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Water sorption and color stability of dental composites light-cured with a broadband LED device and different radiant exposures

Sorção de água e estabilidade da cor de compósitos dentários fotopolimerizados com um dispositivo LED de amplo espectro e diferentes exposições radiantes

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ABSTRACT

Objective: This study assessed the properties of four resin-based composites (Charisma Classic, Charisma Diamond, Filtek Z350XT, and Filtek Bulk Fill) concerning water sorption and color stability under different radiant exposures of a broadband LED device (Valo Cordless, Ultradent) and immersion solutions. **Material and Methods:** Disc-shaped specimens of the composites were prepared and subjected to two different radiant exposures: 20 J/cm² at 1000 mW/cm² for 20 seconds and 16.8 J/cm² at 1400 mW/cm² for 12 seconds. Water sorption (SO) was measured using a precision weighing scale, while color stability (ΔE_{00}) was evaluated after immersion in distilled water, coke soft drink, or beer for 28 days. Factorial ANOVA and Tukey HSD test ($\alpha = 5\%$) were employed for data analysis. **Results:** Significant differences were observed in water sorption (p = 0.000) among the composites and radiant exposures (p = 0.022). Likewise, color stability (ΔE_{00}) exhibited significant differences among composites ($p = 0.000$), radiant exposures ($p = 0.000$), and immersion solutions ($p = 0.000$). Interaction between factors was also significant (p = 0.000). **Conclusion:** Radiant exposure significantly impacts the properties of resin-based composites, particularly affecting water sorption and color stability. Optimal performance in clinical settings can be achieved by tailoring polymerization conditions. The study found that exposure to 20 J/cm² at 1000 mW/cm² for 20 seconds resulted in the lower water sorption values, while the lowest color alteration ($\Delta \rm{E_{00}}$) was observed with 16.8 J/cm 2 at 1400 mW/cm 2 for 12 seconds.

KEYWORDS

Color; Composite resins; Curing lights; Dental restoration; Esthetics.

RESUMO

1 Braz Dent Sci 2024 July/Sept;27 (3): e4379 **Objetivo:** Este estudo avaliou as propriedades de quatro compósitos à base de resina (Charisma Classic, Charisma Diamond, Filtek Z350XT e Filtek Bulk Fill) em relação à sorção de água e estabilidade da cor sob diferentes exposições radiantes de um dispositivo LED de amplo espectro (Valo Cordless, Ultradent) e soluções de imersão. **Material e Métodos:** Foram preparados espécimes em forma de disco dos compósitos e submetidos a duas exposições radiantes diferentes: 20 J/cm² a 1000 mW/cm² durante 20 segundos e 16.8 J/cm² a 1400 mW/cm² durante 12 segundos. A sorção de água (SO) foi medida usando uma balança de precisão, enquanto a estabilidade da cor (∆E00) foi avaliada após imersão em água destilada, refrigerante de cola ou cerveja por 28 dias. Para a análise dos dados, foram utilizados ANOVA fatorial e o teste HSD de Tukey (α = 5%). **Resultados:** Foram observadas diferenças significativas na sorção de água ($p = 0.000$) entre os compósitos e exposições

radiantes (p = 0.022). Da mesma forma, a estabilidade da cor (ΔE_{00}) apresentou diferenças significativas entre os compósitos (p = 0.000), as exposições radiantes (p = 0.000) e as soluções de imersão (p = 0.000). A interação entre fatores também foi significativa (p = 0.000). **Conclusão:** A exposição radiante tem um impacto significativo nas propriedades dos compósitos à base de resina, afetando particularmente a absorção de água e a estabilidade da cor. O desempenho ideal em ambientes clínicos pode ser alcançado através da adaptação das condições de polimerização. O estudo concluiu que a exposição a 20 J/cm² a 1000 mW/cm² durante 20 segundos resultou nos valores mais baixos de sorção de água, enquanto maior estabilidade de cor (∆E00) foi observada com 16,8 J/cm² a 1400 mW/cm² durante 12 segundos.

PALAVRAS-CHAVE

Cor; Resinas compostas; Lâmpadas de polimerização dentária, Restauração dentária permanente, Estética.

