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ORIGINAL ARTICLE

Changes in color and contrast ratio of resin composites after curing and storage in water



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KEYWORDS

CIELAB; Conservative dentistry; Dental materials; Resin composites Abstract *Purpose:* To verify the color change and contrast ratio of resin composites after curing and after 30 days of storage in water.

Methods: Dentin A2 shades of different light-cured dental resin composites (Vittra APS, FGM, Brazil; Z350 XT, 3M ESPE, EUA; Tetric N-Ceram, Ivoclar Vivadent, Liechtenstein, and Charisma Diamond, Heraeus Kulzer, Germany) were tested. Ten rounded specimens (8 mm × 2 mm) were prepared for each material. Reflectance for all samples was obtained using a spectrophotometer (Minolta CM 3700d, Konica Minolta, Japan) before curing, immediately after curing, and after 30 days of storage in water. The color change (ΔE^*lab) and contrast ratio (CR) data were analyzed using a one-way analysis of variance with Tukey's and paired t-tests ($\alpha = 1\%$).

Results: For all materials tested, significant color changes were noticeable after curing and after 30 days in water (p < 0.01). Significant changes in the CR values before curing, after curing, and 30 days of storage in water were observed in the resin composites investigated (p < 0.01) except for Z350 (p > 0.01).

Conclusion: The CR values and color changes after curing and 30 days of storage in water varied depending on the material tested. This study corroborates the clinical practice of curing a small amount of unpolymerized resin composite on the tooth surface to select the desired shade before undertaking esthetic restorative procedures.

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1. Introduction

Varied shades and opacities of resin composites provide a faithful reproduction of the optical properties of enamel and dentin (Villarroel et al., 2011). Shade matching between the tooth and the resin composite is still challenging because photopolymerization leads to changes in the optical properties of these materials (Çelık et al., 2011; Kim and Lee, 2007). Cure-dependent color changes of resin composites may include

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a higher translucency and reduction in the yellow chroma after light-curing (Seghi et al., 1990).

Despite improvements in the organic and inorganic composition of resin composites, color changes after curing are still noticeable (Çelık et al., 2011). In different studies, resin composites revealed significant color changes after curing when they were subjected to different conditions (Bayindir et al., 2016; Çelık et al., 2011; Da Silva et al., 2018; Erdemir et al., 2013; Falkensammer et al., 2013; Karaarslan et al., 2013; Sabatini, 2015). For this reason, it is suggested that an unpolymerized resin composite increment should be positioned over the compromised or adjacent tooth to confirm the shade of the resin composite before performing any restorative procedures (Çelık et al., 2011; Erdemir et al., 2013).

It is important to understand the optical properties of restorative materials so that an appropriate choice can be made. The color and contrast ratio are essential optical characteristics of restorative materials intended to mimic the tooth structure (da Costa et al., 2009). Therefore, this study aimed to evaluate changes in the color and contrast ratio of four resin composites after curing and after 30 days of storage in water. The null hypothesis tested was that changes in the color parameters and contrast ratios after curing and after 30 days of storage in water would be similar regardless of the resin composite used.

2. Material and methods

2.1. Specimen fabrication

Forty specimens (8 mm in diameter and 2-mm thick) were fabricated from four resin composites (n = 10 each): Vittra APS (VA), FGM, Joinville, SC, Brazil, Z350 XT (ZXT), 3M ESPE, St. Paul, MN, USA, Tetric N-Ceram (TNC), Ivoclar Vivadent AG, Schaan, Liechtenstein, and Charisma Diamond (CD), Heraeus Kulzer, Hanau, Germany. The sample size was calculated according to previous studies (Çelık et al., 2011; Karaarslan et al., 2013). Table 1 presents the manufacturer's information regarding the materials tested. The dentin A2 resin composites 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 (Domingos et al., 2011).

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 irradiation time was determined based on the manufacturers' recommendations and scientific literature (Daugherty et al., 2018) to obtain a dose of 20 J/cm². The output of the curing light was checked after ten consecutive uses with a radiometer for LED (LED Radiometer, SDI, Victoria, Australia). The LCU was placed directly over and perpendicular to the specimen surface. After curing, the height and diameter of each specimen were measured with a digital caliper (Mitutoyo, Aurora, Illinois, USA).

After curing, the specimens were stored individually in lightproof containers with deionized water for 30 days at a constant temperature of 37 °C (Albino et al., 2011; Çelık et al., 2011). The water was renewed every two days.

