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Original Research Article

Color changes induced by light curing of resin composites

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Abstract

Introduction: Color changes that occur after the light-activation of resin composites should be understood. **Objective:** To evaluate the influence of light-curing devices on the color of resin composites immediately after light-activation and after one-week, at 37°C, into water storage. **Material and methods:** Three A2-shade composites (Z100, Z250, and Z350), and four light-curing devices (three halogens and one LED) were evaluated. Seventy-five cylindrical specimens were light-activated for 20s. CIE-Lab color was analyzed using a spectrophotometer. Color changes between uncured and immediately light-activated materials (ΔE_1), and between immediately light-activated and one-week-37°C-water-stored materials (ΔE_2) were obtained. Data were evaluated by two-way Anova, followed by Tukey test ($\alpha = 0.05$). **Results:** For ΔE_1 , composites ($p = 0.0008$), lights ($p = 0.015$), and the interaction ($p = 0.017$) were significant. Z100 showed the smallest value (3.08 ± 1.73). The halogen 210 mW/cm² device showed the smallest ΔE_1 (3.09 ± 1.25), while the LED 200mW/cm² showed the highest value (4.94 ± 2.37). For ΔE_2 , composites ($p = 0.00016$), lights ($p < 0.0001$), and the interaction effect ($p = 0.0002$) were significant. Z350 showed the smallest value (2.24 ± 1.17). The halogen 400mW/cm² device showed the smallest ΔE_2 (2.15 ± 2.15), while the halogen device 525mW/cm² showed the highest value (4.45 ± 2.15). **Conclusion:** The color of resin composites change significantly from the uncured to the cured and water-aged phases.

Introduction

The use of resin composites for dental restorations currently occupies a large part of the routine day of dentists due not only to patients' demands for esthetic restorations, but favorable handling properties, adhesive properties, and capacity of mimicking the dental structure as well [16]. In this context, the color of the material plays a major role in obtaining natural results, especially when maxillary anterior teeth and premolars are considered. Thus, an appropriate color matching is essential to the final outcome and patient's satisfaction. It is known, however, that resin composites may present color change after light-activation [6] due to changes in the polymer network at the post-cure period.

It has been demonstrated that curing is a process that continues over time and is not finished immediately after the completion of the light-activation [10, 11]. Thus, it is expected that a good color match between resin composites and the dental structure may be obtained only when the restorative material achieves its final polymerization, which may occur after days due the continued chain-reaction [10].

Several studies have been conducted to address the influence of exogenous factors such as dyes from food and beverages and weathering conditions on the color stability of resin-based materials [3, 9, 14, 22]. However, little is known about the variables influencing the intrinsic color change occurring after the light-activation [21]. Since the final color of a resin composite is curing-dependent, it may be hypothesized that different light-activation protocols would play a role in the final color of the material. This color change occurring at the post-cure period is important in the case of anterior teeth because patients may complain if a noticeable color mismatch occurs in a short period after the completion of the restoration.

Additionally, the incomplete polymerization has a considerable influence on the color stability [13, 15], since no resin-based material can achieve 100% of final curing [23]. The discoloration is commonly attributed to the degeneration of the chemical union between filler particles and resin matrix, and to the solubility of the resin matrix itself and the photo-initiator system [8, 24]. This process of discoloration may start immediately after light-activation upon contact of the cured resin composite with the oral environment.

The proper curing of resin composites is important to ensure optimum physical and mechanical properties. The effectiveness of polymerization depends not only on the chemical composition of the restorative material or the particle size and type, but also on the light-curing devices, including the light spectrum, exposure time and irradiance [24]. It has been observed that resin composites with increased degree of conversion have better mechanical properties, better wear resistance and improved color stability [7, 15].

Finally, the selection of the color of the resin composite to achieve a satisfactory restoration can be a difficult process because the available shade guides do not always reflect the color change that can occur after polymerization or after the first stages of hydration in contact with oral fluids. Thus, the purpose of this study was to evaluate the influence of two light-curing devices with different irradiances on the color change of three resin composites caused by the light-activation (immediately after light-activation) and after aging (one week of water storage). The null hypotheses evaluated were: 1) the color of uncured materials is similar to that of immediately light-activated ones; 2) the color of immediately light-activated materials is similar to that of one-week-stored ones.

