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Laboratory Research

Effect of Repeated Preheating Cycles on Flexural Strength of Resin Composites

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Clinical Relevance

Highly repeated preheating cycles seem to negatively influence the flexural strength of resin composites. Assuming dental clinicians are aware they are using the same composite syringe for more than 20 cavities and a preheating procedure is steadily adopted, then the use of single-use composite compules instead of syringes should be adopted.

SUMMARY

The aim of this study was to assess the flexural strengths of three resin composites prepared at room temperature or cured after 20 or 40 cycles of preheating to a temperature of 45°C. Three resin composites were evaluated: Enamel Plus HFO (Micerium) (HFO), Enamel Plus HRi (Micerium) (HRi), Opallis + (FGM) (OPA). One group of specimens for each composite was fabricated under ambient laboratory conditions, whereas in the other groups, the composites were cured after 20 or 40 preheating cycles to a temperature of 45°C in a preheating device. Ten specimens were prepared for each group. A three-point bending test was performed using a universal testing machine at a crosshead speed of 0.5 mm/min. Data were analyzed with a two-way analysis of variance (ANOVA) test and a Games-Howell test ($\alpha = 0.05$). The two-way ANOVA showed that both the material and the number of heating cycles were significant factors, able to influence the flexural strength values ($p<0.05$). However, there was not a statistically significant interaction ($p>0.05$). For all three composites flexural strengths were not affected after 20 preheating cycles in comparison with the control groups (0 preheating cycles) but were, however, significantly decreased

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DOI: 10.2341/11-476-L

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Table 1: *Summary of the Resin Composites Tested*

Material (Group)	Shade	Composition	Total Content of Filler	Particle Size	Classification
Enamel Plus HFO (HFO)	UD3	UDMA, Bis-GMA, 1,4-butandioldimethacrylate; glass filler, highly dispersed silicone dioxide	75% by weight (53% by volume)	Glass filler: mean particle size of 0.7 μm; highly dispersed silicone dioxide: mean particle size of 0.04 μm	Microhybrid
Enamel Plus HRi (HRi)	UE2	UDMA Bis-GMA, 1,4-butandioldimethacrylate; surface-treated nano zirconium oxide particles with high refractive index (12% by weight); new type of filling glass with high refractive index (68% by weight)	80% by weight (63% by volume)	Glass filler: mean particle size of 1 μm; nano zirconium oxide particles: mean particle size of 20 nm	Nanofilled
Opallis + (OPA)	EA3	Bis-GMA monomers, Bis-EMA, TEGDMA, UDMA; barium-aluminum, silanized silicate, silicon dioxide, camphoroquinone, accelerators, stabilizers, and pigments	78.5% to 79.8% by weight (57% by volume)	Between 40 nm and 3.0 μm, with a mean particle size of 0.5 μm	Microhybrid

Abbreviations: UDMA: diurethane dimethacrylate; Bis-GMA: Iso-propyliden-bis (2(3)-hydroxy-3(2)-4(phenoxy)propyl)-bis (methacrylate) or bisphenol A diglycidyl methacrylate; Bis-EMA: bisphenol A diglycidyl methacrylate ethoxylated; TEGDMA: triethylene glycol dimethacrylate

when 40 prewarming cycles were conducted. The HRi and OPA groups had the highest flexural strengths, with no statistically significant differences among them. HFO presented significantly lower flexural strengths in comparison with HRi.

INTRODUCTION

Handling characteristics such as paste viscosity, packability, stickiness, and polishability play a critical role for composite resins used in restorative dentistry.[1] To achieve a perfectly sealed, long-lasting restoration, material adaptation to cavity walls is of primary importance.[2] In fact, recent literature suggests that there are benefits in increasing the flowability of composite resins by raising the temperature of the composite before placement and thus obtaining a better adaptation in the cavity.[3–14] Warming resin-based restorative materials prior to placement and contouring enhances composite adaptation to preparation walls by decreasing the viscosity of unpolymerized resin composite paste. Preheating may be achieved by placing compules, or syringes, of the resin composite material into commercially available preheating devices that operate at a temperature range of 39°C-68°C. Some *in vitro* studies using commercially available resin composites indicate a significant increase in conversion with an increasing curing temperature, as well as an increase in both polymerization and conversion rates seen at maximum cure rate.[3,4] However, in a recent *in vivo* study, Rueggeberg and others[15] showed that warmed composite lost heat quickly once removed from the heating device and inserted into a tooth preparation. This study indicated that the composite temperature (preheated using a 60°C preheating setting) remained only 6°C to 8°C above intraoral temperature once it was injected; only a slight increase in monomer conversion of preheated composite compared with that of room temperature (RT) material was recorded. From these findings, the authors suggest using the current preheating techniques, being aware of their limitations and with the intent to improve the ease of handling and composite placement.[15] Deb and others[14] showed that the cytocompatibility of composites after preheating remains unaffected. Many studies[8,11,14] disclosed that preheating protocols did not have any harmful effect on the mechanical properties of resin composite materials. However, all the *in vitro* studies in the literature have compared the me-

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Table 1: Extended.

