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EVALUATION OF THE EFFECTS OF POST-POLYMERIZATION HEAT TREATMENT ON THE MECHANICAL AND OPTICAL PROPERTIES OF RESTORATIVE COMPOSITES

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Abstract: Objective: To evaluate the flexural strength (FR), modulus of elasticity (ME), color difference (∆ E) and Contrast Ratio (CR) of composites subjected to heat treatment (TT) after polymerization and the effect of storage in dyes (distilled water, soda and wine). Methodology: Rectangular specimens 10mm X 2mm X 1mm were made for the analysis of RF and ME and disks of 2mm height X 8mm diameter for the study of∆ E and RC using 2 composites (nano-particulate and hybrid). All CPs (n=76) were subjected to 170ºc heat treatment for 10 or 30 minutes in an oven, except for the TT control groups (n=38). The CIELAB system was used to evaluate∆ E and the reflectance of the sample on a black background (Yb) and the reflectance of the same sample on a white background (Yw) were used to determine the RC of the materials, where RC = Yb/Yw. The three-point bending test was used to analyze RF and ME. The data obtained was submitted to the Shapiro-Wilk, Levene and Tukey tests (α =0.05). Results: The 30-minute TT showed lower∆ E and RC compared to the 10-minute TT and control groups. The wine dye showed greater staining potential compared to water and soda. In the nano-particulated composite both TTs showed higher RF and ME values after the TTs compared to the control group and in the hybrid composite there was no statistical difference between the means observed in the control group and the groups with TTs. Conclusion: TT in 30 minutes proved to be effective for color stability and contrast ratio, as well as improving RF and ME properties in nano-particulated composites.
Keywords: resin composites; flexural strength; modulus of elasticity; color; contrast ratio; heat treatment.

INTRODUCTION

Direct-use composites have become popular due to their practicality, ease of handling and low cost, as they do not require a laboratory stage. However, the use of these materials has its limitations. The use of composites in dental elements with major loss of structure or extensive cavities can lead to fractures, margin staining, post-operative sensitivity, loss of proximal contact, recurrent caries, etc. Using direct-use composites, indirect restorations can be made. Some professionals call this procedure the direct-indirect technique. This technique involves the dentist making the indirect restoration himself, in the office, on the working model obtained by taking the impression or scanning the dental element, and then being able to associate this technique with an additional polymerization method, such as heat treatment, for example. [1, 2]

The main difference between direct-use and indirect-use composites lies in the polymerization method, because the polymerization of these indirect-use composites requires specialized equipment and often high intensities of light, pressure, heat or both are used in the process. Post-curing heat treatment of the material can improve flexural strength, wear resistance, fracture toughness and microhardness. The authors associate these improvements with an increase in the degree of conversion of the composites, making it possible to improve their physical and mechanical properties. For this purpose, researchers often use conventional ovens, microwaves and ceramic ovens in an attempt to improve these properties. However, this process for additional polymerization must be carried out without causing undesirable changes to the material. [1, 2, 3]

It has already been observed that when heat treatments are carried out above the glass transition temperature of the matrix, they can result in an increase in the degree of conversion of the monomers, generating an improvement in the modulus of elasticity, microhardness and fracture resistance.[4] Miyazaki found that the glass transition temperature (Tg) of the composite resins evaluated was above 157°C and below 170°C, in addition to reporting mass loss above 180°C. [5] It has also been found that the temperature at which the maximum degree of conversion was achieved was not the same as the temperature at which the maximum gain in mechanical properties was achieved, the latter being lower.[6] In addition, the thermal treatment may also enable relaxation of the stresses induced during the polymerization process, which could also improve the strength of the material. [1, (7,) 8] Most post-polymerization thermal treatment studies relate the increase in the degree of conversion to an improvement in mechanical properties, without investigating their optical properties. However, the color stability of composites can be influenced by the amount of unreacted monomers over time. [12] The success and longevity of a restoration is also related to the color stability of the composite. [13]

To evaluate the color of a material we can use the L*a*b* color space, also known as the CIELAB color space. This method was created after the theory of opposite colors, where two colors cannot be green and red at the same time, or yellow and blue at the same time. L* indicates luminosity and the chromaticity coordinates are indicated by a* (red/green) and b* (yellow/blue). The color differences are known as Deltas (Δ). The Deltas for L* (ΔL), a* (Δa) and b* (Δb) can be negative (-) or positive (+) as shown in Table 1. The total difference, Delta E (ΔE), is always positive. [20, 21]

In addition to color, translucency affects the aesthetic result of restorations, as it allows the appearance to be verified in order to maintain the naturalness of the tooth, as its absence results in an opaque and artificial appearance. Professionals should also be aware that translucency can vary for each composite depending on the commercial brand and type of composite. Another factor that can generate noticeable differences in translucency is the thickness of the composite increments. Translucency, in the field of optics, is the physical property of allowing light to pass through the material without being scattered. The opposite property of translucency is opacity, i.e. materials that do not transmit light are called opaque. [28, 29, 30] Translucency can be calculated by determining the index values for the translucency coefficient, translucency parameter and contrast ratio. The CR is calculated from the spectral reflectance of the sample (Y), with black (Yb) and white (Yw) backgrounds, obtaining the Yb/Yw ratio. When using the contrast ratio to assess translucency, we can consider the value of RC=0.07 to be the limit of perception for the human eye. Therefore, when the values are above this limit, changes in translucency may be detecTable to the human eye. [28, 31, 32]

Variable	Parameter	Results
ΔL	>0	Clearer
	<0	Darker
Δa	>0	More red (less green)
	<0	More green (less red)
Δb	>0	More yellow (less blue)
	<0	More blue (less yellow)
ΔE	<1	Not observed by the human eye
	1< ΔE< 3.3	Perceptible to the professional
	>3,3	Perceptible to laypeople
RC	=1	Opaque
	=0	Translucent
ΔRC	Formula	Final **C**R - Initial **C**R
	>0	More opaque (less translu**c**ent)
	<0	More translu**c**ent (less opaque)

Table 1 - evaluation parameters

An important factor when discussing the translucency of a material is that clinically there is no air between the material and the substrate and this fact must be maintained during the laboratory test, because the air present between the sample and the background (black or white) can alter the results. In routine dentistry, the space between a substrate and a restorative material is filled with cement and/or dentin adhesive. For this reason, a coupling agent with a refractive index similar to the materials used, such as glycerin, was used during the test [30].

Despite the favorable results of the post--curing heat treatment technique, there is no consensus on the temperature used, which can vary from 50° to 170°C, and there is also no consensus on the exposure time to these temperatures. In addition, few studies have evaluated the effects of post-curing heat treatment on the optical properties of composites, with no consensus between methodologies. [14, 15, 16, 17, 18] In view of these factors, the aim of this study was to investigate the influence of post-curing heat treatment on modulus of elasticity and flexural strength, color stability and contrast ratio after immersion in dyes.

MATERIAL AND METHODS

The specimens (n=76) were made with two composites (Table 2), both in A2D color, and divided into two large groups: nano-particulate (Filtek z350 XT) and hybrid (Forma). Each group was then divided into three subgroups according to the heat treatment (TT) in a dry oven: 170 °C for 10 and 30 minutes and control (no heat treatment), shown in Figure 1.