Material and methods

The present study was designed to evaluate the color change of resin composites immediately after light-activation and after one week of water storage. The experimental design consisted of two factors: resin composites (in three levels) and light-curing devices (in five levels). Three A2-shade resin composites were evaluated: Filtek Z100, Filtek Z250 and Filtek Z350 (table I). Three halogen and two LED devices were evaluated. For the selection of the halogen light-curing devices, the measurement of all 25 units routinely used at the local undergraduate dental clinic was made using a radiometer (Model 100, Demetron Corporation Research, Danbury, CT, USA). Three units (Dabi Atlante, Ribeirão Preto, SP, Brazil) were selected: H1 with the highest irradiance (525 mW/cm²), H2 with intermediate irradiance (400 mW/cm²), and H3 with the lowest irradiance (210 mW/cm²). A LED light-curing unit was used (Ultrablue IS, DMC Equipments, São Carlos, SP, Brazil) with two irradiances: L1 (400 mW/cm²) and L2 (200 mW/cm²).

Table I - Resin composites employed in the study

Composite resin	Manufacturer and shade	Composition
Z 100	3M Espe, St Paul, EUA Shade A2	Bis-GMA, TEGDMA Filler: zirconia/ 0.6µm silica
Z 250	3M Espe, St Paul, EUA Shade A2	Bis-GMA, UDMA, Bis-EMA, PEGDMA and TEGDMA Filler: zirconia / 3 µm silica and surface-treated silica, 20 nm of non-agglomerated / aggregated non
Z 350	3M Espe, St Paul, EUA Shade A2	Bis-GMA, UDMA, TEGDMA, and bis-EMA (6) Filler: 20nm non-agglomerated / non-aggregated silica, 4-11nm non-agglomerated / non- aggregated zirconia and clusters of aggregated zirconia / silica particles

A Teflon mold was used to prepare cylindrical specimens of 7 mm in diameter and 2 mm in height. The resin composites were inserted in one portion and pressed between two glass coverslips. For each material (Z100, Z250, or Z350), five groups (n = 5)

were prepared depending on the type of light and irradiance employed (H1, H2, H3, L1, or L2). The 75 prepared specimens were light activated for 20 s each. The distribution of the groups, irradiance and energy applied is presented in table II.

Table II - Distribution of groups and energy applied

Group	Curing device	Irradiance (mW/cm²)	Energy (in Joules)
Z100 H1	Halogen	525	10.5 J
Z100 H2		400	8.0 J
Z100 H3		210	4.2 J
Z100 L1	LED	400	8.0 J
Z100 L2		200	4.0 J
Z250 H1	Halogen	525	10.5 J
Z250 H2		400	8.0 J
Z250 H3		210	4.2 J
Z250 L1	LED	400	8.0 J
Z250 L2		200	4.0 J
Z350 H1	Halogen	525	10.5 J
Z350 H2		400	8.0 J
Z350 H3		210	4.2 J
Z350 L1	LED	400	8.0 J
Z350 L2		200	4.0 J

The color readings were performed using a spectrophotometer (Easyshade, VITA Zahnfabrik, Bad Säckingen, Germany). The CIE-Lab color space was used to evaluate the color parameters L* (lightness), a* (red, when a* is positive; and green, a* is negative), and b* (yellow, when b* is positive; and blue, when b* is negative). The color was evaluated

three times: T1) before light-activation (uncured material), T2) immediately after light-activation, and T3) one week after dark storage in deionized water at 37°C. The average of three consecutive readings was considered as the value for each specimen. The color changes between uncured and immediately light-activated resin composites (ΔE_1) and between

immediately light-activated and one-week-stored resin composites (ΔE_2) were obtained by using the following formula:

$$\Delta E = ((\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2)^{0.5} \quad (1)$$

where ΔL^* , Δa^* and Δb^* are the differences in the respective values before and after each period as described above.

A color change (ΔE) of one unit was considered unperceivable [19]. Values higher than 1 unit and below 3.3 units were considered perceivable but clinically acceptable, and clinically unacceptable when higher than 3.3 [17].

Data of ΔE_1 and ΔE_2 were individually evaluated by two-way Anova and Tukey's HSD considering resin composites and light-curing device as independent

variables. A global level of significance of 5% was adopted.

