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Influence of a Repeated Preheating Procedure on Mechanical Properties of Three Resin Composites

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Clinical Relevance

There is a lack of information about the effect of repeated preheating cycles on the mechanical properties of resin composites. It could be of high clinical interest to assess whether dental clinicians can steadily adopt preheating procedures without compromising composite mechanical strength.

SUMMARY

The aim of this study was to assess the flexural strength, flexural elastic modulus and Vickers microhardness of three resin composites prepared at room temperature or cured after one or repeated preheating cycles to a temperature of 39°C. Three resin composites were evaluated: Enamel Plus HFO (Micerium), Opallis (FGM), and Ceram X Duo (Dentsply DeTrey). 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, 10, 20, 30, or 40 preheating cycles to a temperature of 39°C in a preheating device. Ten rectangular prismatic specimens (25 × 2 × 2 mm) were prepared for each group (N=180; n=10) and subjected to a three-point bending test for

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DOI: 10.2341/13-238-L

flexural strength and flexural modulus evaluation. Vickers microhardness was assessed on 10 cylindrical specimens from each group (N=180; n=10). Statistical analysis showed that, regardless of the material, the number of heating cycles was not a significant factor and 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.

INTRODUCTION

Chairside warming of 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] 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 the polymerization rate.[9] As a result, more highly cross-linked polymer networking and improved mechanical and physical properties may be anticipated.[9] Daronch and others[7,9] calculated the conversion rate of a preheated composite and found that by heating the resin composites to 140°F (60°C), the conversion rate increased between 31.6% and 67.3% and therefore that less polymerization time was required. Deb and others[2] showed that the cytocompatibility of composites after preheating remains unaffected. 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. It is estimated that when a composite is heated up to 60°C and removed from the device, the temperature is reduced by 50% after 2 minutes and 90% after 5 minutes.[14] Therefore, it is clinically important to evaluate the influence of preheating under a nonisothermal condition, to simulate the real clinical scenario.[3]

Many studies[1,2,15] have 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 mechanical properties of resin composites cured at room temperature (RT) with those of the same materials cured after a preheating cycle to a determined temperature. Only two studies analyzed the effect of repeated preheating and cooling cycles as well as extended periods of preheating on composite cure.[10,14] 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 that improves the ease of handling and composite placement.

The aim of this *in vitro* study was to assess the flexural strength, flexural modulus, and Vickers microhardness of three different resin composites prepared at RT or cured after 1, 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. Specimens were fabricated for two different mechanical tests: 180 beam-shaped specimens were prepared for the three-point bending test (N=180), and 180 disc-shaped specimens were subjected to the Vickers microhardness (VH) indentation test (N=180). For each test, 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, 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

Table 1: *Summary of the Resin Composites Tested*

Material (Group)	Shade	Composition	Total Content of Filler	Particle Size	Classification	Batch Number	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, 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	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-aluminum-borosilicate glass, methacrylate functionalized silicon dioxide nanofiller, iron oxide pigments and titanium oxide pigments, aluminum sulfosilicate pigments	76% by weight (57% by volume)	Organically modified ceramic nanoparticles (mean 2.3 nm) and nanofillers (mean 10 nm) combined with conventional glass fillers of ~1 μm	Nanoceramic	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.

(GBC KDM 350, KON EL CO SpA, Milano, Italy). The composites needed a maximum of 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 of composite heating in a heating device and 10 minutes of composite cooling at RT. The same heating unit was used to heat all the composite syringes tested in this study.

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 × 2 × 2mm) and positioned over a polyester strip.[10] The materials were inserted into rectangular molds at RT (control groups) or after 1, 10, 20, 30, or 40 preheating cycles. Each preheating cycle consisted of 10 minutes of composite heating in the heating device set at 39°C and 10 minutes of composite cooling at RT. After the last heating cycle, the heated samples were immediately packed into the molds, 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 800 mW/cm^2 output; Ivoclar Vivadent AG, Schaan, Liechtenstein). Polymerization was performed by placing the curing unit tip in direct contact with the glass slide upper surface and perpendicular to the composite specimens. The proper output and the LED efficiency of the light-curing unit were checked every 10 samples using the built-in digital radiometer of a T-LED Anthos light-curing unit (T-LED; Anthos SRL, Imola, Italy). 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