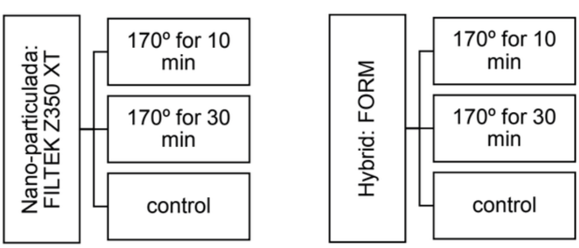

Figure1 - Division of the groups and subgroups of the specimens used

Composite	Inorganic Phase (% vol)	Organic Phase
Hybrid Shape -Ultradent	Zirconia/ Silica and Barium Glass 63.50%	Bisphenol-A Diglycidyl Dimethacrylate (BisGMA)
		Bisphenol-A Diglycidyl Ethoxylated Dimethacrylate (Bis-EMA)
		Urethane Dimethacrylate (UDMA)
		Triethylene Glycol Dimethacrylate (TEGDMA)
Nanoparticulate Z350 - 3M ESPE	Zirconia/Silica 71%	Bisphenol A diglycidyl ether dimethacrylate (BisGMA)
		Bisphenol A polyethylene glycol diether dimethacrylate (BISEMA-6)
		Diurethane dimethacrylate (UDMA)
		Polyethylene glycol dimethacrylate (PEGDMA)

Table 2 - Information on the composites used

OPTICAL PROPERTIES

To evaluate the effect of storage in dye, 54 CPs were used (Figure 2). The CPs were made in the shape of a disk measuring 2mm in height and 8mm in diameter, using a split metal matrix.

To make the specimens, the metal matrix was positioned on a polyester strip that was on a microscope slide and the composite was inserted in a single increment into the mold, using a #1 suprafil spatula. After this, a polyester strip was positioned over the top of the mold and a glass slide was placed over the polyester strip. A 500g weight was then placed on the composite for 30 seconds and photoactivation was carried out with the Radii light curing device (1000mW/cm^2) on both sides of the composite for 20 seconds, with the tip of the photoactivator resting against the polyester tape on top of the mold with the composite, in order to standardize the distance from the light source. After photoactivation, the specimens were carefully removed from the mold and evaluated for possible defects under an optical microscope at 40x magnification (SZ61TR, Olympus, USA) in order to discard

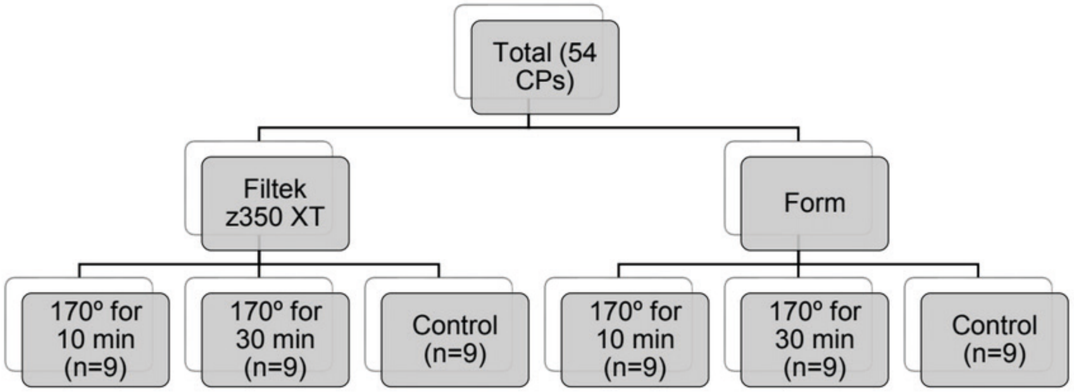

Figure 2 - Division of groups

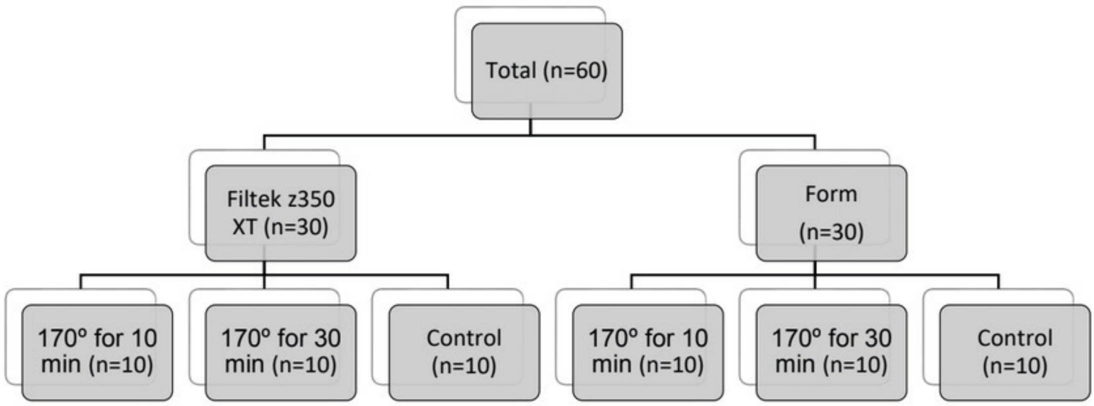

Figure 3 - Division of groups

those with imperfections, after which their surfaces were polished with silicon carbide sandpaper # 1200, 2500 and 4000 (DPU 10, Struers, Denmark) and water

After preparing the CPs, the respective heat treatments were carried out in an oven and, for the group without TT, the CPs were stored in a black box for 5 minutes at 24ºC until evaluation in the spectrophotometer. The CPs were analyzed in a CM-2600d spectrophotometer (Konica Minolta, Japan) 5 min after the respective TTs. The tests were carried out using a white bulkhead for evaluatingΔ E and a white and black bulkhead for evaluating RC. For both analyses, glycerin was used as a binder to attach the specimen to the bulkhead. Three measurements were taken on each PC and the average of these measurements was used for statistical analysis. After this first color analysis, the PCs were stored individually for a period of 7 days, kept in an oven at 37°C, in 3 media: distilled water, soda (Coca-Cola) and red wine (Concha e Toro - Carmenere, 2020). The solutions were changed every 2 days in the amount of 2ml of dye or distilled water for each specimen. After the 7-day period, the specimens were cleaned in running water for 30 seconds per side and immersed in distilled water for 30 minutes in an ultrasonic tank before the second color analysis in the spectrophotometer.

The color difference (ΔE) was calculated using the formula:

$$\Delta E = [(L` - L)^2 + (a` - a)^2 + (b` - b)^{(2)}]^{1/2}$$

in which the parameters "L", "a" and "b" are determined by the color system created by the Comisión Internacionale de l'Éclair (CIELAB). The L*a*b* color space quantitatively determines the color of the object, with the L* coordinate representing the brightness (lightness), a* representing the red-green chromatic coordinate and b* representing the yellow--blue chromatic coordinate. Color differences were assessed by reflectance on a white background. For greater detail of the color variation, the variations in each axis were analyzed separately: L*, a* and b*.

The contrast ratio (CR) was obtained from the reflectance of the sample on the black background (Yb) and the reflectance of the same sample on the white background (Yw), where

$$RC = Yb/Yw$$

The CR can vary from 0 to 1, where CR=0 is considered transparent and CR=1 is considered opaque. The CR obtained after the final evaluation of the tests on the effect of heat treatment and storage on dyes was evaluated. For greater understanding, the difference between the final and initial CR values (ΔRC) was also used

ANALYSIS OF FLEXURAL STRENGTH AND MODULUS OF ELASTICITY

For the flexural strength (FS) and modulus of elasticity (ME) tests, 60 specimens were used: 10 specimens for each composite and experimental condition (n=10). The CPs were made in the shape of a rectangular stick, measuring 10mm x 2mm x 1mm, using a split metal matrix.