Table 1 Materials	tested and man	nufacturer's information.				
Composite	Classification	Matrix	Filler	% Load	Manufacturer	Batch #
Vittra APS	Nanofilled	UDMA, TEGDMA, APS Photo-initiator, co- initiators, stabilizers, silane and pigments.	zirconia and silica (200 nm)	52-60% v/72-82% wt	FGM, Joinville, SC, Brazil.	071118
Z350 XT	Nanofilled	bis-GMA, UDMA, TEGDMA, and bis EMA,	silica (20 nm), non-agglomerated/non-	63.3% v/78.5% wt	3M ESPE, St.	1911400352
		camphorquinone, and pigments.	aggregated zirconia (4–11 nm) and aggregated zirconia/silica (20 nm silica and 4–11 nm zirconia).		Paul, MN, USA.	
Tetric N-Ceram	Nano-hybrid	UDMA, bis-GMA, ethyoxylated bisphenol A dimethacrylate, pre-polymers, lucirin-TPO, additions, etablificant outputsts and nitrometic	Barium Glass, Ytterbium Trifluoride, mixed oxides, silicon dioxide (40 nm)	63.5% wt	Ivoclar Vivadent AG, Schaan, Listehtenstein	X49045
Charisma Diamond	Nano-hybrid	TCD-DI-HEA, UDMA, camphorquinone, and pigments.	Barium fluoride glass, aluminum, highly dispersed nano particles (5-20 μm)	64%v	Heraeus Kulzer, Hanau, Germany.	K010071

2.2. Color measurements

Measurements on white and black backgrounds for all samples were obtained before curing, immediately after curing, and after 30 days of storage in water. The samples were dried with gentle air pressure before the 30-day color measurements.

A spectrophotometer (Minolta CM 3700d, Konica Minolta, Japan) was used to measure all specimens. This device was set with D65 light using the second observation angle, 100% UV energy, and small aperture size. The same background, operator, place, and lighting conditions were considered for all samples. Three measurements were recorded for each sample, and the means were calculated. All color measurements were presented according to the Commission Internationale de l'éclairage L*, a*, and b* values. The CIELAB system provides values for L*a*b*, where L* represents lightness, a* the red-green axis, and b* the yellow-blue axis. Color differences (Δ E) were calculated using the following formula:

$$\Delta \mathbf{E} = \left[\left(\Delta L \right)^2 + \left(\Delta \mathbf{a} \right)^2 + \left(\Delta \mathbf{b} \right)^2 \right]^{1/2}$$

Ranges of ΔL^* , Δa^* , and Δb^* were also considered in the analysis.

The contrast ratio was calculated from the luminous 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) (Vichi et al., 2014).

2.3. Statistical analysis

Data were analyzed using GraphPad Prism version 7.0 (GraphPad Software, San Diego, CA, USA) with a significance level of 1%. Normal data distribution was verified using the Shapiro-Wilk test. A one-way analysis of variance (ANOVA) with Tukey's test was used to analyze the differences in the color parameters and contrast ratios between the composites. Multiple paired Student's t-tests were used to verify differences between the different evaluation periods within the same color parameter. A repeated-measures ANOVA with Tukey's test was used to analyze the differences in the contrast ratio between the differences in the contrast ratio between the differences in the contrast ratio between the different evaluation periods within the same composite.

3. Results

Table 2 shows the mean and standard deviation of the color parameters immediately after polymerization (initial) and after 30 days of storage in water, with a white background. Significant differences between the resin composites and different evaluation times were observed.

The initial ΔE values were significantly lower for VA, followed by ZXT, TNC, and CD (p < 0.01). After 30 days of storage in water, statistically higher values of ΔE were found for VA, followed by TNC (p > 0.01). ZXT and CD presented significantly lower color changes compared to the other resin composites (p > 0.01). VA showed significantly higher color changes in the 30-day evaluation than the initial ΔE values (p < 0.01). For ZXT and CD, color changes in the 30-day evaluation significantly decreased (p < 0.01), while no significant differences were found in the ΔE values of TNC (p > 0.01).

The resin composites presented different ranges of ΔL^* , Δa^* , and Δb^* values after curing and after 30 days of storage in water. Compared with the initial evaluation, after 30 days of storage in water, the ΔL^* values significantly increased for VA and decreased for ZXT and CD (p < 0.01). The Δa^* values were significantly reduced for ZXT (p < 0.01). Regarding the Δb^* values, they were significantly increased for VA and decreased for ZXT (p < 0.01).

Table 3 shows the mean and standard deviation of the contrast ratio of the resin composites before curing, after curing, and after 30 days of storage in water. Except for ZXT, the resin composites presented significant differences in the contrast ratio before curing, after curing, and after 30 days in water (p < 0.01). VA, TNC, and CD showed a statistically significant reduction in the contrast ratio after curing (p < 0.01). However, after 30 days, these values significantly increased and tended to be similar to the unpolymerized resin composite (p < 0.01).