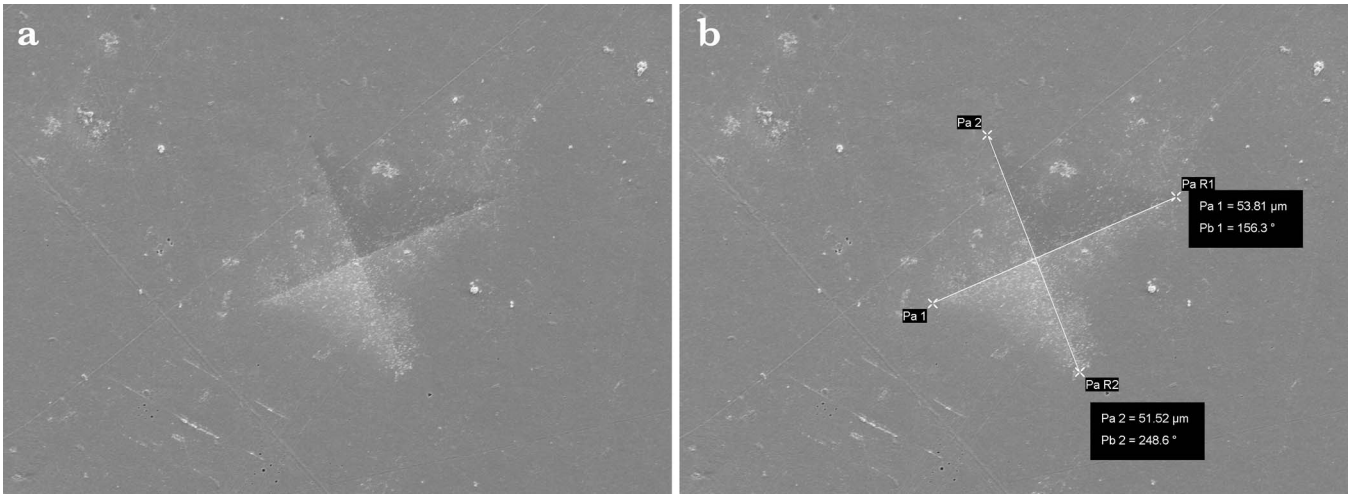

Figure 1. *Scanning electron micrograph showing a Vickers hardness (VH) indentation (a) and the measurement of its diagonals (b) on one specimen from the HFO group with no heating cycles.*

with 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 (LLOYD LR 30K, Lloyd Instruments Ltd, Fareham, UK) at a crosshead speed of 0.5 mm/min and with a 20-mm-span distance; the load-deflection curves were recorded with PC software (Nexygen-Ondio Version 4.0, Lloyd Instruments. The fracture load (N) of the specimens was measured. Flexural strength (MPa) and flexural modulus (MPa) were then calculated for each specimen.

VH Measurement

For VH evaluation, composite pastes were placed into cylindrical molds with a 10-mm inner diameter and 2 mm high. The materials were employed at RT (control groups) or after 1, 10, 20, 30, or 40 preheating cycles. With each one of the three resin composites under investigation, 60 samples were manufactured (N=180), 10 at RT and 10 for any group of 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 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 (800 mW/cm^2 output), maintaining the curing unit tip in direct contact with the glass slide upper surface and perpendicular to the composite specimens. The proper output and the LED efficiency of the unit were checked every 10 samples using the built-in digital radiometer of a T-LED. 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. The obtained specimens were stored at RT in black film canisters for 24 hours before subsequent procedures. VH readings were recorded on the top smooth surface of the specimens. Vickers indentations were produced by applying a 1 N load for 10 seconds using a universal testing machine with a 500 N load cell (Lloyd LR 30K, Lloyd Instruments) 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 (Figures 1 through 3). Subsequently, VH numbers were calculated considering the measured diagonals (mm), and the predetermined applied load was 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 analysis of variance (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