To make the specimens, the metal matrix was positioned on a polyester tape that was on a microscope slide and the composite was inserted in a single increment into the mold, using a #1 suprafil spatula. After that, a polyester tape was positioned over the mold and a glass slide on top of the polyester tape and a 500g weight was placed on the device for 30 seconds. The glass sheet was then removed and photoactivation was carried out with the Radii light curing device (1000mW/cm^2) on the polyester tape on both sides of the composite for 20 seconds, with the tip of the photoactivator touching the polyester tape to standardize the distance from the light source. After photoactivation, the specimens were carefully removed from the mold and evaluated for possible defects under an optical microscope at 40x magnification (SZ61TR, Olympus, USA) in order to discard those with imperfections. They were then submitted to the respective TTs.

The flexural strength and modulus of elasticity were measured using a three-point bending test. The specimens were positioned in the EMIC DL 2000 universal testing machine (Emic, Curitiba, Paraná, Brazil), keeping a distance of 8 mm between the lower supports. A load of 50 N was applied to the center of the specimen at a displacement speed of 1 mm per minute until it broke. The strength values were obtained in MPa and were calculated using the formula: s = 3PL / 2wb2 where "P" is the load applied at the moment of fracture; "L" is the distance between the two support points (mm); "w" is the width (mm) and "b" is the thickness of the specimen (mm).

STATISTICAL TREATMENT

The data obtained was subjected to the Shapiro-Wilk test to check for normality and the Levene test to assess the homoscedasticity of the variances. Subsequently, a multifactorial analysis of variance was carried out for all the tests, and Tukey's test was used to contrast the means, at a 95% confidence level (α =0.05).

RESULTS

The data was subjected to the Shapiro-Wilk test and found to be normal, as well as the Levene test, which confirmed that the data was homoscedastic

COLOR DIFFERENCE

The variations in ΔE, ΔL, Δa and Δb can be used to compare the behavior of the composites after heat treatment and storage in dyes.

The results obtained before and after heat treatment (TT00, TT10 and TT30) were compared, followed by the color differences resulting from immersion in the dyes (WINE, COCA and WATER) in the four variables that show the color difference of the samples.

ANALYSIS ΔE

EFFECT OF THE DYE

There was a statistically significant difference between the mean values obtained, both for the factors (composites, heat treatments and dyes) alone and for the interactions between the factors (all results showed a p-value = 0.000).

a) **Comparison between the composites:** there was a significant difference in color between the two composites, with the nano-particulated composite having a greater color difference.

Composite	Nano-particulate	Hybrid
ΔE	2,21 ± 2,83 **A**	1,51 ± 1,56 **B**

Table 1 - Averages and standard deviations of the color difference (ΔE) for the composites tested

b) **Comparison between heat treatments:** there was a significant difference between the heat treatments, with TT00 showing the greatest difference in color and TT30 the least.

Heat treatment	TT00	TT10	TT30
ΔE	2,39 ± 3,06 **A**	1,72 ± 1,93 **B**	1,45 ± 1,60 **C**

Table 2 - Averages and standard deviations of the color difference (ΔE) for the temperatures tested

c) **Comparison between** colorants: the difference in color was not significant between the water and coca colorants, but they were statistically different from wine.

Dye	Water (H_2O)	Coca-Cola	Wine
ΔE	0,39 ± 0,08 **A**	0,46 ± 0,12 **A**	4,72 ± 1,83 **B**

Table 3 - Averages and standard deviations of the color difference (ΔE) for the tested dyes

Considering the **interactions between the factors** (composites with heat treatments, composites with dyes, heat treatments with dyes), we can see the differences listed below:

a) **Interaction of composite factors X heat treatments:** when comparing the results of the effects of heat treatment and composites, we can see that the nano-particulate composite without heat treatment had the highest ΔE and the hybrid with heat treatment for 30 minutes had the lowest ΔE. For the nano-particulate composite, heat treatment for 10 minutes or 30 minutes showed statistically similar performance. And in the case of the hybrid composite, heat treatment for 30 minutes had the best performance compared to no heat treatment and heat treatment for 10 minutes.

ΔE		
Composite/Thermal Treatment	**Nano-particulate**	**Hybrid**
TT00	3,07 ± 4,03 **A**	1.72 ± 1.78 **BC**
TT10	1,84 ± 2,16 **B**	1,60 ± 1,78 **C**
TT30	1.71 ± 1.96 **BC**	1,20 ± 1,19 **D**

Table 4 Averages and standard deviations of color difference (ΔE) for X TT composites

b) **Interaction between composites** and dyes**:** when comparing the composites and the dye solutions, it was observed that the greatest difference in color was found in the nano-particulated composite with wine dye, followed by the hybrid composite with wine dye. There was no difference between the nano-particulated and hybrid composites with the other coloring solutions (coca and water).

ΔE		
Composite/ Colorant	**Nano-particulate**	**Hybrid**
Water (H_2O)	0.34 ± 0.08 **C**	0.45 ± 0.04 **C**
Coca-Cola	0.46 ± 0.09 **C**	0.46 ± 0.16 **C**
Wine	5,82 ± 1,99 **A**	3.61 ± 0.65 **B**

Table 5 - Averages and standard deviations of the color difference (ΔE) for Composites X Dyes

c) **Interaction of factors heat treatments X coloring solutions** : the highest representative color change values were observed after immersion in wine, and there was no statistically significant difference between the color change effects of the composites after immersion in the other coloring solutions. Furthermore, for the composites immersed in the wine coloring solution, heat treatment proved to be effective in reducing the degree of color change. It was also observed that the composites that were subjected to TTPP for longer (30min.) experienced less color change than those subjected to TTPP for 10 minutes: wine TT30 < wine TT10 < wine TT00.

ΔE			
Heat treatment/colorant	**TT00**	**TT10**	**TT30**
Water (H_2O)	0,36 ± 0,06 **D**	0,37 ± 0,11 **D**	0,45 ± 0,03 **D**
Coca-Cola	0,55 ± 0,12 **D**	0,46 ± 0,12 **D**	0,46 ± 0,04 **D**
Wine	6,27 ± 2,39 **A**	4,34 ± 0,51 **B**	3,55 ± 0,87 **C**

Table 6 - Averages and standard deviations of color difference (ΔE) for heat treatments X dye solutions

Finally, in the **interaction between the factors " composites X heat treatments X dyes "**, the highest average color difference values were observed in the nano-particulated composite group without heat treatment and with storage in the wine dye solution. The composites immersed in the wine dye solution always showed a greater color difference than the composites immersed in the other dye solutions, regardless of the type of composite and the time of heat treatment. Considering the deleterious effects on maintenance caused by immersing the composites in the wine dye solution, the 30-minute post-polymerization heat treatment of the hybrid composite generated fewer chromatic changes when compared to the other experimental conditions. Regarding the color changes observed in the composites, after immersion in the wine dye solution, the nano-particulate composite showed the need for TTPP, regardless of the time. As for the hybrid composite, the most favorable results were seen after 30 minutes of TTPP. No statistically significant differences were observed in the interaction between the factors heat treatment and type of composite when immersed in coca or water.