4. Discussion

The null hypothesis of this study was rejected, as significant changes in the color parameters and contrast ratio of the resin

 Table 2
 Mean and standard deviation in color parameters of the composite resins after polymerization (initial) and after 30 days after polymerization.

	ΔΕ		ΔL^*		Δa^*		Δb^*	
Composites	Initial	30 days	Initial	30 days	Initial	30 days	Initial	30 days
VA	$3.06~\pm~0.38$	$11.0~\pm~0.92$	$1.65~\pm~0.43$	$3.12~\pm~1.34$	-0.845 ± 0.27	$-0.84 ~\pm~ 0.25$	$2.83~\pm~0.32$	-10.6 ± 0.87
	Aa	Ab	Aa	Ab	Aa	Aa	Aa	Ab
ZXT	$4.24~\pm~0.26$	$1.62~\pm~0.55$	$1.15~\pm~0.56$	$0.86~\pm~0.49$	$-1.07 ~\pm~ 0.05$	$-0.25 ~\pm~ 0.19$	$3.65~\pm~0.14$	-1.24 ± 0.72
	Ва	Bb	Ba	Bb	Aa	Bb	ABa	Bb
TNC	$4.04~\pm~0.41$	$4.20~\pm~0.78$	$3.18~\pm~0.20$	$3.79~\pm~0.92$	$-0.491 ~\pm~ 0.08$	$-0.62 ~\pm~ 0.24$	$2.24~\pm~0.65$	$1.29~\pm~1.02$
	Ва	Ca	Ca	Aa	Ba	ACa	ACa	CDa
CD	$5.00~\pm~0.47$	$1.98~\pm~0.68$	$3.78~\pm~0.15$	$1.33~\pm~0.67$	$-0.340 \pm 0.13B$	-0.43 ± 0.15	1.18 ± 0.547	$1.65~\pm~0.99$
	Ca	Bb	Da	Bb	Ва	BCa	Ca	Da

Different uppercase letters in the same column indicate significant differences in color parameter among the composite resins within the same evaluation period (One-way Anova with Tukey post-hoc test. p < 0.01); Different lowercase letters in the same line indicate significant differences between the different evaluation periods within same color parameter (Multiple paired student's *t* test - p < 0.01).

Table 3 Mean and standard deviation in contrast ratio of theresin composites tested before and immediately after curing and30 days of storage in water.

Composites	Before curing	After curing (Immediate)	After 30 days
VA	$0.99~\pm~0.12$	$0.64~\pm~0.05$	$0.86~\pm~0.06$
	Aa	Ab	Ac
ZXT	$0.77~\pm~0.06$	$0.81~\pm~0.05$	$0.84~\pm~0.04$
	Ba	Aa	Aa
TNC	$0.79~\pm~0.04$	$0.61~\pm~0.04$	$0.89~\pm~0.03$
	Ba	Ab	Ac
CD	$0.81~\pm~0.03$	$0.65~\pm~0.02$	$0.87~\pm~0.04$
	Ba	Bb	Ac

Different uppercase letters in the same column indicate significant differences in contrast ratio values between the resin composites within the same evaluation period (One-way ANOVA with Tukey post-hoc test, p < 0.01); Different lowercase letters in the same line indicate significant differences between the different evaluation periods within same resin composites (One-way ANOVA for repeated measures with Tukey's test, p < 0.01).

composites after curing and 30 days of storage in water were observed.

The color differences in this study were reported on a white background as white compensates for chromatic shortage (Villarroel et al., 2011). Color difference (ΔE) is defined by a numerical comparison in absolute color coordinates (L, a, and b) (Joiner, 2004). There is no current agreement on how much of a color difference is acceptable or perceptible (Douglas et al., 2007). A study organized changes in color into five different ranges: $0 < \Delta E < 1$, the difference is unnoticeable; $1 < \Delta E < 2$, the difference is only noticed by an experienced observer; $2 < \Delta E < 3.5$, the difference is also noticed by an inexperienced observer; $3.5 < \Delta E < 5$, clearly noticeable color differences; and 5 < ΔE , gives the impression that these are two different colors (Mokrzycki and Tatol, 2011). In this study, the color differences after curing were beyond the limit of color perceptibility, which is in agreement with other studies (Çelık et al., 2011; Lee and Powers, 2001).

In dentistry, lightness (L* - CIELAB) is known as "value," a visual perception according to which a specimen appears to reflect the incident light. The parameter a^* measures the color along with the red-green axis and b^* measures color along the yellow-blue axis (Paravina, 2004). In this study, we observed different ranges of ΔL^* , Δa^* , and Δb^* values after curing and after 30 days of storage in water.