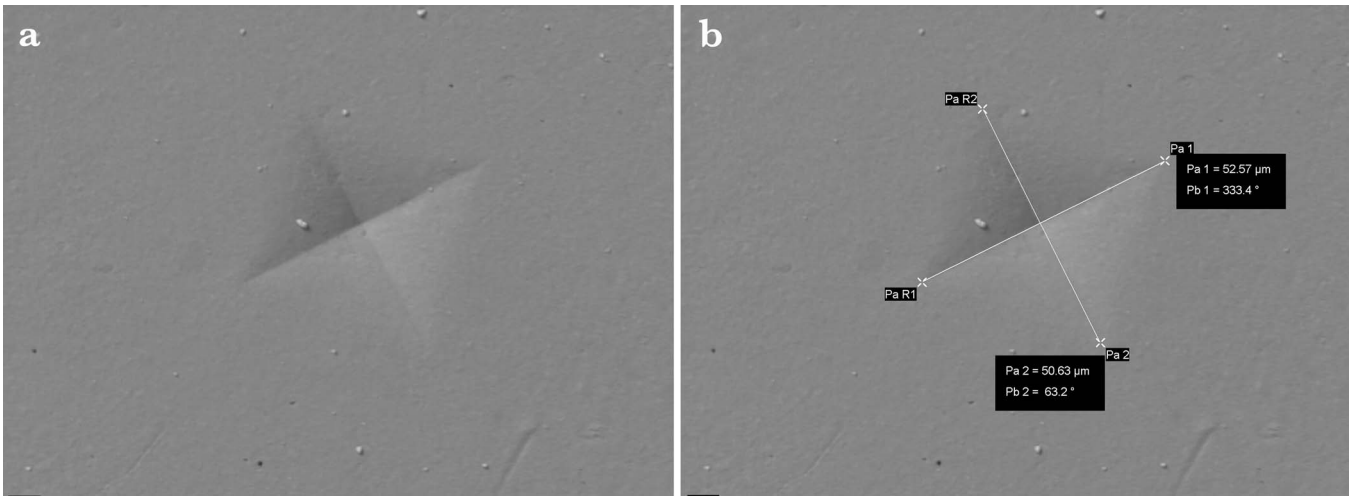

Figure 2. *Scanning electron micrograph showing a Vickers hardness (VH) indentation (a) and the measurement of its diagonals (b) on one specimen from the OPA group with 30 heating cycles.*

investigation (flexural strength, flexural modulus, and VH). Multiple comparisons were carried out according to the Tukey method. Considering each material separately, two-variable linear regression analyses were performed to investigate the presence of a linear relationship between the number of heating cycles and each mechanical property under evaluation. The number of cycles was assumed as the explanatory variable; the observed values for flexural strength, flexural modulus, and VH were the dependent variables. The sample regression function coefficients (intercept and slope) were calculated according to the ordinary least squares (OLS) method. ANOVA tables were computed to test the null hypothesis that the explanatory variable had no significant influence on each specific dependent variable; subsequently, the r^2 coefficient of determination (R^2) was calculated as the ratio between the regression sum of squares (RSS) and the total sum of squares (TSS) (R^2 = RSS/TSS). 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 was unable to influence flexural strength, flexural modulus, and VH values. However, a significant main effect of the material factor on the marginal means of the three dependent variables was detected. There was no statistically

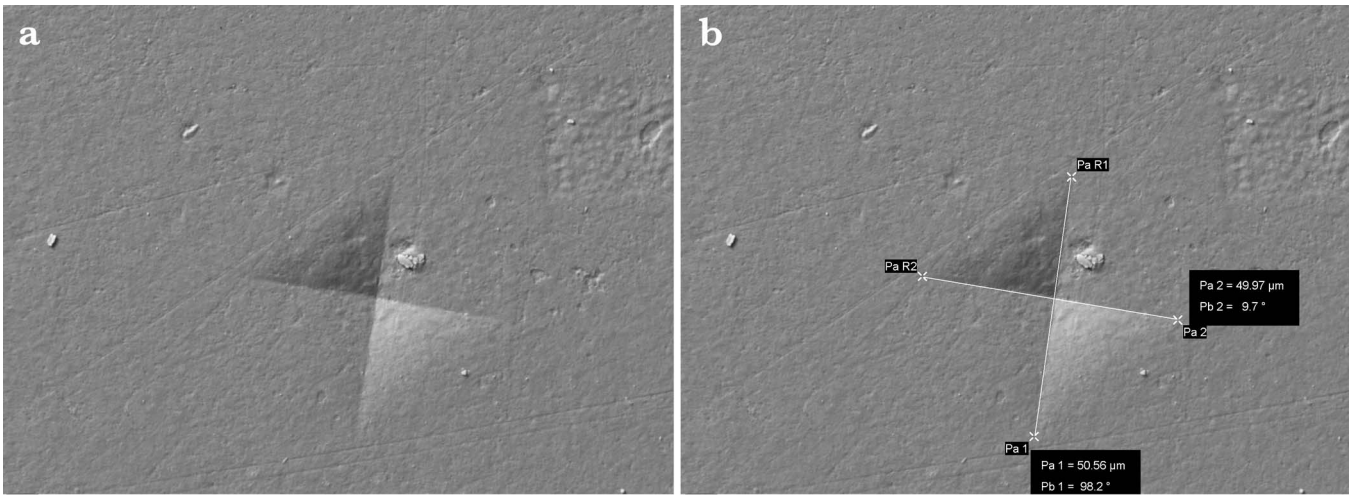

Figure 3. *Scanning electron micrograph showing a Vickers hardness (VH) indentation (a) and the measurement of its diagonals (b) on one specimen from the CER group with 30 heating cycles.*