ΔE			
Composite	**Nano-particulate**		
Heat treatment/colorant	TT00	TT10	TT30
Water (H_2O)	0,31 ± 0,01 **E**	0,27 ± 0,02 **E**	0,44 ± 0,02 **E**
Coca-Cola	0,44 ± 0,02 **E**	0,55 ± 0,08 **E**	0,37 ± 0,05 **E**
Wine	8,44 ± 0,09 **A**	4,71 ± 0,42 **B**	4.32 ± 0.37 **BC**
Hybrid composite			
Heat treatment/colorant	TT00	TT10	TT30
Water (H_2O)	0,41 ± 0,02 **E**	0,48 ± 0,04 **E**	0,46 ± 0,04 **E**
Coca-Cola	0,66 ± 0,03 **E**	0,36 ± 0,01 **E**	0,35 ± 0,02 **E**
Wine	4,09 ± 0,18 **C**	3,97 ± 0,26 **C**	2,78 ± 0,04 **D**

Table 7 - Averages and standard deviations of color difference (ΔE) for composites X heat treatments X dyes

ΔL ANALYSIS

Statistically significant differences were observed when considering the factors composites ($p=0.013$), heat treatments ($p=0.000$) and dyes ($p=0.000$) alone. Differences were also observed in the interactions of the factors **composites x dyes** ($p=0.000$), **heat treatments x dyes** ($p=0.000$) and the interaction of the factors **composites x dyes x heat treatment** ($p=0.000$). The interaction of the factors **composites x heat treatment** showed no statistically significant differences ($p=0.574$).

a) **Comparison between the composites:** Both composites showed negative and statistically significant variations in the average delta L values, the greatest variation was observed in the nano-particulate composite.

Composite	Nano-particulate	Hybrid
ΔL	-1,22 ± 1,56 **A**	-1,08 ± 1,28 **B**

Table 8 - Means and standard deviations ΔL for the composites

b) **Comparison between Heat Treatments**: Composites submitted to post-curing heat treatment showed better brightness results after immersion in the dye solutions. However, no statistically significant difference was observed between the 10 and 30 minute times.

Heat treatment	TT00	TT10	TT30
ΔL	-1,43 ± 1,79 **B**	-1,02 ± 1,17 **A**	-1,01 ± 1,25 **A**

Table 9 - Averages and standard deviations ΔL for the heat treatments

c) **Comparison between dyes** : the greatest variation in average delta L values was observed in the wine group, which was statistically different from the others. The composites immersed in water and coca stain solutions did not show statistically different results.

Dye	Water (H_2O)	Coca-Cola	Wine
ΔL	-3,04 ± 0,67 **B**	-0,26 ± 0,18 **A**	-0,15 ± 0,16 **A**

Table 10 Means and standard deviations ΔL for the dyes

d) **Interaction of factors composites x staining solutions** : No statistically significant differences were observed between the variations in the average delta L values for the two composites immersed in water or coca. Larger and statistically significant delta L variations were noted for both composites when immersed in wine stain solution. This variation (darkening) was statistically greater for the nano-particulated composite than for the hybrid, when immersed in the wine-colored solution.

ΔL		
Composite/ Colorant	Nano-particulate	Hybrid
Water (H_2O)	-0,08 ± 0,20 **A**	-0,21 ± 0,08 **A**
Coca-Cola	-0,31 ± 0,08 **A**	-0,22 ± 0,23 **A**
Wine	-3,27 ± 0,85 **C**	-2,82 ± 0,41 **B**

Table 11 - Averages and standard deviations ΔL for composites x dye solutions

e) **Interaction of factors heat treatments x staining solutions** : Higher degrees of darkening were observed for composites immersed in the wine staining solution. In this immersion medium, the worst results were observed in the absence of post-curing heat treatments, regardless of the TTPP time**.** The other experimental conditions of immersion media (water or coca) and TTPP had no statistically significant difference between them and had less darkening than the groups mentioned above.

ΔL			
Heat treatment/colorant	TT00	TT10	TT30
Water (H_2O)	-0,06 ± 0,28 **A**	-0,19 ± 0,02 **A**	-0,18 ± 0,04 **A**
Coca-Cola	-0,41 ± 0,14 **A**	-0,24 ± 0,17 **A**	-0,14 ± 0,12 **A**
Wine	-3,81 ± 0,70 **C**	-2,61 ± 0,15 **B**	-2,71 ± 0,20 **B**

Table 12 - Means and standard deviations ΔL for heat treatments x dye solutions

Interaction of factors " **composites x heat treatments x dye solutions "** Both composites showed negative ΔL, giving them darker shades. The exception was the nano-particulate composite without heat treatment, immersed in water, which showed a positive ΔL. The wine dye accentuated the difference in ΔL (darker) in the two composites. Also for immersions in wine, the heat treatments (TT10 and TT30) reduced the ΔL variation for both composites compared to TT00. The nano-particulated TT00 wine group showed the greatest darkening, followed by the hybrid TT00 wine group.

ΔL			
Nano-particulate composite			
Heat treatment/colorant	TT00	TT10	TT30
Water (H_2O)	0,19 ± 0,01 **A**	-0.21 ± 0.01 **AB**	-0.21 ± 0.01 **AB**
Coca-Cola	-0.28 ± 0.02 **AB**	-0,40 ± 0,05 **B**	-0.24 ± 0.04 **AB**
Wine	-4,27 ± 0,73 **E**	-2,67 ± 0,19 **C**	-2.87 ± 0.17 **CD**
Hybrid composite			
Heat treatment/colorant	TT00	TT10	TT30
Water (H_2O)	-0.32 ± 0.03 **AB**	-0.18 ± 0.01 **AB**	-0.15 ± 0.03 **AB**
Coca-Cola	-0,53 ± 0,02 **B**	-0.09 ± 0.01 **AB**	-0.04 ± 0.01 **AB**
Wine	-3,34 ± 0,17 **D**	-2,55 ± 0,09 **C**	-2,56 ± 0,06 **C**

Table 13 - Means and standard deviations ΔL for composites x heat treatments x dye solutions

ANALYSIS ΔA

The ANOVA analysis of variance showed a statistically significant difference between the mean Δa values of the factors composites alone (p=0.000), heat treatments (p=0.002) and dye solutions (p=0.000), as well as the interactions of the factors composites **x heat treatments** (p=0.000), **composites x dye solutions** (p=0.005), **heat treatments x dyes** (p=0.024) and the **interaction composites x dye solutions x heat treatment** (p=0.000).

a) **Comparison between the composites:** both composites showed a negative variation in the average Δa values obtained (tendency to green). The nano-particulated composite showed greater negative variations than the hybrid composite, with a statistically significant difference between the composites.

Composite	Nano-particulate	Hybrid
Δa	-0,28 ± 0,69 **B**	-0,20 ± 0,62 **A**

Table 14 - Means and standard deviations Δa for the composites

b) **Comparison between Heat Treatments:** Composites subjected to TTPP for 30 min showed the smallest variations in Δa, so the values were closer to the neutral axis (lower green tone). The average values obtained by this group were statistically higher than those of TT00 and TT10, which were statistically similar to each other.

Heat treatment	TT00	TT10	TT30
Δa	-0,28 ± 0,66 **B**	-0,26 ± 0,69 **B**	-0,19 ± 0,65 **A**

Table 15 - Means and standard deviations Δa for the heat treatments

a) **Comparison between the** dyes**:** when evaluating the effect of the dyes, it can be seen that there was no statistically significant difference between the water and coca groups, with positive results (redder). The wine dye group, on the other hand, showed a greener tone (negative result), differing from the others.

Dye	Water (H_2O)	Coca-Cola	Wine
Δa	0,18 ± 0,06 **A**	0,23 ± 0,13 **A**	-1,13 ± 0,24 **B**

Considering the interaction between the factors: composites X heat treatments X dyes, the following differences can be seen:

a) **Interaction of the factors composites X dyes** : the chromatic effect of the composites after immersion in the dyes was checked at the same time and it was found that there was no statistically significant difference between the coca and H_20 groups, with the predominant shade being red. Both composites showed greenish tones when immersed in the wine dye solution. The nano-particulated composite showed greater color variation than the hybrid, with a statistically significant difference between the two.

