

Post-cure heat treatments influence on mechanical and optical properties of resin composites

Vitor de Oliveira Baldo 1, Marlus da Silva Pedrosa 1, Igor Studart Medeiros 1.

This study evaluated the effect of post-cure heat treatment (PCHT) on the Knoop microhardness (KHN), degree of conversion (DC), color changes, and contrast ratio (CR) of four resin composites (RCs): Z100 (3M ESPE), Z350 XT (3M ESPE), Estelite Omega (Tokuyama) and Empress Direct (Ivoclar Vivadent). Specimens (12 \times 1 mm) were prepared for each material (n = 10 / group). After curing, samples were subjected to PCHT for 10 min at 100°C or 170°C. Control group was maintained at room temperature (24°C) for the same time. The DC was analyzed by FT-NIR immediately and 24 h after the PCHT (n = 3 / group). KHN was analyzed 24 h after PCHT (n = 10 / group). According to CIEDE2000 (ΔE_{00}) , color measurements were obtained immediately after curing, five minutes after PCHT, and after seven days of storage in water, coffee, and red wine. Data were analyzed by One and Two-Way ANOVA (p < 0.05). Z100, Z350, and Estelite Omega showed increases in KHN with increased temperature (p < 0.05). PCHT at 100° C and 170° C led to a higher DC of all RCs (p < 0.05). Initially, the PCHT lead to increased ΔE_{00} values (p < 0.05), which was decreased after immersion in coffee and wine (p < 0.05). Considering the effect of PCHT and staining solutions, lower color changes were observed in the thermally treated specimens (p < 0.05). Taken collectively, the results suggest the PCHT as an economical and practical alternative to enhance direct RC's properties in direct-indirect and indirect restorations.

¹ Department of Biomaterials and Oral Biology, School of Dentistry, University of São Paulo, Brazil

Correspondence: Prof. Igor Studart Medeiros Departamento de Biomateriais e Biologia Oral, Faculdade de Odontologia, Universidade de São Paulo (USP). Av. Prof. Lineu Prestes 2227, 05508-900, São Paulo, SP, Brasil. E-mail address: igorsm@usp.br

Key Words: heat, mechanical properties, dental materials, resin composites

Introduction

The search for aesthetic materials, simplification, and reducing the cost of procedures are some of the dentistry challenges. Resin composites (RCs) for direct use are highly popular due to the less dental tissue removal technique and handling. However, the direct approach in extensive cavities presents disadvantages such as greater difficulty in accessing cavity margins, loss of proximal contact, marginal staining, and development of caries adjacent to restorations (1). In addition, RCs are exposed to higher mechanical load in large cavities (2,3), which might limit its indication.

In this sense, indirect restorations with RCs become an attractive alternative (4). One of the significant advantages of the indirect technique is the increase in the mechanical strength of restorative materials obtained through post-cure treatments that use light, pressure, and heat to increase conversion. However, laboratory apparatus dependence may contribute to a higher cost for indirect restorations (5).

A viable alternative to indirect restorations with RCs is the use of the direct-indirect technique. This technique consists of making the restoration in working models obtained from molding or scanning the dental remnant's preparation. After sculpting the restoration on the model, the dentist performs photo activation of the RCs followed or not by a post-cure treatment (6,7).

Post-cure heat treatment (PCHT) has been recommended to increase the mechanical properties of indirect restorations (6-9). However, there is no consensus in the literature about the ideal device or temperature for heat treatment, ranging from 50 to 170°C (6-8,10). Thus, commercial furnaces, autoclaves, microwaves, and laboratory ovens are commonly used. As the literature has focused on mechanical properties, there is scarce literature investigating the influence of PCHT on the optical properties of resin composites (9).

The present study aimed to evaluate the effect of PCHT at 100 and 170°C on the Knoop microhardness (KHN), degree of conversion (DC), color changes, and contrast ratio (CR) of four RCs. The

null hypotheses tested were: 1 – the PCHT would not lead to significant changes in the microhardness of RCs; 2 – the degree of conversion of the RCs would not be influenced by the PCHT; and 3 – the PCHT would have no significant effect on the color changes of the RCs.

Material and Methods

Specimen Preparation

Four RCs (A2 color) were evaluated: Z100 (3M ESPE, St. Paul, MN, USA), Z350 XT (3M ESPE, St. Paul, MN, USA), Estelite Omega (Tokuyama, Tokyo, Japan) and Empress Direct (Ivoclar Vivadent, Schaan, Liechtenstein) (Table 1). The sample size was calculated according to previous studies (9). Specimens of 12 mm in diameter and 1-mm thick were obtained. The RCs were inserted in a single increment, and a 1-mm-thick glass slide was positioned over the matrix with a 1-kg weight for 30 s (11). Photoactivation was carried out with a light-curing unit (LCU) (Bluephase G2, Ivoclar Vivadent AG, Schaan, Liechtenstein) with an irradiance of 1200 mW/cm2 and an internal tip diameter of 8.4 mm. The LCU was placed inside a spacer that distributed the light beam to all surfaces. The irradiation time was determined to obtain a dose of 20 J/cm2 (12). The output of the curing light was checked after ten consecutive uses with a radiometer (LED Radiometer, SDI, Victoria, Australia).