The lowest color changes observed in the VA after curing may be due to the minimal differences in the refraction index of the matrix and filler particles during photopolymerization (Sidhu et al., 2006). Photoactivation may lead to changes in the lightness and a* values (Watanabe and Covey, 2008). In general, a chromatic shift toward the blue region of the color space resulting in a perceived decrease in yellow chroma from photoactivation has been observed (Kim and Lee, 2007; Watanabe and Covey, 2008).

The color change is effected by the CQ/amine ratio (Camargo et al., 2015; Lee et al., 2005). The coordinates a* and b* are dependent on the CQ/amine or Bis-GMA/ TEGDMA ratios, while L* is not. The b* values are directly related to the amount of amine (Camargo et al., 2015). Except for TNC, in which the photoinitiator was Lucirin® TPO, the resin composites tested contain CQ. It is well known that, after aging, resin composites with CQ became more yellow and with Lucirin® TPO lighter and less yellow. In addition, CQ systems were shown to have higher color stability than Lucirin® TPO (de Oliveira et al., 2015).

The APS technology employed in VA is alleged to result in less color change than other systems. This might be due to the composition, which is free of Bis-GMA and smaller quantities of CQ (Figuerêdo de Siqueira et al., 2020; Oliveira Jr et al., 2019). Corroborating this, in this study, VA presented lower ΔE values after curing compared to the other composites. However, after 30 days of storage in water, significant changes in the color parameters, superior to the clinically accepted values, were observed (Mokrzycki and Tatol, 2011).

The opacity level of resin composites is important for mimicking the translucency of enamel and dentin (Felippe et al., 2004). There is still no agreement on the translucency and opacity levels of commercial resin composites (Villarroel et al., 2011). The contrast ratio can be considered the inverse of translucency (da Costa et al., 2009; Inokoshi et al., 1996; Miyazaki et al., 2003). In this sense, a material with a high contrast ratio is relatively opaque and has lower translucency (Villarroel et al., 2011).

Except for ZXT, all resin composites exhibited differences in the contrast ratio before curing, after curing, and after 30 days. VA, TNC, and CD showed reduced CR after curing. A change in the organic matrix refractive index may cause a decrease in the opacity of the composite resins after polymerization (Azzopardi et al., 2009). This tended to be similar to that of unpolymerized composite after water hydration. A previous study showed an increase in translucency after polymerization of a composite resin, which was explained by reducing the difference between the filler/matrix refractive indexes (Diamantopoulou et al., 2014).

After storage, the contrast ratio was higher than that of the polymerized composite and was similar to its value before polymerization. Initially, the effect of water in the resin composite may cause softening of the organic matrix by hygroscopic expansion and hydrolysis or hydration of siloxane bonds of the silane layer (Krüger et al., 2018; Manojlovic et al., 2016; Panahandeh et al., 2017; Wang et al., 2018). Thus, an analysis of the components released from the resin composites in water could help to understand the color changes observed. As the manufacturers do not provide enough information on the composition and properties of resin composites, studies on their optical properties considering these factors should be performed.

The different results in the color and contrast ratios may be related to variations in their compositions. Despite the organic matrix (Malekipour et al., 2012), many variables can alter various characteristics of the resin composite materials, such as composite materials (Ong et al., 2018), nanofiller content (Scribante et al., 2015), and the presence of antibacterial substances (Almousa et al., 2019). In addition, dietary solvents (Eweis et al., 2018), brushing (Scribante et al., 2015), and finishing methods (Lopes et al., 2018) can alter the mechanical properties.

The color changes observed in this study should be analyzed in future *in vitro* studies to examine the long-term effects of discoloring agents and storage in water. In addition, *in vivo* studies should be considered to evaluate color changes in the oral environment. Thus, a broader experimental approach is necessary to comprehend the causes of the changes in color and contrast ratio and evaluate the color and opacity variations according to different shades and resin thickness.

5. Conclusion

In conclusion, significant changes in color and contrast ratio after curing and storage in water were noticeable. The changes varied depending on the material tested.

For the shade matching of resin composites, clinicians should be aware of color changes after polymerization.

Ethical statement

The author has no ethical issue to declare.

CRediT authorship contribution statement

Marlus Silva Pedrosa: Conceptualization, Methodology, Data curation, Writing - original draft, Supervision, Writing review & editing. Fernando Neves Nogueira: Conceptualization, Supervision, Writing - review & editing. Vitor Oliveira Baldo: Conceptualization, Data curation, Writing - original draft, Supervision, Writing - review & editing. Igor Studart Medeiros: Conceptualization, Supervision.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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