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Running title: repeated preheating procedure.

Clinical Relevance:

The mechanical properties of the resin composites tested were not influenced by repeated cycles of pre-heating to a temperature of 39°C. Dental clinicians can steadily adopt this pre-heating procedure without compromising the mechanical strengths of the heated composites.

SUMMARY

The aim of this study was to assess the flexural strength, flexural elastic modulus and Vickers micro-hardness of three resin composites prepared at RT or cured after 1 or repeated preheating cycles to a temperature of 39 °C. Three resin composites were evaluated: Enamel Plus HFO (Micerium) (HFO), Opallis + (FGM) (OPA), Ceram X Duo + (Dentsply DeTrey) (CER). For each trial, one group of specimens of each material was fabricated under ambient laboratory conditions, whereas in the other groups the composites were cured after 1, 5, 10, 20, 30 or 40 preheating cycles to a temperature of 39°C in a preheating device. Ten specimens were prepared for each group and for each trial. A three-point bending test was performed using a universal testing machine at a crosshead speed of 0.5 mm/min.

Three Vickers hardness (VH) indentations were carried out on each specimen for VH measurements. Data was statistically analyzed. The Two-Way-ANOVA tests showed that, regardless of the material, the number of heating cycles was not a significant factor and it was unable to influence the three mechanical properties tested. However, a significant main effect of the employed material on the marginal means of the three dependent variables was detected.

Keywords: Flexural modulus, Flexural strength, Mechanical properties, Preheating, Resin composite, Vickers hardness.

INTRODUCTION

Chairside warming resin-based restorative materials, prior to placement and contouring, is one of the recent trends in composite application. Preheating reduces viscosity and increases flowability, which facilitates better adaptation to cavity walls.[1,2] This may result in superior marginal adaptation,[3,4] may reduce microleakage and, thus, enhance the durability of restorations.[5,6] The increase in temperature of a composite enhances both radical and monomer mobility, resulting in a high degree of monomer conversion[7,8] as well as an improvement of polymerization rate.[9] As a result, more highly crosslinked polymer networking and improved mechanical and physical properties may be anticipated.[9] 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.[10] Some *in vitro* studies using commercially available resin composites indicate superior surface hardness and greater depth of cure for preheated composites.[1,11,12] However, in a recent *in vivo* study, Rueggeberg and others[13] showed that a warmed composite lost heat quickly once removed from the heating device and inserted into a tooth preparation. 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.

Many studies[1,2,14] 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 literature have compared the mechanical properties of resin composites cured at RT with those of the same materials cured after a preheating cycle to a determinate temperature. Only two studies analyzed the effect of repeated preheating and cooling cycles, as well as extended periods of preheating on composite cure.[10,15] This information could be of extreme importance because the same composite syringe can clinically undergo numerous preheating cycles before it is completely consumed. On these bases, it could be of high interest to assess whether the mechanical properties of a cured composite can be affected by repeated preheating cycles in a preheating device operating at 39°C, which improve the ease of handling and composite placement.

The aim of this *in vitro* study was to assess the flexural strength, flexural modulus and Vickers micro-hardness of three different resin composites prepared at RT or cured after 1, 5, 10, 20, 30 or 40 preheating cycles to a temperature of 39°C. The formulated null hypotheses were that mechanical properties would not show significant differences among 1) the different resin composites or among 2) the number of preheating cycles.

METHODS AND MATERIALS

Three resin composites were evaluated in this study: Enamel Plus HFO (Micerium, Avegno, Genova, Italy) (HFO group), Opallis + (FGM, Produtos Odontológicos, Joinville, Brazil) (OPA group), and Ceram X Duo + (Dentsply DeTrey GmbH, Konstanz, Germany) (CER group). Their specifications are given in Table 1. For each trial, 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 1, 5, 10, 20, 30 or 40 preheating cycles to a temperature of 39°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). The composites needed maximum 10 minutes to reach a temperature of 39°C. The same time was required to return the composites to 21°C. As a consequence, in this study each preheating cycle consisted of 10 minutes composite heating in a heating device and 10 minutes of composite cooling at RT.