Table 1. Materials tested and manufacturer's information

Composite	Composite Type	Matrix	Filler	Photoinitiator system	% Load (V/W)	Manufacturer	Batch #
Estelite Omega	Spherical	Bis-GMA and TEGDMA	Silica-zirconia and composite (mean particle size = 200nm)	Camphorquinone/amine + Radical-Amplified Photopolymerization	82/71	Tokuyama, Tokyo, Japan.	8068; 80815;
Filtek Z100	Micro-hybrid	Bis-GMA and TEGDMA	Silica/Zirconia	Camphorquinone/amine	84.5/66	3M ESPE, St. Paul, MN, USA.	907911; 1813100924; 1826900169
Filtek Z350 XT	Nanofilled	Bis-GMA, Bis- EMA, UDMA, TEGDMA, PEGDMA	Silica (4-20 nm), zirconia (4-11 nm), clusters of zirconia/silica	Camphorquinone/amine	78.5/71	3M ESPE, St. Paul, MN, USA.	688062; 1817700807; 1923800123
Empress Direct	Nano-hybrid	Bis-GMA, UDMA and dimethacrylates	Barium glass, ytterbium trifluoride, mixed oxide, silicon dioxide and copolymer (mean particle size = 550nm)	Camphoroquinone + Lucirin TPO	75-79/ 52-59	Ivoclar Vivadent, Schaan, Liechtenstein	x00591; x54421;

Post-Cure Heat Treatment

After polymerization, experimental groups were subjected to dry PCHT for 10 minutes at 100 or 170 °C. The PCHT was perfomed in oven developed by the Department of Biomaterials and Oral Biology of the School of Dentistry of the University of São Paulo with the support of VRC Equipamentos (Guarulhos, São Paulo, Brazil). The equipment presents a heating rate of 50° C/min with \pm 2°C variation. The control group was maintained at room temperature (24°C) for the same time. All samples (n = 10 / group) were stored in deionized water, coffee (Nescafé Original, Nestlé, SP, Brazil) (9), and red wine (13% vol. Alcohol – Aurora, Brazil – Cabernet Sauvignon, 2018) and kept at 37°C for color measurements. The solutions were renewed every two days. The specimens were cleaned in running water for 30 s per face and 6 min immersion in distilled water in an ultrasonic vat before measurements.

Knoop Microhardness

After the PCHT, each specimen (n = 10 / group) was stored in deionized water in light-proof containers for 24 h at 37° C. A Knoop diamond indenter was used to make indentations in the specimens' bottom and top surfaces with a 50g load for 15 s. For each sample, three measurements were taken with a minimum distance of 200 μ m between each measure. The mean for the 3 measurements were used for statistical analysis. The ratio between bottom and top KHN was also calculated.

Degree of Conversion

The spectrum of the non-polymerized material was obtained by FT-NIR spectroscopy (Vertex 70, Bruker Optik, Ettlingen, Germany) immediately and 24 h after curing. The RCs (n = 3 / group) were light-cured (Bluephase G2, Ivoclar Vivadent AG, Schaan, Liechtenstein) under 1,200 mW/cm2 and stored dry at 37°C for the evaluation at 24 h. Then, the spectrum of the polymerized material was obtained, and the degree of conversion (DC) was calculated (%) (13).

Color Measurements

A spectrophotometer (Minolta CM 3700d, Konica Minolta, Japan) was used. The device was set with D65 light using the second observation angle, 100% UV energy, and small aperture size. White background, operator, place, and lighting conditions were standardized for all samples. According to the Commission Internationale de l'Éclairage (CIE) Delta E 2000 (CIEDE2000), color differences (ΔE_{00}) were calculated using the following formula (14):

$$\Delta E_{00} = \{ (\Delta L'/(k_L S_L)^2 + (\Delta C'/(k_C S_C)^2 + (\Delta H'/(k_H S_H)^2 + R_T[(\Delta C'/K_C S_C) \times (\Delta H'/(k_C S_C)] \}^{1/2} \}$$

Three analyses were considered. The first was performed 10 minutes after curing, 5 minutes after the PCHT at 100 and 170°C. The objective of this first analysis was to evaluate the initial effect of the PCHT on color changes. The second color analysis was done before and after immersion in water, coffee, and wine for seven days. This analysis allows the understanding of the initial effect of PCHT on the color changes. The third analysis was performed after the PCHT and the immersion of the thermally treated specimens for seven days to verify the overall effect of the PCHT on the color changes.