Δa		
Composite/ Colorant	**Nano-particulate**	**Hybrid**
Water (H_2O)	0,15 ± 0,06 **A**	0,20 ± 0,03 **A**
Coca-Cola	0,23 ± 0,17 **A**	0,24 ± 0,07 **A**
Wine	-1,23 ± 0,17 **C**	-1,04 ± 0,26 **B**

Table 16 - Averages and standard deviations Δa for composites X dyes

b) **Interaction between the factors heat treatment X dyes** : the wine group showed greener tones (negative results), with no statistically significant difference between the heat treatment times. In the groups immersed in coca and wine, the results were close to the neutral axis and positive, i.e. redder.

Δa			
Heat treatment/colorant	**TT00**	**TT10**	**TT30**
Water (H_2O)	0.18 ± 0.04 **ABC**	0.14 ± 0.07 **BC**	0.21 ± 0.03 **ABC**
Coca-Cola	0,12 ± 0,12 **C**	0.27± 0.13 **AB**	0.30 ± 0.04 **ABC**
Wine	-1.15 ± 0.30 **D**	-1,18 ± 0,21 **D**	-1,07 ± 0,22 **D**

Table 17 - Means and standard deviations Δa for thermal X dyes

c) **Interaction of the factors composites X heat treatments** : in the nano-particulate group without heat treatment, the greatest chromatic change was observed when compared to the other experimental conditions. The greatest chromatic change for the hybrid composite occurred during the 10-minute heat treatment.

Δa		
Composite/Thermal Treatment	**Nano-particulate**	**Hybrid**
TT00	-0.41 ± 0.75 **D**	-0.15 ± 0.56 **AB**
TT10	-0.19 ± 0.64 **AB**	-0.32 ± 0.76 **CD**
TT30	-0.25 ± 0.75 **BC**	-0.12 ± 0.58 **A**

Table 18 - Means and standard deviations Δa for composites X heat treatments

In the interaction between the factors " **composites X heat treatments X dyes** ", immersion in wine dye showed a negative Δa (green tones), regardless of the heat treatment time and the type of composite. Immersion in coca dyes and water gave the composites results close to 0. However, these results were positive (reddish tones) regardless of the heat treatment time and the type of composite.

Δa			
Nano-particulate composite			
Heat treatment/colorant	TT00	TT10	TT30
Water (H_2O)	0.16 ± 0.02 **ABC**	0.08 ± 0.02 **BC**	0.22 ± 0.02 **ABC**
Coca-Cola	0,01 ± 0,00 **C**	0,38 ± 0,07 **A**	0.28 ± 0.04 **AB**
Wine	-1,40 ± 0,08 **F**	-1.03 ± 0.03 **OF**	-1.24 ± 0.05 **EFF**
Hybrid composite			
Heat treatment/colorant	TT00	TT10	TT30
Water (H_2O)	0.21 ± 0.03 **ABC**	0.20 ± 0.04 **ABC**	0.20 ± 0.03 **ABC**
Coca-Cola	0.23 ± 0.03 **ABC**	0.16 ± 0.01 **ABC**	0,32 ± 0,02 **A**
Wine	-0,90 ± 0,17 **D**	-1,33 ± 0,19 **F**	-0,89 ± 0,15 **D**

Table 19 - Means and standard deviations Δa for composites X heat treatments X dyes

ANALYSIS ΔB:

Statistical analysis showed statistically significant differences when analyzing the factors composites, heat treatments and dyes alone, and in the double interactions: composite X heat treatment, composite X dyes and heat treatment X dyes, as well as the triple interaction of the factors composite X dye X heat treatment (all results showed a p-value = 0.000).

a) **Comparison between the composites:** a statistically significant difference was observed in the Δb values of the composites after immersion in the dyes. The nano-particulate composite showed the highest Δb value.

Composite	Nano-particulate	Hybrid
Δb	1,61 ± 2,42 **A**	0,49 ± 1,12 **B**

Table 20 - Means and standard deviations Δb for the composites

b) **Comparison between Heat Treatments**: Composites subjected to heat treatment showed less variation in Δb than those without heat treatment. The longer heat treatment time also generated a lower average Δb value.

Heat treatment	TT00	TT10	TT30
Δb	1,67 ± 2,63 **A**	0,93 ± 1,71 **B**	0,55 ± 1,17 **C**

Table 21 - Means and standard deviations Δb for the heat treatments

c) **Comparison between dyes:** small variations in Δb were observed when the composites were immersed in water and coca (with no statistically significant difference between these immersion media). The composites immersed in wine dye solution showed higher Δb values, which were statistically different from the other groups of dye solutions.

Dye	Water (H_2O)	Coca-Cola	Wine
Δb	-0,09 ± 0,28 **B**	0,04 ± 0,24 **B**	3,21 ± 2,08 **A**

Table 22 - Means and standard deviations Δb for the dyes

Considering the **interactions between** composites X dyes, heat treatments X dyes and composites X heat treatments, we can see the differences listed below:

a) **Interaction of factors composites X dyes** : no statistically significant differences in Δb were found between composites immersed in water and coca. Only composites immersed in the wine dye solution showed greater Δb variations. The greatest variation was observed in the nano-particulate composite.

Δb		
Composite/Colorant	Nano-particulate	Hybrid
Water (H_2O)	0,12 ± 0,21 **C**	-0,31 ± 0,13 **D**
Coca-Cola	0,11 ± 0,17 **C**	-0.04 ± 0.28 **CD**
Wine	4,60 ± 1,97 **A**	1,83 ± 0,98 **B**

Table 23 - Means and standard deviations Δb for composites X dyes

b) **Interaction of factors heat treatments X dyes** : the highest Δb values were observed for the wine dye. In this immersion medium, the smallest Δb variations were obtained in TT30, followed by TT10 and TT00, with statistically significant differences between these experimental conditions. Small variations close to the 0 axis were observed in the other groups.

Δb			
Heat treatment/ colorant	TT00	TT10	TT30
Water (H_2O)	0.02 ± 0.19 **DE**	-0,27 ± 0,13 **E**	-0.03 ± 0.39 **OF**
Coca-Cola	0,33 ± 0,04 **D**	-0,15 ± 0,17 **E**	-0.07 ± 0.06 **OF**
Wine	4,65 ± 2,72 **A**	3,22 ± 0,70 **B**	1.77 ± 1.35 **C**

Table 24 - Means and standard deviations Δb for heat treatments X dyes

c) **Interaction of factors composites X heat treatments** : in the nano-particulate composite, the heat treatments (TT10 and TT30) promoted lower Δb values. In the hybrid composite, heat treatment TT 00 = TT10 and TT30 showed the lowest Δb value, which was statistically different from the other groups (TT00 and TT10).

Δb		
Composite/Thermal Treatment	Nano-particulate	Hybrid
TT00	2.55 ± 3.43 **A**	0.78 ± 1.07 **CD**
TT10	1.19 ± 1.92 **B**	0,68 ± 1,55 **D**
TT30	1.09 ± 1.44 **BC**	0.02 ± 0.44 **E**

Table 25 - Means and standard deviations Δb for composites X heat treatments

In the interaction between the factors " **composites X heat treatments X dyes** ", both composites showed a positive ΔB for the wine dye, giving them more pronounced yellow tones and statistically differing from each other. The coca and water dyes showed results close to 0.