Three-point Bending Test

Ten specimens for each group (n=10) were prepared using a stainless steel mold with the dimensions recommended by the ISO 4049/2000

specification (25 mm x 2 mm x 2mm) and positioned over a polyester strip.[10] The materials were inserted into rectangular molds at room temperature RT (Control Groups) or after 1, 5, 10, 20, 30 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. Overlapping sections of the composite were then successively light cured for 20 seconds (Bluephase C8, with a 800 mW/cm2 output; Ivoclar Vivadent AG, Schaan, Liechtenstein). The final temperatures of the composites before insertion into the mold were gauged with the digital multimeter (GBC KDM 350). The mean time between removing the composite from the heating device and light polymerization was approximately 40 seconds for all tests. After irradiation, any flash material on the specimens was carefully removed by gently abrading it with 320 grit abrasive paper. Specimen dimensions were checked again by measuring them with a digital caliper (series 500 Caliper; Mitutoyo America Corp, Aurora, IL). 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 (LLOYD LR 30K; Lloyd Instruments Ltd, Fareham, UK) at a crosshead speed of 0.5 mm/min and with 20 mm span distance; the load-deflection curves were recorded with PC software (Nexygen-Ondio Version 4.0, Lloyd Instruments Ltd). The fracture load

(F_{max}; N) of the specimens was measured. The flexural strength (σ; MPa) was calculated from the following formula:

$$\sigma = 3lF_{max} / 2bh^2$$

where l is the span distance (mm), b is the width (mm) and h is the height (mm) of the specimen.

The flexural modulus (E_{flex}; MPa) was calculated on the basis of the initial slope of the stress-strain diagram, according to the following formula:

$$E_{flex} = l^3F / 4bh^3d$$

where F (N) and d (mm) respectively are the applied load and the specimen deflection at any point on the initial straight-line portion of the load-deflection curve.

Vickers Hardness Measurement

For Vickers hardness (VH) evaluation, composite pastes were placed into cylindrical molds with a 10-mm inner diameter and 2 mm high. The materials were employed at room temperature RT (Control Groups) or after 1, 5, 10, 20, 30 or 40 preheating cycles (n=10). Composite layering was carried out in one single increment. To achieve in all samples flat and smooth top surfaces, the uncured paste was placed inside the mold in slight

excess and it was covered with a transparent polyester film followed by a microscope glass. Pressure was then applied to displace the excess material and light curing was performed through the glass for 40 seconds. The final temperatures of the composites before insertion into the mold were gauged with the digital multimeter (GBC KDM 350). The mean time between removing composite from the heating device and light polymerization was approximately 40 seconds for all tests. The obtained specimens were stored at room temperature in black film canisters for 24 h before subsequent procedures. Vickers hardness readings were recorded on the top smooth surface of the specimens. Vickers indentations were produced by applying a 1 N load for 10 s using a universal testing machine with a 500-N load cell (Lloyd LR 30K, Lloyd Instruments; Fareham, UK) provided with a standard 136° Vickers diamond indenter (item #17, Affri, Induno Olona; Varese, Italy).[16] Scanning electron microphotographs (EVO 50 XVP LaB6, Carl Zeiss; Cambridge, UK) were taken at different magnifications in order to measure the linear extent of the diagonal indentations (Figs 1 to 3). Subsequently, VH numbers were calculated according to the following formula:

$$VH = (1.854 \cdot F)/[(d_1+d_2)/2]^2$$

where d_1 and d_2 are the measured diagonals (mm) and F is the predetermined applied load expressed in kilograms-force (1.0204 Kg). For each specimen, the mean value of three VH readings performed at approximately 2 mm distance from one another was used as raw datum.

Statistical Analysis

Data were statistically analyzed. Two-Way-ANOVA tests were performed to analyze the influence of the two factors (Number of Heating Cycles AND Restorative Material) on the mean values of the three dependent variables under investigation (Flexural Strength, Flexural Modulus and the Vickers Hardness). Multiple comparisons were carried out according to the Tukey method. Values of p lower than 0.05 were considered statistically significant in all tests.