Results

Mean values and standard deviations of ΔE_1 and ΔE_2 are shown in table III. For ΔE_1 there were significant differences between resin composites ($p = 0.0008$) and curing devices ($p = 0.015$). An interaction effect was also observed ($p = 0.017$). All values were higher than one unit, and, for this reason, were considered clinically perceivable. Considering the resin composites, Z100 showed the smallest ΔE_1 (3.08 ± 1.73) while Z250 and Z350 showed similar results (4.35 ± 1.87 and 4.67 ± 1.47 , respectively) ($p < 0.05$). Considering the light-curing devices, the halogen device H3 operating at 210 mW/cm^2 showed the smallest ΔE_1 (3.09 ± 1.25), while the LED L2 200 mW/cm^2 showed the highest value (4.94 ± 2.37).

Table III - Mean values and standard deviations of ΔE_1 (color changes between uncured and immediately light-activated resin composites) and ΔE_2 (between immediately light-activated and one-week-stored resin composites)

Group	ΔE_1	ΔE_2
Z100 H1	4.14 \pm 1.44 AB	3.19 \pm 1.44 AB
Z100 H2	4.28 \pm 2.76 AB	3.38 \pm 0.93 AB
Z100 H3	2.23 \pm 1.05 A	3.47 \pm 0.89 AB
Z100 L1	2.50 \pm 1.13 A	3.32 \pm 2.04 AB
Z100 L2	2.23 \pm 3.64 A	4.01 \pm 0.28 B
Z250 H1	3.72 \pm 1.16 AB	6.39 \pm 2.38 C
Z250 H2	4.89 \pm 1.73 AB	1.47 \pm 0.71 A
Z250 H3	3.24 \pm 1.48 AB	2.19 \pm 0.51 AB
Z250 L1	3.62 \pm 1.85 AB	2.75 \pm 0.69 AB
Z250 L2	6.25 \pm 5.85 B	3.72 \pm 1.28 AB
Z350 H1	3.89 \pm 1.29 AB	3.77 \pm 1.12 AB
Z350 H2	4.20 \pm 1.35 AB	1.61 \pm 0.89 A
Z350 H3	3.8 \pm 0.77 AB	1.67 \pm 0.89 A
Z350 L1	5.17 \pm 0.98 AB	1.71 \pm 0.49 AB
Z350 L2	6.33 \pm 1.47 B	2.45 \pm 0.96 AB

Different uppercase letters indicate statistically significant differences for the same column ($p < 0.05$)

For ΔE_2 there were significant differences among resin composites ($p = 0.00016$) and curing devices ($p < 0.0001$). An interaction effect was also observed ($p = 0.0002$). Almost all groups showed ΔE_2 values between one and 3.3 units, except Z100 L2 and Z250 H1 which showed the highest values (4.01 ± 0.28 and 6.39 ± 2.38 , respectively). Considering the resin composites, Z350 showed the smallest ΔE_2 (2.24 ± 1.17) while Z100 and Z250 showed similar results (3.47 ± 0.86 and 3.30 ± 2.11 , respectively) ($p < 0.05$). Considering the light-curing devices, the halogen device H2 operating at 400 mW/cm^2 showed the smallest ΔE_2 (2.15 ± 2.15), while the halogen device H1 operating at 525 mW/cm^2 showed the highest value (4.45 ± 2.15).

Discussion

The present study was designed to evaluate the influence of light curing units with different irradiances on the color change behavior of resin composites of the same brand. This idea was based on the fact that manufacturers in general present the light-activation time but do not provide the appropriate irradiance. Thus, depending on the characteristics of light-curing device one may expect differences in all polymerization-related properties such as degree of conversion, hardness, and shrinkage as well as color change.

The selection of the color of a tooth to be restored with resin composites is often performed visually by using shade guides and applying a small layer of the resin composite on the surface of the tooth to be restored to check if the color selection was appropriate. This technique of color selection by directly comparing the teeth with multiple color patterns, however, rarely gives an exact match between the color of the tooth and the final color of the resin composite restoration. For this reason, the present study evaluated how the color of different resin composites behaves over time from the uncured resin composite to the one-week-stored. Both null hypotheses evaluated were rejected. As demonstrated in the present study, seven days after the light activation of the resin composite there were significant color changes at all stages in which the color was evaluated. In fact, being higher than one unit, all values of ΔE_1 and ΔE_2 were considered clinically relevant and perceivable visually. These results are in agreement with another study published recently [18].