Contrast Ratio

The contrast ratio was calculated from the reflectance (Y) mean values of the specimens with a black (Yb) and white (Yw) backing to give Yb/Yw (considering 0.0 = transparent; 1.0 = opaque) (15).

Statistical Analysis

Normal data distribution and homogeneity of variances were verified by Shapiro-Wilk and Levene's tests, respectively. For KHN, One and Two-Way ANOVA followed by Tukey's tests (p < 0.05) were used for statistical analysis. Two-way ANOVA with repeated measurements and One-Way anova followed by Tukey's tests (p < 0.05) were used to verify differences in DC (p < 0.05). To analyze differences in color changes, One and Two-way ANOVA and Tukey's tests were used (p < 0.05).

Results

Knoop Microhardness

Z100, Z350 XT, and Estelite Omega showed significant increases in KHN values with increased temperature on both the bottom and top surfaces (P < .05). For Empress Direct, only the PCHT at 170°C led to increases in KHN values (P < 0.05). Also, different results were found between the RCs for the same treatment (P < 0.05) (Table 2).

Table 2. Mean and standard deviation in Knoop Microhardness (MPa) on bottom and top surfaces of the resin composites after post-cure heat treatment

	Bottom				Тор				Bottom/Top (%)		
Composites	Control	100°C	170°C	Control	100°C	170°C	Control	100°C	170°C		
Z100	90.7 ± 1.0 Aa	98.8 ± 2.3 Ba	105.9 ± 1.4 Ca	101.5 ± 1.3 Aa	108.6 ± 1.4 Ba	113.1 ± 1.3 Ca	89.42%	90.95%	93.70%		
Z350 XT	60.3 ± 0.8 Ab	68.6 ± 0.5 Bb	70.7 ± 0.5 Cb	62.7 ± 1.3 Ab	72.1 ± 1.2 Bb	76.9 ± 0.6 Cb	96.15%	95.11%	91.97%		
Estelite Omega	34.9 ± 0.8 Ac	39.9 ± 0.8 Bc	43.8 ± 1.4 Cc	38.9 ± 0.7 Ac	43.4 ± 1.2 Bc	47.8 ± 1.0 Cc	87.73%	91.80%	91.53%		
Empress Direct	22.4 ± 0.9 Ad	22.2 ± 1.2 Ad	26.7 ± 0.6 Bd	25.5 <u>±</u> 0.9 Ad	25.7 ± 1.1 Ad	30.2 ± 1.7 Bd	89.87%	86.26%	88.44%		

Different capital letters in the row represent statistically differences between treatments for the same resin composite by Two-way ANOVA followed by Tukey's post hoc test (p < 0.05). Different lowercase letters in the column represent statistically differences between the resin composites for the same group (same column) by One-Way ANOVA followed by Tukey's post hoc test (p < 0.05).

Degree of Conversion

Table 3 shows that PCHT at 100 and 170°C led to a higher DC of all RCs than the control (P < 0.05). In addition, in the post-cure evaluation, the PCHT at 170°C showed higher DC than PCHT at 100°C (P < 0.05). The DC after 24 h was higher for RCs with PCHT at 170°C compared to control and PCHT at 100°C

(P< 0.05). Besides, the DC after 24 h for PCHT at 170° C was similar to the same treatment in the post-cure (P > 0.05).

Table 3. Mean and standard deviation in Degree of conversion (%) of the resin composites immediately and 24 h after the post-cure heat treatment

		Immediate	24 h				
Composites	Control	100°C	170°C	Control	100°C	170°C	
Z100	65.52 ± 0.38	75.49 ± 0.69	83.73 ± 3.96	73.61 ± 0.74	75.49 ± 0.69	83.04 ± 1.41	
	Aa	Ba	Ca	Ba	Ca	Ca	
Z350 XT	63.33 ± 0.91	74.65 ± 1.74	78.83 ± 0.89	72.02 ± 1.99	74.65 ± 1.74	78.90 ± 0.92	
	Ab	Ba	Cbc	Ba	Ва	Cb	
Estelite Omega	59.36 ± 0.41	69.90 ± 1.65	75.17 ± 1.73	65.92 ± 0.49	69.90 ± 1.65	76.03 ± 0.83	
	Ac	Bab	Cbd	Bb	BDa	Cc	
Empress Direct	53.27 ± 0.34	66.67 ± 5.18	73.31 ± 0.35	61.68 ± 0.19	66.67 ± 5.18	73.57 ± 0.24	
	Ad	Bb	Cb	Вс	BDb	Cc	

Different capital letters in the row represent statistically differences between treatments for the same resin composite by Two-way ANOVA with repeated measurements followed by Tukey's post hoc test (p < 0.05). Different lowercase letters in the column represent statistically differences between the resin composites for the same group (same column) by One-Way ANOVA followed by Tukey's post hoc test (p < 0.05).