RESULTS

The Two-Way-ANOVA tests showed that, regardless of the Material, the Number of Heating Cycles was not a significant factor and it was unable to influence the Flexural Strength, Flexural Modulus and Vickers Hardness values. However, a significant main effect of the Material factor on the marginal means of the three dependent variables was detected. There was not a statistically significant interaction.

Mean values, marginal means and standard deviations achieved in the different groups are shown in Tables 2-4.

DISCUSSION

This study showed that the flexural strength, the flexural modulus and the Vickers hardness of the three composites tested were not significantly affected by the adopted repeated composite preheating technique. The composites had a similar behavior after 1-5-10-20-30-40 prewarming cycles to a temperature of 39 °C, in the sense that the mechanical characteristics were not affected if compared with the unheated groups. In a clinical situation, warming the composite reduces its viscosity, allowing the material to be injected into the preparation, rather than manipulating it into the preparation with hand instruments.[17] The warm composite technique allows handling characteristics similar to those of a flowable composite without sacrificing the benefits of superior mechanical, wear and polymerization shrinkage properties associated with the use of heavily filled restorative composite.[2] The reduced viscosity also allows for improved wetting of cavity walls compared with room temperature heavily-filled restorative composites. This in turn provides for improved adaptation to cavity walls and decreased gap formation.[3] Moreover, preheated light-curing composites are increasingly suggested as luting agents for porcelain veneers[18,19] or indirect composite restorations[20] in place of dual-curing materials.[21,22]

There is a general consensus in literature on the absence of harmful effects of pre-heating procedures on the mechanical properties of resin composites.[2,3,14] In a recent study, Osternack and others[23] concluded that composite hardness was not affected by pre-cooling or preheating procedures. However, the majority of previous studies did not consider repeated pre-heating cycles. Daronch and others[15] reported that neither prolonged preheating nor 10 repeated continuous preheating cycles (cycles of 15 minutes form RT to 60°C) affected the degree of conversion of preheated composites compared with composites maintained at RT. However in a recent study, D'Amario and others[10] concluded that highly repeated preheating cycles (40 preheating cycles to a temperature of 45°C) seem to negatively influence the flexural strengths of three commercially available resin composites; this seems to be the only study that takes into account more than 10 preheating cycles. Since 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, the authors concluded that the adoption of single-use composite compoules instead of syringes would be considered preferable if a preheating procedure to a temperature of 45°C is steadily adopted. In contrast, the present study showed that even highly repeated cycles of pre-heating to a temperature of 39°C did not negatively influence the mechanical properties of the resin composites tested. The effect of warming at 39°C in this study was considered

sufficient to obtain an increased flowability and a better adaptation of the composites. In contrast with other studies which reported a slightly lower composite temperature compared with that of the heating source,[10,15] in this study all the composites achieved a maximum temperature of 39°C after 10 minutes, with the preheating device preset to 39°C.

In conclusion, the preheating procedure tested did not negatively influence the mechanical properties of the resin composites even when highly repeated. Dental clinicians can steadily adopt this pre-heating procedure without compromising the mechanical strengths of the heated composites.

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TABLES AND FIGURES

TABLE 1. Summary of the resin composites tested.

Material (Group)	Shade	Composition	Total content of filler	Particles size	Classification	Batch n.	Manufacturer
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	2009000372	Micerium, Avegno, Genova, Italy
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 40nm and 3.0μm with a mean particle size of 0.5 μm	Microhybrid	80172310008	FGM Produtos Odontológicos, Joinville, Brazil
Ceram X Duo + (CER)	D3	Methacrylate modified polysiloxane, dimethacrylate resin; ---- Fluorescence pigment, UV stabilizer, stabilizer, camphorquinone, ethyl-4(dimethylamino)benzoate, barium-aluminium-borosilicate glass, methacrylate functionalised silicon dioxide nano filler, iron oxide pigments and titanium oxide pigments and aluminium sulfo silicate pigments	76% by weight (57% by volume)	Organically Modified Ceramic nano-particles (mean 2.3 nm) and nanofillers (mean 10 nm) combined with conventional glass fillers of ~1 μm	Nano ceramic	1112001219	Dentsply DeTrey GmbH, Konstanz, Germany

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.

TABLE 2. Flexural Strength Mean Values (Standard Deviations) Achieved in Different groups.