Table 2: *Values of* p *Achieved From Two-Way Analysis of Variance Tests That Were Performed to Evaluate the Effect of the Two Factors (Material and Number of Heating Cycles) and of Their Interaction on the Mean Values of the Three Variables Under Investigation (Flexural Strength, Flexural Modulus, and Vickers Hardness)*[a]

	Flexural Strength	Flexural Modulus	Vickers Hardness
Factor	Material ($p<0.001$)	Material ($p<0.001$)	Material ($p<0.001$)
Factor	Cycles ($p=0.691$)	Cycles ($p=0.278$)	Cycles ($p=0.099$)
Interaction	Material × cycles ($p=0.532$)	Material × cycles ($p=0.814$)	Material × cycles ($p=0.572$)

[a] *Values of* $p<0.05$ *were considered statistically significant.*

significant interaction (Table 2). Mean values, marginal means, and standard deviations achieved in the different groups are shown in Tables 3 through 5.

Following regression analysis, the observed R^2 values were generally rather low, ranging between 0.003 and 0.209. All the R^2 values are given in Figure 4 together with the graphical representation of the corresponding sample regression functions.

The ANOVA tables for the performed simple linear regression analyses showed that almost all the calculated regression functions were not able to adequately account for the observed variability in the dependent variables ($p>0.05$). Concerning the VH in the OPA group, a statistically significant regression function was detected ($p=0.002$): its coefficients were 65.324 (intercept) and 0.144 (slope).

DISCUSSION

This study showed that the flexural strength, the flexural modulus, and the VH of the three composites tested were not significantly affected by the adopted repeated composite preheating technique. Within the predetermined confidence level set at 95%, only the VH variability observed for the OPA group could be statistically correlated to the increasing number of preheating cycles using a linear regression function. It should not be neglected, however, that the calculated slope (0.144) was very close to zero, leading to an almost horizontal regression line. According to this model, therefore, a great number of heating cycles would be necessary to determine even a small change in the VH values. Concerning the other mechanical properties tested and all the remaining resin composites under investigation, it was not possible to determine regression functions that could adequately correlate the observed variability to the number of preheating cycles.

The composites had a similar behavior after 1, 10, 20, 30, and 40 prewarming cycles to a temperature of 39°C in the sense that the mechanical characteristics were not significantly affected if compared with the unheated groups. Studying the effect of prewarming on mechanical properties can provide useful information to practitioners who are considering using this technique to increase flowability of composite materials. 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

Table 3: *Flexural Strength Mean Values (Standard Deviations) Achieved in Different Groups*[a]

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

[a] *Same letters indicate no statistically significant differences among the levels of the heating cycles (reading horizontally). Different superscript numbers indicate significant differences among the levels of composite employed (reading vertically).*