Color Changes after PCHT

Table 4 shows the color changes of the RC after the PCHT. A significant interaction between these factors was observed (p < 0.0001). Overall, the PCHT at 100 and 170°C lead to increased ΔE_{00} (P < 0.05). For Z100, there was no significant difference in the color changes between PCHT at 100 and 170°C (P > 0.05). For Z350 XT, Estelite Omega, and Empress Direct, the PCHT at 170°C led to higher ΔE_{00} values (P < 0.05).

Table 4. Mean and standard deviation in color changes △E₀₀ of the resin composites after post-cure heat treatment

		∆ E ₀₀	
Composites	Control	PCHT at 100°C	PCHT at 170°C
Z100	0.13 ± 0.05 A	2.60 ± 0.25 B	2.00 ± 0.25 B
Z350 XT	0.12 ± 0.04 A	$0.70 \pm 0.26 \ C$	1.10 ± 0.13 D
Estelite Omega	0.16 ± 0.08 A	$1.00 \pm 0.04 D$	$3.50 \pm 0.41 E$
Empress Direct	0.12 ± 0.07 A	$0.72 \pm 0.05 C$	1.30 ± 0.12 D

Different capital letters represent statistically differences by Two-way ANOVA followed by Tukey's post hoc test (p < 0.05).

Color changes after Immersion in Staining Solutions

Table 5 shows the color changes in treated specimens after immersion in water, coffee, and red wine. For Z100, the PCHT at 100 and 170°C led to fewer color changes than the control in water, coffee, and wine (P < 0.05). Also, the PCHT with 170°C presented the lowest ΔE_{00} values than PCHT at 100°C for specimens immersed in water and coffee (P< 0.05). For Z350 XT, the PCHT at 170°C led to lower ΔE_{00} values than PCHT at 100°C and control after immersion in coffee and wine (P < 0.05). There was no ΔE_{00} difference between the control and heat-treated specimens immersed in water (P > 0.05). The PCHT at 100 and 170°C in Estelite Omega led to less color change than control (P < 0.05). The PCHT at 170°C presented ΔE_{00} values compared to 100°C for specimens immersed in coffee (P < 0.05). For samples immersed in wine, ΔE_{00} values in the PCHT at 100 and 170°C were similar (P > 0.05) and lower than control (P < 0.05). For Empress Direct, after immersion in coffee and wine, PCHT values at 100°C were similar to the control group (P> 0.05). Interestingly, ΔE_{00} values for specimens treated at 170°C were lower than control and PCHT at 100°C (P < 0.05).

Table 5. Mean and standard deviation in color changes (ΔE_{00}) of the thermal treated resin composites resin after immersion in water, coffee and wine

		Water			Coffee			Wine	
Composites	Control	100°C	170°C	Control	100°C	170°C	Control	100°C	170°C
Z100	2.23 ± 0.11	0.99 ± 0.072	0.62 ± 0.14	6.90 ± 0.22	4.30 ± 0.55	2.7 ± 0.8	7.50 ± 0.44	4.90 ± 0.13	5.50 ± 0.68
2100	Aa	Ba	Ca	Da	Ea	Aa	Da	Ea	Fa
Z350 XT	0.43 ± 0.21	0.97 ± 0.18	1.50 ± 0.19	16.00 ± 0.46	13.00 ± 1.30	11 ± 0.65	22.00 ± 0.2	19.00 ± 0.54	15.02 ± 0.83
2350 XI	Ab	Aa	Ab	Bb	Cb	Db	Eb	Fb	Bb
Estelite	1.50 ± 0.11	0.65 ± 0.15	1.00 ± 0.13	9.60 ± 0.076	7.60 ± 0.61	5.6 ± 0.31	8.40 ± 0.69	6.30 ± 0.26	6.41 ± 0.26
Omega	Ac	Ab	Ac	Вс	Cc	Dc	Cc	Da	Da

 1.50 ± 0.03 1.2 ± 0.08 23.00 ± 0.26 23.00 ± 0.89 19 ± 0.84 23.00 ± 0.51 0.94 ± 0.14 23.00 ± 1.80 18.00 ± 1.00 **Empress Direct** Bd Bd Αd Ac Abc Bd Cd Вс Cc

Different capital letters in the row represent statistically differences between treatments for the same resin composite by Two-way ANOVA followed by Tukey's post hoc test (p < 0.05). Different lowercase letters in the column represent statistically differences between the resin composites for the same group (same column) by One-Way ANOVA followed by Tukey's post hoc test (p < 0.05).

