites, Filtek Z350XT and Filtek Z250 (3M ESPE, St. Paul, USA), were investigated in this study. 10 syringes of Filtek Z350XT and 10 syringes of Filtek Z250 were used. Both materials were assigned to four experimental conditions according to the number of preheating cycles: 0, 1, 10, and 20 cycles. A composite warmer (AR Heater, HARBOR DENT, China) was used for preheating.

In the repeated preheating process, all syringes were placed entirely into the heating device, and every syringe underwent repeated preheating from 1 to 20 cycles. The experiment began with all syringes (10 syringes of Z350XT and 10 syringes of Z250) at their first use, before any preheating had been applied; this condition was defined as the 0-cycle group. The resin composite was first dispensed from each syringe to prepare specimens for microhardness and flexural strength testing for the 0-cycle group. After specimen preparation, all syringes were stored in a refrigerator overnight. On the following day, all syringes were removed from the refrigerator and allowed to equilibrate at room temperature for 30 minutes. Each syringe was then preheated by placing the entire syringe into the heating device at 54°C for 15 minutes, which was considered one preheating cycle. After removal from the heating device, all syringes were allowed to stand at room temperature for 30 minutes. The resin composite was then dispensed from each syringe to prepare specimens for microhardness and flexural strength testing for the 1-cycle group. After that, all syringes were again stored in a refrigerator overnight.

On the following days, the same preheating procedure was repeated for cycles 2 to 9 without preparing specimens for microhardness or flexural strength testing. On the day of the 10th cycle, all syringes were removed from the refrigerator, allowed to equilibrate at room temperature for 30 minutes, and then preheated at 54°C for 15 minutes by placing the entire syringe into the heating device. After preheating, the syringes were left at room temperature for 30 minutes. Composite was then dispensed from each syringe to prepare specimens for microhardness and flexural strength testing for the 10-cycle group. All syringes were subsequently stored in a refrigerator overnight.

The same procedure was then repeated for cycles 11 to 19 without specimen preparation for testing. On the day of the 20th cycle, all syringes were removed from the refrigerator, allowed to stand at room temperature for 30 minutes, and then preheated at 54°C for 15 minutes by placing the entire syringe into the heating device. After removal from the heating device, the syringes were again left at room temperature for 30 minutes. The resin composite was then dispensed from each syringe to prepare specimens for microhardness and flexural strength testing for the 20-cycle group. This completed the repeated preheating process. The procedures for specimen preparation and testing are described in the following section.

Microhardness test

For microhardness testing, one specimen was prepared from each syringe at each designated cycle. Therefore, in the 0-cycle group, 10 specimens of Z350XT and 10 specimens of Z250 were prepared. Similarly, in the 1-cycle, 10-cycle, and 20-cycle groups, 10 specimens of Z350XT and 10 specimens of Z250 were prepared for each group ($n = 10$ per preheating cycle for each material). To prepare the specimens, the resin composite from each syringe was applied into a silicone mold with a diameter of 6 mm and a thickness of 2 mm. A transparent polyester film was placed over the material, followed by a microscope slide to ensure uniform surface contact. The specimen was then light-cured using a 1200 mW/cm LED (Bluephase, Ivoclar Vivadent, Schaan, Liechtenstein) for 20 seconds at room temperature. After polymerization, the specimens were stored in a dry opaque box for 24 hours. Subsequently, microhardness testing was performed on both the top and bottom surfaces at three areas per surface, one at the center, and two additional points located 2 mm from the center, toward the

left and right sides, using a Vickers indenter with a 50 g load applied for 10 seconds. The mean microhardness value from the three indentations was then calculated and recorded.

Three-point bending test

For flexural strength testing, one specimen was prepared from each syringe at each designated cycle. Therefore, in the 0-cycle group, 10 specimens of Z350XT and 10 specimens of Z250 were prepared. Likewise, in the 1-cycle, 10-cycle, and 20-cycle groups, 10 specimens of Z350XT and 10 specimens of Z250 were prepared for each group ($n = 10$ per preheating cycle for each material). To prepare the specimens, the resin composite from each syringe was applied into a silicone mold with dimensions $25 \times 2 \times 2$ mm. A transparent polyester film was placed over the material, followed by a microscope slide to ensure uniform surface contact. The specimen was then light-cured for 20 seconds, with three overlapping exposures at room temperature. After polymerization, the specimens were stored in 37 °C deionized water for 24 hours. Subsequently, a three-point bending test was conducted using a universal testing machine with a crosshead speed of 0.75 mm/min, applying a downward force until the specimen fractures. The maximum load in Newtons (N) was recorded and converted to flexural strength (MPa).