Δb			
Nano-particulate composite			
Heat treatment/ colorant	TT00	TT10	TT30
Water (H_2O)	0.19 ± 0.03 **EFGH**	-0.15 ± 0.01 **FGH**	0.32 ± 0.03 **EFGH**
Coca-Cola	0.34 ± 0.01 **EF**	0.01 ± 0.00 **EFGH**	-0.02± 0.00 **EFGH**
Wine	7,12 ± 0,36 **A**	3,72 ± 0,61 **B**	2,96 ± 0,54 **C**
Hybrid composite			
Heat treatment/ colorant	TT00	TT10	TT30
Water (H_2O)	-0.15 ± 0.02 **FGH**	-0,39 ± 0,06 **H**	-0.39 ± 0.06 **GH**
Coca-Cola	0.32 ± 0.06 **EFFG**	-0.31 ± 0.01 **FGH**	-0.13 ± 0.02 **FGH**
Wine	2,18 ± 0,15 **D**	2.72 ± 0.36 **CD**	0,58 ± 0,09 **E**

Table 26 - Means and standard deviations Δb for composites X heat treatments X dyes

CONTRAST RATIO - RC AND ΔRC ANALYSIS

This work evaluates the level of translucency of composites before and after heat treatment (TT00, TT10 and TT30) and then the differences in translucency resulting from immersion in colorants (WINE, COCA and WATER).

The values obtained were evaluated by two-factor analysis of variance (ANOVA) as a method for comparing the groups and Tukey's test was used, with 95% confidence (p-value less than 5% - $p < 0.05$) to determine whether there is a relationship between them.

RC analysis

After immersion in the dye solution, average RC values were obtained which showed a statistically significant difference when analyzing the factors composite, dye and the interactions composite X heat treatment X dyes.

a) **Comparison between the composites:** The comparison of the average RC values between the composites after immersion in dyes showed statistically significant differences between them.

Composite	Nano-particulate	Hybrid
RC	0,90 ± 0,02 **B**	0,93 ± 0,02 **A**

Table 27 - RC averages and standard deviations for composites

b) **Comparison between colorants:** the average RC values of the composites obtained after immersion in water and coca were statistically similar, only the composite immersed in wine showed higher RC values, statistically different from the other immersion media.

Dye	Water (H_2O)	Coca-Cola	Wine
RC	0,91 ± 0,01 **B**	0,90 ± 0,02 **B**	0,94 ± 0,02 **A**

Table 28 - Means and standard deviations of the RC for the dyes

Considering the **interactions of the factors** composites X heat treatments, composites X dyes, heat treatments X dyes, we see the differences listed below:

a) **Interaction of factors composites X heat treatments :** in the nano-particulate composite, no difference could be observed between the average CR values for the different heat treatments proposed. In the hybrid composite, heat treatment for 30 minutes increased translucency.

RC		
Composite/Thermal Treatment	**Nano-particulate**	**Hybrid**
TT00	0.90 ± 0.02 **C**	0.93 ± 0.02 **A**
TT10	0.90 ± 0.02 **C**	0.93 ± 0.02 **A**
TT30	0.91 ± 0.01 **C**	0.92 ± 0.01 **C**

Table 29 - RC averages and standard deviations for composites X heat treatments

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b) **Interaction of factors composites X dyes :** composites immersed in wine dye solution always showed higher RC values (greater opacity). The RC values of the hybrid composite were higher regardless of the immersion medium compared to the nano-particulate composite. The hybrid composite immersed in water and coca promoted statistically similar RC values. As for the nano-particulate composite immersed in coca, the RC values obtained were lower than all the other experimental conditions.

RC		
Composite/ Colorant	**Nano-particulate**	**Hybrid**
Water (H_2O)	0,90 ± 0,01 **D**	0,91 ± 0,00 **C**
Coca-Cola	0,89 ± 0,01 **E**	0.92 ± 0.01 **BC**
Wine	0,92 ± 0,01 **B**	0,95 ± 0,01 **A**

Table 30 - RC averages and standard deviations for composites X dyes

c) **Interaction of factors heat treatments X dyes** : immersing the composites in the water and coca dye solutions produced statistically similar values regardless of the heat treatment they were subjected to. For composites immersed in the wine dye solution, the contrast ratio was higher for TT00 and TT10 (more opaque).

RC			
Heat treatment/colorant	**TT00**	**TT10**	**TT30**
Water (H_2O)	0,91 ± 0,01 **C**	0,90 ± 0,02 **C**	0,90 ± 0,01 **C**
Coca-Cola	0,90 ± 0,02 **C**	0,90 ± 0,02 **C**	0,91 ± 0,01 **C**
Wine	0,94 ± 0,02 **A**	0,94 ± 0,01 **A**	0,93 ± 0,01 **B**

Table 31 - Means and standard deviations of RC for heat treatments X dyes

Finally, in the interaction of the factors " **composites X heat treatments X colorants**", the highest average RC values were observed in the hybrid composite immersed in wine for TT00 and TT10, these values were statistically higher than the other groups. The composites immersed in the wine coloring solution always obtained the highest RC values, although statistical differences between the average RC values were not always detected.

RC			
Nano-particulate composite			
Heat treatment/colorant	**TT00**	**TT10**	**TT30**
Water (H_2O)	0.91 ± 0.01 **DEF**	0.89 ± 0.01 **FG**	0.89 ± 0.01 **EFG**
Coca-Cola	0,88 ± 0,00 **G**	0,88 ± 0,00 **G**	0.90 ± 0.00 **DEF**
Wine	0.92 ± 0.00 **BCD**	0.93 ± 0.01 **BC**	0.93 ± 0.01 **BC**
Hybrid composite			
Heat treatment/colorant	TT00	TT10	TT30
Water (H_2O)	0.91 ± 0.01 **CDE**	0.92 ± 0.01 **BCD**	0.91 ± 0.00 **BCD**
Coca-Cola	0.91 ± 0.01 **BCD**	0.92 ± 0.00 **BCD**	0.91 ± 0.01 **BCD**
Wine	0,96 ± 0,00 **A**	0,95 ± 0,00 **A**	0,93 ± 0,01 **B**

Table 32 - RC averages and standard deviations for composites X heat treatments X colorants

ΔRC analysis

The ANOVA analysis of variance showed statistically significant differences between the mean ΔRC values ($p=0.000$) for the heat treatment factor (TT00, TT10 and TT30) and the dye factor analyzed in isolation and the interaction between the heat treatment and dye factors.

a) **Comparison between Heat Treatments:** the lowest average ΔRC value was observed in the TT30 group followed by TT10 and the highest value observed in TT00.

Heat treatment	**TT00**	**TT10**	**TT30**
ΔRC	0,020 ± 0,020 **A**	0,014 ± 0,016 **B**	0,009 ± 0,011 **C**

Table 33 - Means and standard deviations of ΔRC for the heat treatments

b) **Comparison between the dyes:** between the dyes, there was no statistically significant difference between the ΔRC values in the composites immersed in water and coca. The highest ΔRC value was observed in the composites immersed in the wine dye.

Dye	Water (H_2O)	Coca-Cola	Wine
ΔRC	0,004± 0,002 **B**	0,003 ± 0,003 **B**	0,035 ± 0,010 **A**

Table 34 - Means and standard deviations of ΔRC for the dyes

c) **Interaction of factors heat treatments X dyes** : the highest average ΔRC value was observed in the wine group. In this group, the heat treatment generated the smallest variations in ΔRC (TT00>TT10>TT30). For the other immersion media and heat treatments, no statistically significant differences were observed.