Overall Effect of the PCHT and Staining Solutions on Color Changes

Table 6 shows the overall effect of the PCHT treatment and immersion in water, coffee, and wine on the color changes. When immersed in water, the PCHT at 100°C for Z100 led to higher color change than the control and PCHT at 170°C. However, no significant differences were observed for Z350 XT, Estelite Omega and Empress Direct immersed in water.

Table 6. Mean and standard deviation in color changes (ΔE_{00}) of the composite resins after the post-cure heat treatment and immersion in water, coffee and wine

		Water			Coffee		Wine			
Composites	Control	100°C	170°C	Control	100°C	170°C	Control	100°C	170°C	
Z100	2.30 ± 0.01	3.10 ± 0.70	1.80 ± 0.20	6.60 ± 0.33	6.40 ± 0.50	4.30 ± 0.66	7.30 ± 0.39	6.90 ± 0.08	5.80 ± 0.46	
	Aad	Ba	Aa	Ca	CDa	Ea	Ca	Ca	CDa	
Z350 XT	0.52 ± 0.19	0.62 ± 0.17	0.49 ± 0.32	16.00 ± 0.42	13.00 ± 1.30	12.00 ± 0.63	22.00 ± 0.20	19.00 ± 0.53	16.00 ± 0.81	
	Ab	Ab	Ab	Bb	Cb	Db	Eb	Fb	Bb	
Estelite	1.80 ± 0.47	1.40 ± 0.57	2.40 ± 0.28	9.10 ± 0.02	8.20 ± 0.56	7.80 ± 0.09	7.70 ± 0.65	6.20 ± 0.07	8.50 ± 0.27	
Omega	Aa	Ac	Ab	Bc	Cc	Cc	Ca	Da	Cc	
Empress	1.50 ± 0.34	0.95 ± 0.20	0.72 ± 0.21	23.00 ± 0.27	23.00 ± 0.88	19.00 ± 1.80	23.00 ± 0.49	23.00 ± 1.80	19.00 ± 0.97	
Direct	Aae	Ac	Ac	Bd	Bd	Cd	Bb	Bc	Cd	

Different capital letters in the row represent statistically differences between treatments for the same resin composite by Two-way ANOVA followed by Tukey's post hoc test (p < 0.05). Different lowercase letters in the column represent statistically differences between the resin composites for the same group (same column) by One-Way ANOVA followed by Tukey's post hoc test (p < 0.05).

When immersed in coffee, the PCHT at 170° C ΔE_{00} values were lower than control and 100° C-treated specimens for Z100 and Estelite Omega (P < .05), and no difference between control and PCHT at 100° C (P > 0.05). For Z350 XT, PCHT at 170° C presented the lowest ΔE_{00} values than control and 100° C-treated specimens (P < 0.05). Also, ΔE_{00} values for the PCHT at 170° C were lower than those for PCHT at 100° C. For Estelite Omega, PCHT at 170° C lead to the lowest ΔE_{00} values compared to control and 100° C-treated specimens (P < 0.05).

After immersion in wine, the PCHT at 170°C lead to lower ΔE_{00} values compared to control and 100°C-treated specimens for Z100 and Estelite Omega (P < .05), with no difference between control and PCHT at 100°C (P > 0.05). For Z350 XT, PCHT at 170°C lead to the lowest ΔE_{00} values compared to control and 100°C-treated specimens (P < 0.05). Besides, ΔE_{00} values for the PCHT at 170°C were lower than those of PCHT at 100°C. For Estelite Omega, PCHT at 100°C lead to lower ΔE_{00} values than control and 170°C-treated specimens (P < 0.05).

Contrast Ratio

For Z100, Z350 XT, and Estelite Omega, the PHCT at 170° C lead to higher CR values than control and post-cure (P < 0.05) (Table 7). Only for Z100, the PHCT at 100° C lead to higher CR values when compared to the post-cure at 100° C and no PCHT (P < 0.05). For Empress direct, a decrease in CR values was observed in the post-cure and after the PHCT at 100° C (P < 0.05) while no difference was found in the 170° C-treated specimens compared to control (P > 0.05). Also, Δ CR values were only significant for 170° C-treated samples of Z100 and Estelite Omega compared to control (P < 0.05).

Discussion

In this study, the null hypotheses were rejected. The results show that PCHT led to significant increase in KHN and DC. Regarding color changes, initially, the PCHT increased ΔE_{00} values, which was decreased after immersion in coffee and wine. When considering the effect of PCHT and staining solutions, lower color changes were observed in the heat-treated specimens. Also, the PCHT at 170°C presented better results in KHN, DC and color changes compared to the one at 100°C in most of the RCs.