Statistical analyses

The data were analyzed using a statistical software program (SPSS version 29.0, IBM Corp, Armonk, NY, USA). Statistical significance was set at $p = 0.05$. The top and bottom microhardness and flexural strength were analyzed using two-way mixed ANOVA. Mauchly's test was used to assess the assumption of sphericity; when violated, Greenhouse-Geisser corrections were applied. For significant main or interaction effects, post hoc comparisons within preheating cycles were performed using Bonferroni-adjusted pairwise comparisons.

RESEARCH RESULTS

Microhardness

Table 1 Two-way mixed ANOVA for microhardness at top and bottom surfaces

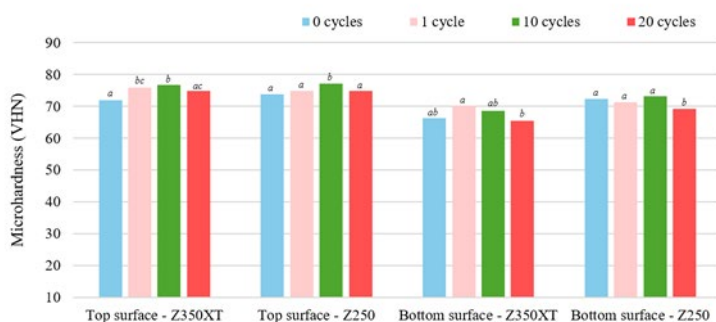
Surface	Effect	df	F	p-value	Partial η^2
Top	Preheating cycles	1, 789, 32.211	19.290	< 0.001	0.517
	Resin composite	1, 18	1.052	0.319	0.055
	Cycles $\times$ Resin composite	1, 789, 32.211	2.642	0.092	0.128
Bottom	Preheating cycles	3, 54	10.869	< 0.001	0.376
	Resin composite	1, 18	83.208	< 0.001	0.822
	Cycles $\times$ Resin composite	3, 54	4.468	0.007	0.199

At the top surface, the number of preheating cycles significantly affected microhardness; however, the resin composite type had no significant effect, and no significant interaction between preheating cycle and resin composite type was detected. At the bottom surface, both the number of preheating cycles and the resin composite type significantly affected microhardness, and a significant interaction between these factors was also found (Table 1).

Table 2 Microhardness at the top and bottom surface of each resin composite following different preheating cycles (VHN, mean $\pm$ SD)

Surface	Resin composite	Preheating cycles			
		0 cycle	1 cycle	10 cycles	20 cycles
Top	Z350XT	71.92 ^a $\pm$ 3.04	75.84 ^{bc} $\pm$ 1.13	76.62 ^b $\pm$ 1.53	74.81 ^{ac} $\pm$ 1.23
	Z250	73.81 ^a $\pm$ 1.04	74.81 ^a $\pm$ 1.55	77.09 ^b $\pm$ 0.82	74.75 ^a $\pm$ 1.49
Bottom	Z350XT	66.19 ^{ab} $\pm$ 3.00	70.30 ^a $\pm$ 1.69	68.49 ^{ab} $\pm$ 2.18	65.40 ^b $\pm$ 2.93
	Z250	72.32 ^a $\pm$ 2.20	71.32 ^a $\pm$ 1.31	73.11 ^a $\pm$ 1.91	69.16 ^b $\pm$ 1.63

Different superscript letters indicate significant differences among preheating cycles within a row (Bonferroni-adjusted, $p < 0.05$).

**Figure 1** Microhardness at the top and bottom surface of each resin composite following different preheating cycles

The microhardness for Z350XT showed an overall increasing trend from 0 to 10 preheating cycles. The 1 and 10 cycles exhibited a significant increase in microhardness compared with 0 cycles ($p < 0.05$), with no significant difference between 1 and 10 cycles. However, the microhardness in 20 cycles showed a significant decrease compared with 10 cycles ($p < 0.05$), while no significant differences were observed relative to 0 and 1 cycles. For Z250, microhardness showed a similar increasing trend from 0 to 10 preheating cycles. However, only 10 cycles demonstrated a significant increase in microhardness compared with 0 cycles ($p < 0.05$), whereas 1 cycle did not differ significantly from the control. The microhardness at 20 cycles significantly decreased compared with 10 cycles ($p < 0.05$), but showed no significant differences compared with 0 or 1 cycles (Table 2 and Fig 1).

At the bottom surface, the microhardness of Z350XT slightly increased after one preheating cycle compared with 0 cycles, although the difference was not statistically significant. When the material was preheated to 10 and 20 cycles, the microhardness decreased compared with 1 cycle; however, a significant decrease relative to 1 cycle was observed only at 20 cycles ($p < 0.05$). The microhardness at 20 cycles was slightly lower than that at 10 cycles, but this difference was not statistically significant, and neither the 10 nor 20 cycles differed significantly from 0 cycles. On the other hand, the microhardness of Z250 was comparable among 0, 1, and 10 cycles, with no statistically significant differences. However, after 20 preheating cycles, the microhardness significantly decreased compared with 0, 1, and 10 cycles ($p < 0.05$) (Table 2 and Fig 1).