Batch No.	Manufacturer
2009000372	Micerium, Avegno, Genova, Italy
2010009717	Micerium, Avegno, Genova, Italy
061208	FGM Produtos Odontológicos, Joinville, Brazil

chanical properties of resin composites cured at RT with those of the same materials cured after a preheating cycle to a determinate temperature. To the extent of the authors' knowledge, only one study analyzed the effect of repeated preheating and cooling cycles, as well as extended periods of preheating on composite cure.[2] This information could be of extreme importance because the same composite syringe can clinically undergo numerous preheating cycles before it is completely consumed. Daronch and others[2] reported that neither prolonged preheating nor repeated (10 continuous cycles of preheating and cooling) compule heating affected the degree of conversion of preheated composites compared with composites maintained at RT. On the basis of these findings, it seems interesting to assess whether the mechanical properties of cured composite can be affected if the number of preheating and cooling cycles are increased to the maximum number that a composite syringe is expected to clinically undergo.

The aim of this *in vitro* study was to assess the flexural strengths of three resin composites prepared at RT or cured after 20 or 40 preheating cycles to a temperature of 45°C. The formulated null hypotheses tested were that the flexural strengths would not be affected by 1) composite selection or by 2) preheating procedures.

METHODS AND MATERIALS

Three resin composites were evaluated in this study: Enamel Plus HFO (Micerium, Avegno, Genova, Italy) (HFO group), Enamel Plus HRi (Micerium) (HRi group), and Opallis + (FGM, Produtos Odontológicos, Joinville, Brazil) (OPA group). Their specifications are given in Table 1. One group of specimens of each material was fabricated under ambient laboratory conditions (21°C ± 1°C), whereas in the other groups the composites were cured after 20 or 40 preheating cycles to a temperature of 45°C in a commercially available preheating device (ENA HEAT composite heating conditioner, Micerium; batch no. SN C1102004).

Preliminary tests were carried out on the three materials to evaluate the heating and cooling times needed at RT (21°C ± 1°C). Temperature variations of the materials were monitored with a digital multimeter equipped with a temperature microprobe (GBC KDM 350, KON EL CO SpA, Milano, Italy). Maximum composite temperature attained was 48.5°C, with the preheating device preset to 55°C. However, after the first 12 minutes (time needed to heat the resin composites to a temperature of 45°C), further small increases in composite temperature required several minutes of heating. Following the results of these preliminary tests, the time needed to heat the resin composites to 45°C (about 12 minutes) was considered to be the most clinically acceptable time to allow the composite to reach a temperature as close as possible to that of the heating device. The same time was required to return the composites to 21°C. Then, each preheating cycle in this study consisted of 12 minutes composite heating in a heating device and 12 minutes of composite cooling at RT.

Ten specimens for each group (n=10) were then prepared using a stainless steel mold with the dimensions specified by the ISO 4049/2000 specification[16] (25 × 2 × 2 mm), positioned over a polyester strip. The materials were inserted into rectangular molds at RT (control groups) or after 20 or 40 preheating cycles. Resin composites were packed into the mold, covered by an acrylate strip, and smoothed with a glass slide to achieve a uniform surface finish. Three overlapping sections of the composite were light cured for 20 seconds with a curing light (Bluephase C8, with a 800 mW/cm^2 output, Ivoclar Vivadent AG, Schaan, Liechtenstein). The final temperatures of the composites before insertion into the mold were gauged with the digital multimeter (KON EL CO). The mean time between removing composite from the heating device

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Table 2: *Mean Values (Standard Deviations) Achieved in the Different Groups According to Heating Cycles*

		Groups			Total
		HFO	HRi	OPA	
Heating cycles	0	110.26 (19.73)	126.68 (25.27)	121.98 (10.88)	119.64[a] (20.12)
	20	112.72 (23.79)	128.76 (14.28)	116.01 (17.28)	119.16[a] (19.52)
	40	80.21 (24.59)	95.90 (25.69)	93.96 (6.51)	90.02[b] (18.74)
	Total	101.06[b] (24.59)	117.11[a] (26.46)	110.65[a,b] (17.11)	

[a] *Same superscripted letters indicate means that were not statistically different (p>0.05).*

and light polymerization was approximately 40 seconds for all tests. After irradiation, the excess material on the specimens was carefully removed with a scalpel blade. Specimen dimensions were measured using a digital caliper (series 500 Caliper, Mitutoyo America Corp, Aurora, IL, USA). The specimens were placed into deionized water at 37°C for 24 hours. A three-point bending test was then performed using a computer-controlled universal testing machine (LMT 150, LAM Technologies Electronic Equipment, Firenze, Italy) at a crosshead speed of 0.5 mm/min. The maximum loads were obtained, and the flexural strength (FS) was calculated in megapascals by using the following formula: $FS = 3FL / (2BH^2)$, where F is the maximum load (in newtons), L is the distance between the supports (in millimeters), B is the width of the specimen (in millimeters), and H is the height (in millimeters). The data were statistically analyzed. A two-way analysis of variance (ANOVA) test was performed to analyze the influence of the two factors under investigation (number of heating cycles and material) on the flexural strength mean values. Given that the homogeneity of the variances could not be assumed (Levene test), a Games-Howell test was chosen for post hoc multiple comparisons, with the significance level set at $\alpha = 0.05$.