In this study, higher microhardness with increasing temperature of PCHT was also observed in the literature (7,10). Ideally, the RCs should have most of their monomers converted into polymers after curing as the DC (16) influences their properties. Besides the composition of the materials, the DC

increase may explain the increased KHN values after PCHT, as low hardness and color stability of RCs are associated with low DC (17,18).

In this study, the DC was analyzed by FT-NIR give it is an efficient, practical, precise, and convenient method for this purpose (19). The 170°C PCHT led to higher DC than to control and 100°C PCHT, for most RCs. This relationship between the PCHT temperature and DC has already been reported in the literature (10). In this sense, the increase in temperature may have led to a high kinetic energy and monomers' conversion.

Overall, when the PCHT approaches from the glass transition temperature (Tg) of the material, there is greater mobility of a segment of the chain, especially in light-cured RCs. This phenomenon allows a reduction of unreacted radicals as well as greater stress relaxation (6,7). Therefore, the proportional increase in the PCHT and DC temperature observed in this study corroborates the hypothesis that PCHT beyond the Tg of the material leads to better mechanical results.

The RC composition is also a factor affecting the DC. RCs with TEGDMA in their polymeric matrix generally present higher values of DC due to their reduced molecular weight and increased mobility during polymerization (20). In this study, UDMA and Bis-EMA might be responsible for the intermediate DC values found for Z350 XT. Particles type and size also influence the DC. Micro-hybrid or micro-filled RCs present higher DC than nanoparticle RCs (16).

Color changes were analyzed based on the CIEDE2000 (14). Studies have used different acceptable or noticeable values of Δ E00, even though there is not a consensus on what color difference is considered clinically acceptable. For the present study, the clinical acceptance threshold was Δ E₀₀= 2.25 (21).

The PCHT at 100 and 170°C led to color changes in the RCs. In the initial analysis, except for Z100 treated at 100°C and Estelite Omega at 170°C, all groups presented clinically acceptable ΔE_{00} values. After immersion in water, coffee, and wine, ΔE_{00} values were only clinically acceptable for the PCHT groups stored in water. By analyzing the overall effect, only Z100 Control and 100 and 170°C-PCHT Estelite Omega groups presented clinically unacceptable ΔE_{00} values when immersed in water. When storage in coffee, the 170°C-PCHT led to less color change than 100°C-PCHT and control for the most RCs.

The degradation of amines used together with the camphorquinone may have led to the color changes of the RCs in the 100 and 170°C-PCHT. Heat and UV light create higher energy states and these molecules with a high degree of excitation can react with oxygen and other aromatic groups. From these reactions, chromophores are formed, leading to color changes. Also, unreacted monomers trapped in the polymer network as well as monomer leaching, water sorption and the potential photobleaching of camphorquinone during the PCHT may result in color changes in the RCs. Besides, other factors such as type, size, and filler particle volume fraction may influence the different results obtained for the RCs (22).

The contrast ratio is defined as the ratio of the reflectance obtained with black and white background and can be considered the inverse of translucency. Thus, a resin composite with a high CR would present a high opacity (23). There is still no agreement on which CR could be clinically significant or detectable. In this study, the Δ CR values found were not clinically perceptible, assuming a Δ CR of 0.06 as the threshold (24). There is a gap in the literature concerning the effects of PCHT on the contrast ratio of RCs.

The shedding of the filler caused by the monomer leaching may produce RCs higher opacity and rough surface over time. For Z100, Z350 XT, and Estelite Omega, the 170°C-PCHT lead to higher contrast ratio values than control. Only for Empress direct, there was a decrease in CR values after post-cure and 100°C-PCHT. The increase in the CR values after PCHT may be related to a change in the organic matrix refractive index. As observed in this study, the RC translucency matrix might be influenced by chemical composition, content and size of fillers, and microstructural aspects. In this way, nano-hybrid RCs may show higher translucency than microhybrid (25).

Table 7. Mean and standard deviation in contrast ratio and △CR values of the resin composites before and after post-cure heat treatment

			ACD							
Composites	Control (Room	Control (Room temperature)		PCHT at 100°C		PCHT at 170°C		ΔCR		
	After Curing	After 10min	After Curing	After PCHT	After Curing	After Curing After PCHT		PCHT at 100°C	PCHT at 170°C	
Z100	0.50 ± 0.007 Aa	0.50 ± 0.009 Aa	0.50 ± 0.009 Aa	0.52 ± 0.009 Ba	0.51 ± 0.011 Aa	0.52 ± 0.012 Ba	0.0031 ± 0.003 A	0.0180 ± 0.005 B	0.0085 ± 0.012 B	
Z350 XT	0.44 ± 0.008 Ab	0.44 ± 0.006 Ab	0.45 ± 0.014 Ab	0.45 ± 0.015 Ab	0.46 ± 0.005 Ab	0.47 ± 0.005 Bb	-0.004 ± 0.0061 A	0.0006 ± 0.019 A	0.0071 ± 0.0047 A	
Estelite Omega	0.47 ± 0.004 Ac	0.47 ± 0.006 Ac	0.48 ± 0.008 Ac	0.48 ± 0.008 Ac	0.47 ± 0.004 Ac	0.49 ± 0.046 Bc	-0.0001 ± 0.0051 A	0.004 ± 0.0069 A	0.0202 ± 0.004 B	
Empress Direct	0.55 ± 0.008 Ad	0.55 ± 0.007 Ad	0.53 ± 0.005 Bd	0.53 ± 0.005 Bd	0.54 ± 0.009 Ad	0.54 ± 0.012 Ad	- 0.0003 ± 0.0037 A	- 0.0016 ± 0.006 A	0.0005 ± 0.011 A	