Flexural strength

Table 3 Two-way mixed ANOVA for flexural strength

Effect	df	F	p-value	Partial η^2
Preheating cycles	3, 54	4.900	0.004	0.214
Resin composite	1, 18	30.730	< 0.001	0.631
Cycles $\times$ Resin composite	3, 54	3.223	0.030	0.152

Statistically, the flexural strength was significantly affected by preheating cycle and resin composite type, with a significant interaction between these factors (Table 3).

Table 4 Flexural strength of each resin composite at different preheating cycles (MPa, mean $\pm$ SD)

Resin Composite	Preheating cycles			
	0 cycle	1 cycle	10 cycles	20 cycles
Z350XT	130.51 ^a ± 15.44	149.13 ^b ± 13.82	139.21 ^{ab} ± 15.52	153.02 ^{ab} ± 10.91
Z250	154.86 ^a ± 7.58	164.91 ^a ± 9.22	159.06 ^a ± 10.63	153.99 ^a ± 14.20

Different superscript letters indicate significant differences among preheating cycles within a row (Bonferroni-adjusted, $p < 0.05$).

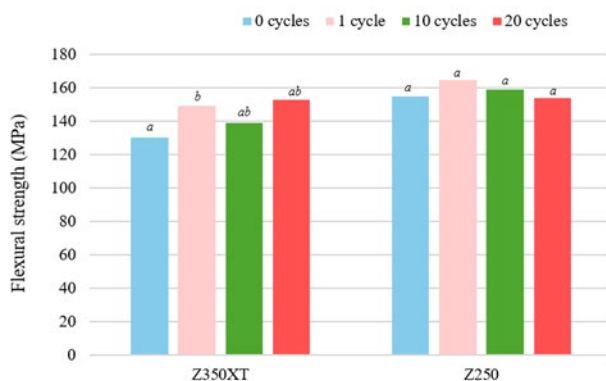


Figure 2 Flexural strength of each resin composite at different preheating cycles

The flexural strength showed a slight increasing trend after preheating in both materials. For Z350XT, flexural strength increased after 1, 10, and 20 preheating cycles compared with 0 cycles. In particular, the flexural strength after 1 cycle was significantly higher than that before preheating ($p < 0.05$). However, after 10 and 20 cycles, flexural strength decreased compared with 1 cycle. Nevertheless, no significant differences were observed among 1, 10, and 20 cycles. Although the flexural strength at 10 and 20 cycles remained higher than that at 0 cycles, these differences were not statistically significant.

For Z250, the flexural strength increased slightly after 1 preheating cycle compared with 0 cycles, but the difference was not statistically significant. After 10 and 20 cycles, the flexural strength decreased slightly compared with 1 cycle. However, no significant differences in the flexural strength across preheating cycles were observed for Z250 (Table 4 and Fig 2).

DISCUSSION & CONCLUSION

The present study was designed to simulate a clinical scenario in which an operator uses the same syringe of resin composite for both preheating during cementation of indirect restorations and for routine direct restorative procedures. The aim was to determine whether repeated preheating of a single syringe would influence the microhardness and flexural strength of the resin composite. Both microhardness and flexural strength specimens were stored for 24 hours before testing to allow for further polymerization (Ferracane, 1985).

The microhardness at the top surface was higher than at the bottom surface for both materials. This difference is attributable to light attenuation within the resin composite, which reduces irradiance reaching the bottom surface. The top surface, which receives higher irradiance, undergoes greater polymerization and achieves a higher degree of conversion (Daronch et al., 2006). In addition, a correlation between degree of conversion and surface hardness has been reported (Ferracane, 1985), further supporting the consistently higher microhardness observed at the top surface.

Statistical analysis revealed that the number of preheating cycles significantly influenced microhardness. For the top-surface microhardness, Z350XT showed a significant increase at 1 and 10 cycles compared with 0 cycles. Z250 exhibited a similar increasing trend in microhardness from 0 to 10 preheating cycles. However, only 10 cycles demonstrated a significant increase in microhardness compared with 0 cycles. At 20 cycles, both materials showed a similar pattern, with microhardness decreasing significantly compared with 10 cycles. Nevertheless, the microhardness values at 20 cycles remained slightly higher than those of 0 cycles, although the differences were not statistically significant. These findings differ from a previous study that reported no correlation between repeated preheating cycles and top-surface microhardness after heating up to six cycles (Gebril et al., 2021). Similarly, another study found that repeated preheating up to 40 cycles did not significantly affect top-surface microhardness (D'Amario et al., 2015).