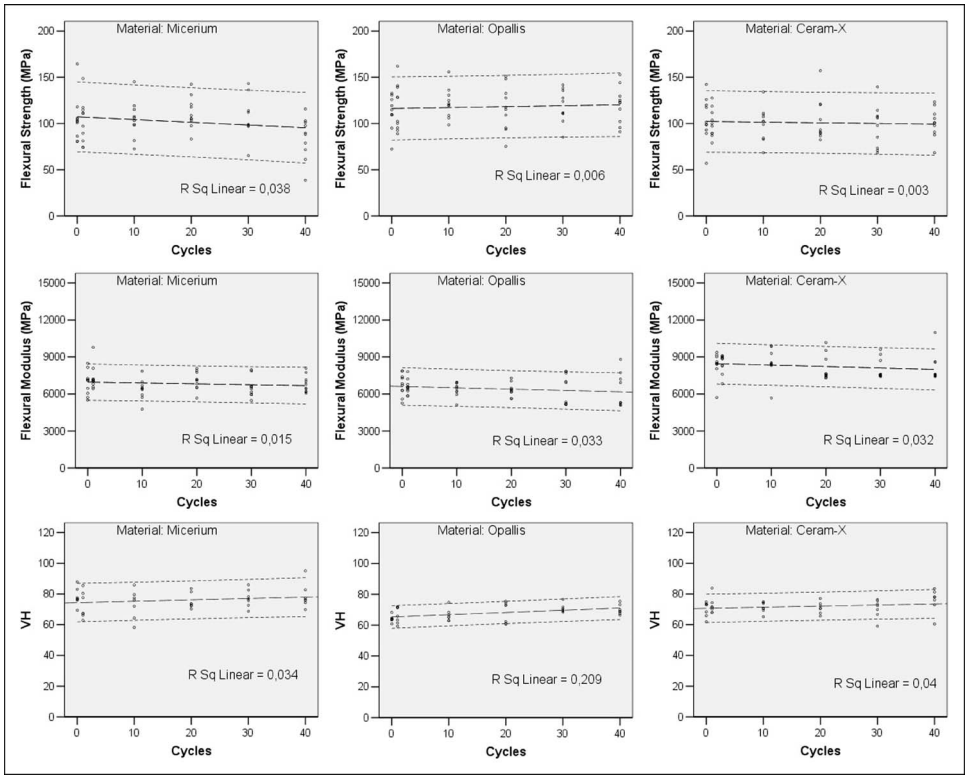

Figure 4. *Scatter plots and linear regression functions (showing also the 95% confidence intervals) obtained looking at the number of preheating cycles as the independent variable (horizontal axis) and the observed flexural strength, flexural modulus, and the Vickers hardness (VH) values as the dependent variables (vertical axes). Each material was considered separately. R Sq Linear = r^2 coefficient of determination (R^2).*

viscosity also allows for improved wetting of cavity walls compared with RT heavily filled restorative composites. This in turn provides for improved adaptation to cavity walls and decreased gap formation.[3] Viscosity of composite resin pastes is a complicated phenomenon, especially when the factor of heat is introduced.[18] The extent of viscosity change may be attributed to many factors, including resin composition, filler content, and shade. Thus, because of the wide variety in chemistry and composition of resin composites currently used, a great variation in the viscosity of these materials in response to evaluated temperatures may be expected.[15] With increasing molecular weight and the greater potential for hydrogen bonding, viscosity of the resin component will increase.[19,20] Also, increases in chain length and extent of side chain structures (branching) will tend to increase viscosity as polymer chains become more entangled.[19] Likewise, with heating, sufficient energy must be given to overcome these obstacles (hydrogen bonding and chain entanglement) to allow molecules freedom to move in a less hindered sheering pattern with respect to one another. Filler particle content, shape, and size also influence composite resin paste flow.[18] In general, the filler loading level, the filler surface contour, and

Table 4: *Flexural Modulus Mean Values (Standard Deviations) Achieved in Different Groups*[a]

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

[a] *Same letters indicate no statistically significant differences among the levels of the heating cycles (reading horizontally). Different superscript numbers indicate significant differences among the levels of composite employed (reading vertically).*

Table 5: *Vickers Hardness Mean Values (Standard Deviations) Achieved in Different Groups*[a]

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

[a] *Same letters indicate no statistically significant differences among the levels of the heating cycles (reading horizontally). Different superscript numbers indicate significant differences among the levels of composite employed (reading vertically).*

the distribution of filler size impacts the ability of particles to easily slide past one another. Heating would not directly affect the glassy particle itself because, within the temperature range imparted at clinically relevant temperatures, the viscosity of the filler particle (a ceramic) remains unchanged. Coatings on the filler particle could affect the ease with which a filler particle would move in the warmed resin fluid. Particles not silanated would be more difficult to move than those that are coated, as silanization imparts better resin wetting and, thus, ease of fluid movement around the particle.[20] An additional advantage of heating the resin composite is that preheated light-curing composites can be easily used as luting agents for porcelain veneers[22,23] or indirect composite restorations[24] in place of dual-curing materials.[25,26]

There is a general consensus in the literature on the absence of harmful effects of preheating procedures on the mechanical properties of resin composites.[2,3,15] In a recent study, Osternack and others[27] concluded that composite hardness was not affected by precooling or preheating procedures. However, the majority of previous studies did not consider repeated preheating cycles. Daronch and others[14] 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 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 multishade 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 were steadily adopted. In contrast, the present study showed that even highly repeated cycles of preheating 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 that reported a slightly lower composite temperature compared with that of the heating source,[10,14] in this study all the composites achieved a maximum temperature of 39°C after 10 minutes with the preheating device preset to 39°C.

CONCLUSIONS

The tested preheating procedure did not negatively influence the mechanical properties of the resin composites even when highly repeated. Further studies might be needed to assess the clinical relevance of the other variables connected to the repeated preheating and cooling cycles. Based on these findings and within the limitations of the study, dental clinicians can steadily adopt this preheating procedure without compromising the mechanical strengths of the heated composites.

Conflict of Interest

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 27 January 2014)

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