ΔRC			
Heat treatment/ colorant	**TT00**	**TT10**	**TT30**
Water (H_2O)	0,006 ± 0,001 **D**	0,004 ± 0,002 **D**	0,003 ± 0,001 **D**
Coca-Cola	0,0061 ± 0,0022 **D**	0,0021 ± 0,0002 **D**	0,0005 ± 0,0000 **D**
Wine	0,046 ± 0,008 **A**	0,036 ± 0,004 **B**	0,024 ± 0,004 **C**

Table 35 - Means and standard deviations of ΔRC for heat treatments X dyes

FLEXURAL STRENGTH AND MODULUS OF ELASTICITY

FLEXURAL STRENGTH (RF)

The average flexural strength values obtained showed statistically significant differences for the factors: composites ($p = 0.000$) and heat treatments ($p = 0.000$). As well as for the interactions between composites and heat treatments ($p = 0.010$).

The results and analysis of the composites and heat treatments **alone will** be presented below.

a) **Comparison between the composites:** The average flexural strength values were statistically different, the nano-particulate composite showed higher flexural strength values than the hybrid composite.

Composite	Nano-particulate	Hybrid
Maximum voltage	199,73 ± 39,22 **A**	173.23 ± 23.37 **B**

Table 36 - RF averages and standard deviations for composites

b) **Comparison between Heat Treatments:** Both composites subjected to heat treatment showed an increase in average flexural strength values. However, there was no difference between the heat treatment times (10 and 30 minutes).

Heat treatment	TT00	TT10	TT30
Maximum voltage	163,95 ± 23,49 **B**	195,40 ± 33,59 **A**	200,10 ± 35,25 **A**

Table 37 - RF averages and standard deviations for heat treatments

Considering the **interaction between the factors composites X heat treatments**, it was observed that the composites showed statistically different flexural strength values:

- The hybrid composite showed no statistically significant differences after the heat treatments;
- The nano-particulate composite showed statistically significant differences after the heat treatments. However, no statistically significant differences were observed between the different times tested (TT10 and TT30);

Maximum voltage		
Composite/Thermal Treatment	**Nano-particulate**	**Hybrid**
TT00	162,10 ± 29,73 **B**	165,80 ± 16,54 **B**
TT10	213,60 ± 28,24 **A**	177,20 ± 29,13 **B**
TT30	223,50 ± 29,33 **A**	176,70 ± 23,35 **B**

Table 38 - RF averages and standard deviations for composites X heat treatments

MODULUS OF ELASTICITY (ME)

The modulus of elasticity expresses the rigidity of the material and is the ratio between the variation in stress and the corresponding variation in deformation. The average modulus of elasticity values obtained showed statistically significant differences for the factors: composites and heat treatments. As well as for the interactions between composites and heat treatments.

The results and analysis of the composites and heat treatments **alone will** be presented below.

a) **Comparison between the composites:** The comparison of the average values of the modulus of elasticity between the composites showed statistically significant differences. The nano-particulated composite had a higher elasticity value, i.e. greater rigidity.

Composite	Nano-particulate	Hybrid
Modulus of Elasticity	9,07 ± 1,02 **A**	8,57 ± 0,98 **B**

Table 39 - Means and standard deviations of the ME for the composites

c) **Comparison between heat treatments:** The heat treatment caused an increase in the average modulus of elasticity values for the composites. However, there was no difference between the heat treatment times (10 and 30 minutes).

Heat treatment	TT00	TT10	TT30
Modulus of Elasticity	8,09 ± 0,81 **B**	9,34 ± 0,84 **A**	9,02 ± 1,00 **A**

Table 40 - Means and standard deviations of the ME for the heat treatments

Finally, in the interaction of the factors " **composites X heat treatments ",** the highest results for the modulus of elasticity were obtained after heat treatment, with the nano-particulate composite having a significantly higher modulus of elasticity than the hybrid composite with the heat treatments.

Modulus of Elasticity		
Composite/Thermal Treatment	**Nano-particulate**	**Hybrid**
TT00	8,01 ± 0,55 **C**	8.17 ± 1.04 **BC**
TT10	9,46 ± 0,99 **A**	9.22 ± 0.69 **AB**
TT30	9,72 ± 0,41 **A**	8.32 ± 0.92 **BC**

Table 41 Means and standard deviations of ME for composites X heat treatments

DISCUSSION

The hypothesis was partially accepted. In the evaluation of ΔE, the group without heat treatment, after immersion in dyes, showed the greatest difference in color and the group with heat treatment for 30 minutes showed the smallest difference. In the analysis of flexural strength and modulus of elasticity for the nano-particulated composite, both heat treatments (TT10 and TT30) showed an improvement and for the hybrid composite there was no difference between TT00, TT10 and TT30.

The data from the study can be interpreted clinically based on a classification of color differences in aesthetic restorations divided into three different groups: $\Delta E < 1$, imperceptible to the human eye; $1.0 < \Delta E < 3.3$, observed only by a professional and clinically accepTable ; and $\Delta E > 3.3$, perceptible to laypeople, i.e. not clinically accepTable.[22] To classify color tone variation we use: $\Delta L>0$, lighter, $\Delta L<0$, darker; $\Delta a>0$, redder, $\Delta a<0$, greener; $\Delta b>0$, more yellow; $\Delta b<0$, more blue. [7, 17, 20, 21] Douglas *et al.* [20] evaluated the a*b* axes individually and reported that accepTable variations were up to 1.1 for the a* axis (red-green) and up to 2.1 for the b* axis (blue-yellow). According to these authors, analysis of the a* and b* axes in isolation can be of great value when assessing the effect of coloring solutions on composites. This same evaluation is contraindicated for the analysis of the clinical performance of restorations, since the color variation can only be determined by analyzing the L*a*b* variables (together), i.e. from the determination of ΔE.

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The change in color of restorative composites may be related to their chemical properties, such as greater water sorption by their resin matrix, causing cracks in the interface with the inorganic filler particles and also enabling the absorption of dyes and discoloration. It is known that hydrophilic materials can be more degraded by water sorption than hydrophobic materials, but hydrophobic matrices such as Bis-GMA and UDMA are also susceptible to chemical reactions by alcohol. UDMA has lower water sorption than Bis-GMA, which reinforces that the sorption and solubility of composites are closely related to the type of resin matrix, its composition and also the characteristics of the dye in which they were immersed. In addition, the discrepancy between the light refractive index of the filler particles and the organic matrix can increase after water absorption and this considerably alters color perception [23, 24, 25, 26, 27].

After immersion in the different coloring solutions, the performance of the composites was compared. It was possible to see that there was a significant difference in color between the two composites, with the nano-particulate showing more discrepant values. However, the ΔE values obtained (2.21 and 1.51) are within the range perceived only by professionals. In the study by Leite *et al.* [24] it was also shown that nano-particulated composite showed greater staining as a result of storage time in staining solutions, compared to hybrid composite. This can be explained by the fact that the nano-particulate composite has a larger surface area for the filler particles than the hybrid composite.

In this study, when comparing the effects of dyes on composites subjected to heat treatment, it was possible to see that there was a statistically significant difference between the proposed heat treatments. The composites that were not heat-treated had the highest ΔE (2.39). While the TT30 group showed a lower value (1.45), and the TT10 group showed intermediate values to the other groups (1.72). Baldo's [7] study testing the effect of different post-polymerization heat treatment temperatures (100°C and 170°C for 10 minutes) on composites stored in different coloring solutions also found that lower ΔE variations after immersion in wine were obtained when the composites were subjected to TT at 170°C for 10 minutes, when compared to the group that was not subjected to heat treatment.