For the bottom surface, Z350XT exhibited a slight increase in microhardness after one preheating cycle compared with 0 cycles. At 10 and 20 cycles, microhardness slightly decreased relative to 1 cycle, but the values remained comparable to those of 0 cycles. Overall, none of the Z350XT groups differed significantly from the 0 cycles group. In contrast, Z250 exhibited a significant reduction in the bottom surface microhardness only at 20 cycles compared with 0, 1, and 10 cycles. This material-dependent response is consistent with a previous study demonstrating that different resin composites may exhibit different microhardness responses to repeated preheating cycles (Gebril et al., 2021).

Regarding the flexural strength, Z350XT showed a slight increasing trend following repeated preheating; however, only 1 cycle significantly increased from 0 cycles. No significant differences were observed among 1 to 20 cycles. Z250 also demonstrated a slight increasing trend, but no group differed significantly from the control. These results align with previous reports indicating that repeated preheating does not significantly affect flexural strength in resin composites (Safwat Mustafa, 2024; D'Amario et al., 2015).

Previous studies have indicated that the microhardness and flexural strength are influenced by both the degree of conversion and the filler components of resin composites. In addition, the literature has consistently shown that increasing the temperature of resin composites prior to light curing increases the degree of conversion compared with curing at room temperature (Daronch et al., 2006). Moreover, surface hardness has been shown to correlate positively with the degree of conversion (Ferracane, 1985), and preheating has been associated with increased surface hardness in several studies. Evidence has also suggested that increases in flexural strength observed in certain preheated resin composites are attributable to the higher degree of conversion achieved at elevated temperatures. Accordingly, some studies have reported clear increases in microhardness and flexural strength following preheating.

Additionally, fillers are the principal components contributing to the microhardness and flexural strength of resin composites. The measured microhardness values were likely influenced predominantly by the filler phase because both materials contained more than 60 vol% filler. Likewise, fillers are considered major contributors to the flexural strength of these materials. Although preheating at 54 °C may affect certain monomers, the inorganic fillers are unlikely to be affected by this temperature. As a result, the filler particles likely remained stable, which may explain why the microhardness and flexural strength remained relatively unchanged.

However, an important methodological distinction exists between many previous studies and this study. Most prior investigations evaluated resin composites after a single preheating episode and typically prepared specimens and light-cured the material immediately after heating. In contrast, the present study focuses on repeated heating-cooling cycles; after heating, the material is allowed to return to room temperature for 30 minutes before specimen preparation, in order to specifically examine the effects of repeated heating-cooling rather than the effects of curing at an elevated temperature. A comparable experimental design has been reported, in which repeated heating followed by cooling to room temperature for up to six cycles produced minimal or no effect on the degree of conversion across four tested materials (Gebril et al., 2021).

Based on the present findings, it was observed that in both materials, after up to 20 preheating cycles, most of the results showed that the top- and bottom-surface microhardness did not decrease compared with those before preheating, regardless of whether the differences were statistically significant. The only exception was the bottom-surface microhardness of Z250 at 20 cycles, which was significantly lower than that before preheating. As previously reported, microhardness is directly correlated with the degree of polymerization (Daronch et al., 2006; Ferracane, 1985). This finding may be interpreted to suggest that the reduced microhardness at the bottom surface may, at least in part, be associated with a decrease in the degree of conversion. This, in turn, indicates that after 20 preheating cycles, Z250 may exhibit a reduction in depth of cure. Regarding flexural strength, the Z350XT group, after 1 preheating cycle, was the only group that showed a significant increase, which may be considered clinically favorable. For the other groups in both materials, neither material showed a statistically significant reduction in flexural strength after up to 20 preheating cycles compared with the pre-preheating condition. Therefore, from a clinical perspective, when preheated resin composites are used for cementation requiring repeated warming of the resin composite syringe, repeated use of these two materials up to 20 preheating cycles may not adversely affect microhardness or flexural strength in a clinically meaningful manner. However, caution should be exercised with Z250, as repeated preheating up to 20 cycles may compromise the depth of cure; therefore, particular attention should be paid to light-curing procedures and adequate depth of cure, whether the material is used for direct restorations or for cementation.

Within the limitations of this study, it can be concluded that repeated heating and cooling for up to 10 and 20 cycles did not adversely affect the microhardness or flexural strength of either material, except for the bottom-surface microhardness of Z250, which showed a slight decrease at 20 cycles compared with the control group. Clinically, caution should be taken when using Z250, as its depth of cure decreased after 20 preheating cycles.

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Data Availability Statement: The raw data supporting the conclusions of this article will be made available by the authors, without undue reservation.

Conflicts of Interest: The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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