The problems inherent to the restorative material may be responsible for discrepancies in color match, such as discoloration. This is a result

of several factors such as polymerization, chemical reactions, exposure to different light sources, water absorption, surface smoothness and die [12, 13, 24]. It has been suggested that the susceptibility to color changes of resin composites is more related chemical alterations occurring at the resin matrix [12]. Since in the present study the resin composites were not exposed to staining agents, the color alterations resulted from internal reactions within the polymer network. In the present study, it could be assumed that the color changes observed immediately after light-activation (ΔE_1) was caused by modifications in the network structure of the monomers being converted into polymers. This initial color change may be attributed to the changes in the translucency while the material is curing as well to a process so-called photobleaching in which photoinitiators break their chromophore groups after light-activation with the effect of diminishing their yellow color [1, 5].

On the other hand, the color changes observed between the immediately light-activated materials and the one-week-stored ones could be attributed to both post-irradiation conversion and hydrolytic degradation. It is important to discuss the difference between expected color changes caused by post-irradiation conversion as opposed to the color change caused by a possible aging effect caused by the storage in water at 37°C for one week. The post-irradiation conversion, also known as dark-cure, is a phenomenon related to the continued chain reaction that occurs after the light-activation and continues over time. It has been shown that resin-based materials continue to cure over time and properties such as degree of conversion, shrinkage and hardness increase [10]. The hydrolytic degradation is related to the leaching of photoinitiators and unreacted monomers by water dissolution.

The resin composites evaluated showed significant color changes before and after polymerization (ΔE_1) regardless of the employed light source. The same occurred to color of the different resin composites after storage in water for one week (ΔE_2). Both ΔE_1 and ΔE_2 showed clinically relevant values that were higher than one unit, while no correlation to the type of light curing device or its irradiance was observed. This fact indicated that changes are inherent to the resin composites. Thus, it is expected that the final color of a resin restoration may be obtained only in days after light curing.

There are several methods for aging resin composites (e.g. UV lights and immersion in colorants such as coffee and wine). As the main

idea of the present study was to address the color change in the short term and not to simulate years of clinical exposure to UV or colorants, the immersion in water was used instead. This is a simpler method and is directly related to which in fact occurs immediately after a restoration is made. The color changes demonstrated in both ΔE_1 and ΔE_2 may not only impact the shade selection of a restoration but also the patient and clinician's acceptance of the esthetic result.

The CIE-Lab color evaluation used in the present study is still the standard procedure to evaluate color change in dentistry as it allows direct comparisons of visual perceptibility between studies in the literature. It should be noted that the color change could also be obtained by the CIEDE2000 standard. However, the CIEDE2000 (ΔE_{00}) is more indicated for small color changes when compared samples that have colors close together [2, 20]. Although, CIE-Lab and CIEDE2000 color changes evaluations generate different absolute values, it has been shown that changes in color follow similar trends [4].

Future research should be developed to more accurately predict the changes in color of different resin composites when considering the effects of polymerization and the effects of aging in the short, medium and long terms. Finally, it can be suggested that the resin composite should be cured before the color selection, as comparing color of the tooth to the uncured resin composite may cause errors in visual perception.