Different uppercase letters in the row represent significant differences between treatments for the same resin composite for contrast ratio. Two-way ANOVA followed by Tukey's post hoc test (p < 0.05). Different lowercase letters in the column represent significant differences between the resin composites for the same group. One ANOVA followed by Tukey's post hoc test (p < 0.05). Different capital letters for \triangle CR represent statistically differences by Two-way ANOVA followed by Tukey's post hoc test (p < 0.05).

The present study results suggest the 170°C-PCHT is an economical and practical alternative to enhance the properties of RCs used in direct-indirect and indirect restorations. However, the results must be interpreted with limitations since the in vitro study may not fully simulate in vivo performance. An analysis of the RC leached components could lead to a better understanding of the effect of PCHT on the DC, color changes, and contrast ratio. Long-term clinical investigations should also complement these in vitro findings.

Considering the analysis of the set of results, it can be concluded that the post-cure heat treatment promoted an overall increase in the microhardness, DC, color stability, and CR of the RCs. Besides, 170°C-PCHT led to better results on the microhardness, DC, color stability, and contrast ratios than 100°C-PCHT for most RCs.

Resumo

Este estudo avaliou o efeito do tratamento térmico pós-cura (PCHT) na microdureza Knoop (KHN). grau de conversão (DC), mudanças de cor e razão de contraste (CR) de guatro compósitos resinosos (RCs): Z100 (3M ESPE), Z350 XT (3M ESPE), Estelite Omega (Tokuyama) e Empress Direct (Ivoclar Vivadent). Corpos de prova (12×1 mm) foram preparadas para cada material (n = 10 / qrupo). Após a cura, as amostras foram submetidas ao PCHT por 10 min a 100 ou 170° C. O grupo controle foi mantido à temperatura ambiente (24° C) pelo mesmo tempo. O DC foi analisada por FT-NIR imediatamente e 24 h após a PCHT (n = 3 / grupo). KHN foi analisado 24 h após PCHT (n = 10 / grupo). De acordo com o CIEDE2000 (ΔE_{00}), as medidas de cor foram obtidas imediatamente após a cura, cinco minutos após a PCHT e após sete dias de armazenamento em áqua, café e vinho tinto. Os dados foram analisados por ANOVA de um e dois fatores (P < 0,05). Z100, Z350 XT e Estelite Omega mostraram aumentos no KHN com o aumento da temperatura (P < 0,05). PCHT a 100 ° C e 170 ° C levou a uma maior DC de todos os RCs (P < 0,05). Inicialmente, o PCHT levou ao aumento dos valores de ΔE_{00} (P < 0,05), que diminuiu após a imersão em café e vinho (P < 0,05). Considerando o efeito de PCHT e soluções de coloração, menores mudanças de cor foram observadas nas amostras tratadas termicamente (P < 0.05). Os resultados sugerem o PCHT como uma alternativa econômica e prática para aumentar as propriedades diretas de compósitos resinosos em restaurações diretas-indiretas e indiretas.