RESULTS

The two-way ANOVA showed that both the material (p=0.006) and the number of heating cycles (p=0.000) were significant factors, able to influence the flexural strength values. However, there was not a statistically significant interaction between them (p=0.881). As a consequence, pairwise comparisons among the marginal means of the significant main effects were performed. Mean values achieved in the different groups, together with the marginal means and the statistical significance, are shown in Table 2. For all three composites, flexural strengths were not affected after 20 prewarming cycles in comparison with the control groups (0 preheating cycles) but were significantly decreased when 40 prewarming cycles were conducted. For the material, the HRi and OPA groups had the highest flexural strengths with no statistically significant differences among them. HFO presented significantly lower flexural strengths in comparison with HRi.

DISCUSSION

This study showed that flexural strengths of the three different composites tested were significantly affected by composite selection and by repeated composite preheating cycles. Therefore, both the null hypotheses of the present investigation have to be rejected. The three composites had a similar behavior: After 20 prewarming cycles flexural strengths were not affected if compared with the unheated group. However, when 40 prewarming cycles were conducted before light curing, the mean flexural strengths of the composites tested showed a significant decrease. In a clinical situation, the composite can be prewarmed to gain some benefits, such as increased flow and easier adaptation to the cavity.[3–14] The use of temperature to improve flow avoids some of the possible problems associated with a flowable resin material, such as the ongoing release of unreacted monomer and less favorable physical characteristics.[14] Fróes-Salgado and others[12] recently demonstrated that preheated composites showed better marginal adaptation compared with RT composites. Blalock and others[5] showed that the extent of flow varies between brands and resin composite classifications without any correlation

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between composite resin classification, filler content, or shape. The decrease in viscosity offered by preheated resin composite never reaches the low levels of a RT flowable composite.[5]

The results of the present study have an important clinical significance, given that there is a consensus in the literature on the absence of harmful effects of preheating procedures on the mechanical properties of resin composites.[8,12,14] Uctasli and others[8] showed that different preheating protocols (40°C, 45°C, or 50°C) do not have any harmful effect on the flexural strength and flexural modulus of tested composite materials. However, the majority of previous studies did not consider repeated preheating cycles. From this point of view, the present study is not in opposition to previous studies[8,12,14] because its findings also suggest that after 20 preheating cycles, the tested composites mechanical properties are not negatively influenced by the heating procedure. The present study is in accordance also with the findings from Daronch and others,[2] who reported that neither prolonged preheating nor 10 repeated continuous preheating cycles (cycles of 15 minutes from RT to 60°C) affected the degree of conversion of preheated composites. The study tested three different commercially available resin composites. For each composite tested, monomer conversion, either after repeated or extended preheating, remained equivalent to RT values, indicating that no resin polymerizable components were lost upon heating nor was there any degradation of monomer during the different heating treatments.[2] However, in clinical use, a standard composite syringe can be used to fill more than 20 cavities especially if a multi-shade layering technique is steadily adopted. From the results of the present study, in these cases the mechanical properties of the preheated resin composites would be decreased.

In this study, flexural strength was investigated to compare the composite groups. Flexural strength is a fundamental mechanical property for brittle materials, although the results cannot be directly extrapolated to the clinical behavior without considering some other aspects, namely flaw distribution[17] and the structural reliability of the material.[18] Nonetheless, the *in vitro* three-point bending flexural test is recommended by the ISO 4049/2000 specification[16] for polymer-based materials and is widely used for comparative purposes.[19,20] The resin-based composites tested showed significant differences in flexural results: the nanofilled resin composite (HRi) showed the highest mean flexural strengths. This is in contrast with other research studies[8,21] that reported higher flexural strength values for microhybrid resin composite compared with nanohybrid resin composite. The present results are probably related to the different filler loading of the composites tested (Table 2). It has been shown that resin composite filler volume fraction and filler load level have a strong correlation to the materials' strength and elastic modulus, as well as fracture toughness.[22,23] Kim and others[24] found that the mechanical properties of resin composites are related to their filler content. According to these findings, in the present study resin composite with the highest filler loading (HRi) exhibited the highest flexural strength; the composite with the lowest filler content (HFO) presented significantly lower flexural strengths in comparison with HRi.

In this study, a slightly lower composite temperature was achieved compared with that of the heating source. This would be expected, as already reported,[2] because composites are filled with inorganic particles and organic resins that function as thermal insulators. For this reason, it was decided to test the composites warmed to 45°C after a standard clinically acceptable preheating time.

In conclusion, from the results of this study it can be assumed that highly repeated preheating cycles seem to negatively influence the flexural strength of resin composites. If dental clinicians are aware that they are using the same composite syringe in more than 20 cavities with a steadily adopted preheating procedure, then the adoption of single-use composite compules instead of syringes would be considered preferable, according to the findings in this study.

Conflict of Interest Declaration

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 13 March 2012)

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