When comparing only the effect of the different dye solutions on the color variation of the composites, there was no statistically significant difference between the water (0.39) and coca (0.46) dyes, values which were imperceptible to the human eye. On the other hand, the effect caused on the composites after immersion in the wine dye (4.72) was not only statistically different from the others, but also perceptible to the layman (higher than 3.3). Other studies corroborate these results and demonstrate the occurrence of more pronounced pigmentation of wine in relation to the other dyes tested [7, 24].

The large difference in color between Coca-Cola® and wine can be explained by the degradation of the composite due to the acidic pH of Coca-Cola (around 2.5, according to the company's own website), which interferes with the integrity of the composite surfaces. In the case of water, this difference in color, although slight, can be explained by an intrinsic discoloration. In the case of red wine, this greater color difference can be explained by the fact that red wine is rich in anthocyanins, providing this strong coloration. The greater color difference observed after immersion in wine can be explained by the presence of alcohol, which can lead to degradation on the surface of the composite, resulting in a rougher surface and consequently a greater surface area for the adsorption of these pigments. The lower pH of the wine may also contribute to greater absorption of the dye [24, 25, 26].

In the present study, the nano-particulate composite group not subjected to heat treatment (TT00) showed the worst performance in terms of ΔE (3.07) when compared to the other groups. The hybrid composite with TT30 had the lowest average ΔE value (1.20), differing statistically significantly from the other composite x TT combinations. Although the discrepancy between the values obtained by these two groups in this test is evident, according to Douglas *et al.* [9] they fall within the range of variation (between 1 and 3.3) that would only be perceived by professionals

When analyzing the performance in relation to the ΔE variation of the composites immersed in different dye solutions, no statistically significant differences could be detected for both composites immersed in water and coca solutions. On the other hand, the worst performance was found for the nano-particulate composite in wine, followed by the hybrid composite also when immersed in wine. In the study Leite *et al.* [24] comparing nano-particulated and hybrid composites after storage in wine dye, there was a statistically significant change in color only after the 2nd week of storage, with both values being statistically different from each other.

When comparing the heat treatment and dye solutions, the worst ΔE results were seen in the TT00 wine group (6.27), followed by TT10 wine (4.34) and then TT30 wine (3.55), all of which were statistically different. When evaluating the mean ΔE values obtained in relation to visual perception, all the groups immersed in wine obtained ΔE values > 3.3, i.e. all easily observable and not clinically accepTable. The results of the coca and water group, regardless of the TT performed, showed no statistically significant differences and had ΔE < 1, being imperceptible to the human eye. These results are also confirmed in the studies by Leite *et al.*[24] which show greater pigmentation in wine and in Baldo *et al.* [7] in which the results of storage in wine after heat treatment showed lower ΔE when compared to the groups without heat treatment.

Finally, when making a general comparison of the ΔE (of the composites with heat treatments and with dyes), all the groups stored in wine had ΔE > 3.3, i.e. they are all easily observable and not clinically accepTable, except for the TT30 wine hybrid, which was only perceptible to professionals. All the other groups stored in coca and water, regardless of the heat treatment (00, 10 or 30) had ΔE < 1, being imperceptible to the human eye.

The contrast difference is known as Delta RC (ΔRC), which can be negative

(-) or positive (+): [28, 29]

- ΔRC positive = more opaque (less translucent)
- ΔRC negative = more translucent (less opaque)

Regarding the Contrast Ratio (CR), the hybrid composite (0.91), regardless of the TT performed, was more opaque compared to the nano-particulate composite (0.89). Despite this statistically significant difference, both are perceptible to the human eye (RC>0.07). A preliminary study found a relationship between the greater the amount of Bis-GMA present in the composition and the greater translucency presented by the composite. A possible explanation for this phenomenon is that the light refraction index of this monomer is close to that of silica, barium and zirconia particles (both 1.55). [33] In the case of the composites evaluated in this study, Z350 (nano-particulate) and Forma (hybrid) both have Bis-GMA in the formulae given on the package leaflets, but Forma does not give the amount of this monomer, making it impossible to compare the amounts of Bis-GMA between the composites.[31, 34] In the study by Fujita *et al.* [35] they concluded that the amount of light transmitted can be influenced by the size of the silica particle, so increasing the size can considerably reduce light transmission. The heat treatment did not show an increase in RC between the TT00, TT10 and TT30 groups for the hybrid composite, the values obtained were statistically similar and all the RC values were >0.07 (perceptible to the human eye). In the case of the nano-particulated composite, there was a difference in the RC value only between groups TT00 (0.88) and TT30 (0.90), also with all RC>0.07 (perceptible to the human eye).

For the differences in the Contrast Ratio (ΔRC) of each composite, the average values obtained before and after heat treatment were initially considered. Comparing the composites in isolation, both became more opaque, with the greatest difference being observed in the nano-particulate composite (ΔRC=0.004). The heat treatment analyzed alone or together with the composites showed that both post--polymerization heat treatments (TT10 or TT30) accentuated the difference in contrast ratio for both composites, making them more opaque compared to the groups without heat treatment. In the study by Junior Rodrigues *et. al.* [29] it was found that heat treatment did not alter the translucency of the conventional microhybrid composite, as well as the bulk-fill composites studied.

With regard to the flexural strength and modulus of elasticity values obtained, we observed higher average values for the nano-particulated composite (199.73 MPa and 9.07 GPa, respectively) and the hybrid composite (1.73.23 MPa and 8.57 GPa, respectively), with a statistically significant difference between the values. It was also possible to observe a statistically significant increase in relation to the group that was not subjected to heat treatment (163.95 MPa and 8.09 GPa, respectively) and the groups subjected to TT. There were no statistically significant differences in the mean values obtained for flexural strength and modulus of elasticity between groups TT10 (195.40 MPa and 9.34 GPa, respectively) and TT30 (200.10 MPa and 9.02 GPa, respectively). As in the study by Santana [36], which compared the times of 5, 10 and 15 minutes at a temperature of 170ºC, it was found that treatment for 5 minutes was enough to obtain a statistically higher value than the group without heat treatment. When comparing the times of 5, 10 and 15 minutes, there was no statistical difference between them. Although there was a considerable increase in the average values of flexural strength and modulus of elasticity for the nano-particulated composite after the proposed TTs, no statistically significant difference could be observed between the different treatment times (TT10 = 213.60 Mpa/ 9.46 GPa and TT30 = 223.50 Mpa/ 9.72 GPa, respectively). In the study by Baldo [7], the nano-particulated composite showed similar behavior when comparing the results without heat treatment and with heat treatment, with statistically significant differences between the groups with and without heat treatment, but with no statistically detecTable differences between the groups treated at 100ºC and 170ºC. For the hybrid composite, all the combinations with the TTs evaluated showed statistically similar average flexural strength values. The study by Junior Rodrigues *et al.* [29] also compared the modulus of elasticity between bulk-fill resins with and without heat treatment and concluded that heat treatment at 170º for 10 minutes increased the modulus of elasticity compared to the group without heat treatment.

CONCLUSIONS

Heat treatment at 170ºC for 30 minutes showed the lowest ΔE, followed by heat treatment at 170ºC for 10 minutes and the control group (TT30 minutes < TT10 minutes < control). In the case of the flexural strength and modulus of elasticity analysis, the nano-particulated composite showed improved results at both heat treatment times (TT10 and TT30) and for the hybrid composite there was no difference between the TT00, TT10 and TT30 groups.

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