References

- 1. Pallesen U, Qvist V. Composite resin fillings and inlays. An 11-year evaluation. Clin Oral Investig 2003;7:71-9.
- 2. Ausiello P, Ciaramella S, Garcia-Godoy F, Gloria A, Lanzotti A, Maietta S, et al. The effects of cavity-margin-angles and bolus stiffness on the mechanical behavior of indirect resin composite class II restorations. Dent Mater 2017;33:e39-e47.
- 3. Magne P, Belser UC. Porcelain versus composite inlays/onlays: effects of mechanical loads on stress distribution, adhesion, and crown flexure. Int J Periodontics Restorative Dent 2003;23:543–55.
- 4. Lynch CD, Opdam NJ, Hickel R, Brunton PA, Gurgan S, Kakaboura A, et al. Guidance on posterior resin composites: Academy of Operative Dentistry European Section. J Dent 2014;42:377–83.
- 5. Torres CRG, Zanatta RF, Huhtala MFRL, Borges AB. Semidirect posterior composite restorations with a flexible die technique: A case series. J Am Dent Assoc 2017;148:671-6.
- 6. Miyazaki CL, Medeiros IS, Santana IL, Matos Jdo R, Rodrigues Filho LE. Heat treatment of a direct composite resin: influence on flexural strength. Braz Oral Res 2009;23:241-7.
- 7. Santana IL, Lodovici E, Matos JR, Medeiros IS, Miyazaki CL, Rodrigues-Filho LE. Effect of experimental heat treatment on mechanical properties of resin composites. Braz Dent J 2009;20:205-10.
- 8. Bagis YH, Rueggeberg FA. The effect of post-cure heating on residual, unreacted monomer in a commercial resin composite. Dent Mater 2000;16:244–7.
- 9. Grazioli G, Francia A, Cuevas-Suárez CE, Zanchi CH, Moraes RRD. Simple and low-cost thermal treatments on direct resin composites for indirect use. Braz Dent J 2019;30:279-84.
- 10. Ferracane JL, Condon JR. Post-cure heat treatments for composites: properties and fractography. Dent Mater 1992;8:290-5.
- 11. Domingos PAS, Garcia PPNS, Oliveira ALBMd, Palma-Dibb RG. Composite resin color stability: influence of light sources and immersion media. J Appl Oral Sci 2011;19:204-11.

- 12. Daugherty MM, Lien W, Mansell MR, Risk DL, Savett DA, Vandewalle KS. Effect of high-intensity curing lights on the polymerization of bulk-fill composites. Dent Mater 2018;34:1531-41.
- 13. Xavier TA, Fróes-Salgado NRG, Meier MM, Braga RR. Influence of silane content and filler distribution on chemical-mechanical properties of resin composites. Braz Oral Res 2015;29:1–8.
- 14. Luo MR, Cui G, Rigg B. The development of the CIE 2000 colour-difference formula: CIEDE2000. Color Research & Application: Endorsed by Inter-Society Color Council, The Colour Group (Great Britain), Canadian Society for Color, Color Science Association of Japan, Dutch Society for the Study of Color, The Swedish Colour Centre Foundation, Colour Society of Australia, Centre Français de la Couleur 2001;26:340–50.
- 15. Vichi A, Carrabba M, Paravina R, Ferrari M. Translucency of ceramic materials for CEREC CAD/CAM system. J Esthet Restor Dent 2014;26:224–31.
- 16. Ribeiro BCI, Boaventura JMC, Brito-Gonçalves Jd, Rastelli ANdS, Bagnato VS, Saad JRC. Degree of conversion of nanofilled and microhybrid composite resins photo-activated by different generations of LEDs. J Appl Oral Sci 2012;20:212-7.
- 17. Oliveira DCRS, Souza-Júnior EJ, Prieto LT, Coppini EK, Maia RR, Paulillo LAMS. Color stability and polymerization behavior of direct esthetic restorations. J Esthet Restor Dent 2014;26:288–95.
- 18. Neves AD, Discacciati JA, Orêfice RL, Jansen WC. Correlation between degree of conversion, microhardness and inorganic content in composites. Pesqui Odontol Bras 2002;16:349-54.
- 19. Stansbury J, Dickens SH. Determination of double bond conversion in dental resins by near infrared spectroscopy. Dental Mater 2001;17:71–9.
- 20. Gajewski VE, Pfeifer CS, Fróes-Salgado NR, Boaro LC, Braga RR. Monomers used in resin composites: degree of conversion, mechanical properties and water sorption/solubility. Braz Dent J 2012:23:508-14.
- 21. Miotti LL, Santos IS, Nicoloso GF, Pozzobon RT, Susin AH, Durand LB. The Use of Resin Composite Layering Technique to Mask Discolored Background: A CIELAB/CIEDE2000 Analysis. Oper Dent 2017;42:165-74.
- 22. Poggio C, Ceci M, Beltrami R, Mirando M, Wassim J, Colombo M. Color stability of esthetic restorative materials: a spectrophotometric analysis. Acta Biomater Odontol Scand 2016;2:95–101.
- 23. Johnston WM. Review of translucency determinations and applications to dental materials. J Esthet Restor Dent 2014;26:217–23.
- 24. Liu MC, Aquilino SA, Lund PS, Vargas MA, Diaz-Arnold AM, Gratton DG, et al. Human perception of dental porcelain translucency correlated to spectrophotometric measurements. J Prosthodont 2010;19:187-93.
- 25. Kim JJ, Moon HJ, Lim BS, Lee YK, Rhee SH, Yang HC. The effect of nanofiller on the opacity of experimental composites. J Biomed Mater Res B Appl Biomater 2007;80:332–8.

Received: 09/03/2021 Accepted:15/06/2021