Flexural Strength (MPa)	***Heating Cycles***						
	0	**1**	**10**	**20**	**30**	**40**	**Overall**
HFO	104.6 (24.2)	102.0 (23.3)	104.8 (20.0)	111.5 (17.1)	106.1 (22.0)	84.5 (22.7)	102.2_{2} (22.4)
OPA	111.9 (18.0)	117.8 (25.1)	122.2 (16.4)	116.6 (24.3)	118.9 (17.8)	120.2 (19.9)	117.9_{1} (19.9)
CER	104.4 (23.5)	100.4 (15.3)	100.1 (18.2)	103.5 (23.2)	97.1 (22.6)	100.6 (16.0)	101.0_{2} (19.4)
Overall	107.0^{a} (21.6)	106.7^{a} (22.4)	109.0^{a} (20.1)	110.5^{a} (21.7)	107.4^{a} (22.2)	101.8^{a} (24.1)	

Same superscript lower-case letters indicate no statistically significant differences among the levels of the heating cycles (reading horizontally). Different subscript numbers indicate significant differences among the levels of composite employed (reading vertically).

TABLE 3. Flexural Modulus Mean Values (Standard Deviations) Achieved in Different groups.

Flexural Modulus (MPa)	***Heating Cycles***						
	0	**1**	**10**	**20**	**30**	**40**	**Overall**
HFO	6904.0 (979.6)	7327.9 (972.7)	6366.6 (807.6)	7072.4 (731.7)	6561.1 (802.1)	6811.2 (688.8)	6840.5_{2} (862.5)
OPA	6737.3 (894.6)	6576.1 (594.0)	6390.7 (557.4)	6343.2 (528.9)	6337.6 (1225.0)	6187.8 (1360.1)	6428.8_{3} (899.8)
CER	8376.6 (1015.2)	8486.0 (752.2)	8528.4 (1179.0)	8091.4 (1029.9)	8013.8 (832.4)	8079.5 (1118.5)	8262.6_{1} (978.6)
Overall	7339.3^{a} (1194.7)	7463.4^{a} (1103.2)	7095.2^{a} (1338.9)	7169.0^{a} (1055.4)	6970.8^{a} (1204.9)	7026.1^{a} (1323.1)	

Same superscript lower-case letters indicate no statistically significant differences among the levels of the heating cycles (reading horizontally). Different subscript numbers indicate significant differences among the levels of composite employed (reading vertically).

TABLE 4. Vickers hardness Mean Values (Standard Deviations) Achieved in Different groups.

VH	*Heating Cycles*						
	0	**1**	**10**	**20**	**30**	**40**	**Overall**
HFO	78.2 (5.8)	72.5 (8.6)	73.4 (9.4)	75.3 (5.1)	77.2 (5.8)	78.8 (8.2)	75.9 $_{1}$ (7.3)
OPA	64.1 (2.2)	66.4 (5.2)	66.6 (4.1)	68.6 (6.8)	70.9 (2.8)	70.0 (3.2)	67.8 $_{3}$ (4.7)
CER	70.1 (4,8)	72.5 (5,5)	71.3 (3,5)	71.1 (3,8)	70.5 (6,0)	75.8 (7,6)	71.9 $_{2}$ (5,4)
Overall	70.8 ^{a} (7.3)	70.4 ^{a} (6.9)	70.4 ^{a} (6.6)	71.6 ^{a} (5.8)	72.9 ^{a} (5.8)	74.9 ^{a} (7.4)	

Same superscript lower-case letters indicate no statistically significant differences among the levels of the heating cycles (reading horizontally). Different subscript numbers indicate significant differences among the levels of composite employed (reading vertically).

FIGURE 1. Scanning electron micrograph showing a VH indentation (a) and the measurement of its diagonals (b) on one specimen from the HFO group, 0 heating.

FIGURE 2. Scanning electron micrograph showing a VH indentation (a) and the measurement of its diagonals (b) on one specimen from the OPA group, 30 heating cycles.

FIGURE 3. Scanning electron micrograph showing a VH indentation (a) and the measurement of its diagonals (b) on one specimen from the CER group, 30 heating cycles.