References

1. Asmusen S, Arenas G, Cook WD, Vallo C. Photobleaching of camphorquinone during polymerization of dimethacrylate-based resins. *Dent Mater.* 2009 Dec;25(12):1603-11.
2. Commission Internationale de l'Eclairage. CIE technical report: colorimetry. Viena: CIE; 2004.
3. Cunha LF, Santana LOC, Baratto SSP, Mondelli J, Haragushiku GA, Gonzaga CC et al.. Staining susceptibility of methacrylate and silorane-based materials: influence of resin type and storage time. *RSBO.* 2013;10(2):161-6.
4. de Oliveira DC, Ayres AP, Rocha MG, Giannini M, Puppim Rontani RM, Ferracane JL et al.. Effect of different in vitro aging methods on color stability of a dental resin-based composite using CIELAB and CIEDE2000 color-difference formulas. *J Esthet Restor Dent.* 2015 Sep-Oct;27(5):322-30.
5. del Mar Perez M, Saleh A, Pulgar R, Paravina RD. Light polymerization-dependent changes in color and translucency of resin composites. *Am J Dent.* 2009 Apr;22(2):97-101.
6. Diamantopoulou S, Papazoglou E, Margaritis V, Kakaboura A. Change of optical properties of contemporary polychromatic resin composites after light curing and finishing. *Int J Esthet Dent.* 2014 Summer;9(2):224-37.
7. Ferracane JL. Resin composite – state of the art. *Dent Mater.* 2011 Jan;27(1):29-38.
8. Fontes ST, Fernandez MR, de Moura CM, Meireles SS. Color stability of a nanofill composite: effect of different immersion media. *J Appl Oral Sci.* 2009 Sep-Oct;17(5):388-91.
9. Furuse AY, Gordon K, Rodrigues FP, Silikas N, Watts DC. Colour-stability and gloss-retention of silorane and dimethacrylate composites with accelerated aging. *J Dent.* 2008 Nov;36(11):945-52.
10. Furuse AY, Mondelli J, Watts DC. Network structures of Bis-GMA/TEGDMA resins differ in DC, shrinkage-strain, hardness and optical properties as a function of reducing agent. *Dent Mater.* 2011 May;27(5):497-506.
11. Furuse AY, Santana LO, Rizzante FA, Ishikiriyama SK, Bombonatti JF, Correr GM et al.. Delayed light activation improves color stability of dual-cured resin cements. *J Prosthodont.* 2016 Jul 25.
12. Jeong TS, Kang HS, Kim SK, Kim S, Kim HI, Kwon YH. The effect of resin shades on microhardness, polymerization shrinkage, and color change of dental composite resins. *Dent Mater J.* 2009 Jul;28(4):438-45.
13. Nasim I, Neelakantan P, Sujeer R, Subbarao CV. Color stability of microfilled, microhybrid and nanocomposite resins – an in vitro study. *J Dent.* 2010;38 Suppl 2:e137-42.
14. Oliveira DC, Souza-Junior EJ, Prieto LT, Coppini EK, Maia RR, Paulillo LA. Color stability and polymerization behavior of direct esthetic restorations. *J Esthet Restor Dent.* 2014 Jul-Aug;26(4):288-95.
15. Pires-de-Souza F de C, Garcia L da F, Hamida HM, Casemiro LA. Color stability of composites subjected to accelerated aging after curing using either a halogen or a light emitting diode source. *Braz Dent J.* 2007;18(2):119-23.
16. Pontons-Melo JC, Pizzatto E, Furuse AY, Mondelli J. A conservative approach for restoring

anterior guidance: a case report. *J Esthet Restor Dent.* 2012 Jun;24(3):171-82.

17. Ruyter IE, Nilner K, Moller B. Color stability of dental composite resin materials for crown and bridge veneers. *Dent Mater.* 1987 Oct;3(5):246-51.

18. Sabatini C. Color stability behavior of methacrylate-based resin composites polymerized with light-emitting diodes and quartz-tungsten-halogen. *Oper Dent.* 2015 May-Jun;40(3):271-81.

19. Seghi RR, Johnston WM, O'Brien WJ. Performance assessment of colorimetric devices on dental porcelains. *J Dent Res.* 1989 Dec;68(12):1755-9.

20. Sharma G, Wu W, Dalal EN. The CIEDE2000 Color-difference formula: Implementation notes, supplementary test data, and mathematical observations. *Color Res Applic.* 2005;20(1):21-30.

21. Uchimura JY, Sato F, Bianchi G, Baesso ML, Santana RG, Pascotto RC. Color stability over time of three resin-based restorative materials stored dry and in artificial saliva. *J Esthet Restor Dent.* 2014 Jul-Aug;26(4):279-87.

22. Vance M, Lawson NC, Rupal M, Beck P, Burgess JO. Color and gloss of nano-filled resin-modified glass ionomers and resin composites. *J Esthet Restor Dent.* 2014 Dec 1.

23. Watts DC. Reaction kinetics and mechanics in photo-polymerised networks. *Dent Mater.* 2005 Jan;21(1):27-35.

24. Yazici AR, Celik C, Dayangac B, Ozgunaltay G. The effect of curing units and staining solutions on the color stability of resin composites. *Oper Dent.* 2007 Nov-Dec;32(6):616-22.