INTRODUCTION

The association of aesthetic, mechanical, handling properties and the low cost makes resin composites currently be the material of choice for direct restoration of teeth [1-4]. However, the stability and longevity of this material are still a major concern since factors of the oral environment affect these parameters [5,6].

Properties of the resin-based composites can be influenced by their physic-chemical composition and by extrinsic factors, such as the buccal environment and polymerization conditions [5-7]. The oral environment directly influences the hydrolytic degradation of the material through constant changes in temperature and pH from the presence of chemicals such as water, saliva, acids. The deterioration begins with the absorption of water by the material, which consists of a controlled diffusion process, causing the chemical degradation of the material, leading to problems such as detachment of the particles of the polymer matrix, thus reducing the wear resistance and decreasing its mechanical properties [8,9].

In general, the amount of water sorption by the polymer matrix is influenced by the degree of conversion and the degree of hydrophilicity of the polymer chains [10,11]. On the other hand, the evaluation of mechanical and physical properties can provide an indirect measure for predicting the conversion of monomers in composite resins [8,10,12]. The absorption of water also contributes to the hygroscopic expansion and consequently hygroscopic stress of the material, which can result in microcracks or even cracks in the cusps of restored teeth [8].

Color alteration of a resin composite restoration is caused by intrinsic and extrinsic factors, such as discoloration of the resin itself

associated with insufficient polymerization, immersion in liquid, adsorption of food dyes and heating [13-17]. Dyes present in cola-based soft drinks, tea, coffee, wine, and juices can alter the color of teeth and resin composites interfering with the esthetical properties [13,15,18].

The higher the percentage of unreacted monomers in the polymer matrix, the greater the occurrence of the sorption and pigmentation phenomena in the resin composite [19,20].

Therefore, the success of resin-based restorations also depends on the polymerization method and efficiency. For a suitable polymerization the material must receive a certain dose of energy for a certain time, which is called radiant exposure, which is the product of the irradiance $(mW/cm²)$ by the time (s). Thus, different combinations of time and irradiance can lead to significant differences in material properties within the same radiant exposure [21,22]. It has been emphasized that new generations of light emitting diodes (LED) can reduce the time of irradiation, decreasing clinical working time without significant loss of the mechanical properties [23].

New generations of LED include broadband, also known as polywave, devices that contains both blue and violet spectra. These devices have been developed to offer clinicians multiple irradiances and exposure times. This is an important issue since such devices are advertised as having standard (lower irradiances) and high power/turbo modes (higher irradiances), while manufacturers recommend reduced light activation times (lower radiant exposures) when high power modes are used. This variability in irradiance and exposure times necessitates further investigation to determine whether different radiant exposures can produce polymers with distinct properties [24]. With the development of new composite resin

formulations and photoinitiators, it is essential to continuously evaluate their behavior under various polymerization protocols, since, the formation of the polymer network may vary significantly, depending on the radiant exposure and chemycal composition of the material. Existing studies often isolate individual properties, whereas our research aims to explore the combined impact of water absorption and color stability, both crucial for the long-term aesthetic and functional performance of composites.Thus, further studies should be conducted to evaluate whether this difference in radiant exposure would produce polymers with different properties. The objective of this study was to evaluate the degree of water sorption of resin composites, cured with two radiant exposures after 7 days of storage in distilled water and the color stability after 28 days of immersion in staining solutions. Null hypothesis was that there would be no differences in the water sorption and color stability of different resin composites when two radiant exposures were used.

MATERIAL AND METHODS

Factors analyzed were resin composite in four levels, and the radiant exposure in two levels. Quantitative response variables were the degree of water sorption (SO) in μ g/mm³ and color stability (ΔE_{00}). Four resin composites were evaluated: Charisma Classic A2 (Heraeus Kulzer GmbH, Hanau, Germany), Charisma Diamond A2 (Heraeus Kulzer GmbH, Hanau, Germany), Filtek Z350XT A2B (3M ESPE, Saint Paul, USA) and Filtek Bulk Fill A2 (3M ESPE, Saint Paul, USA). The broadband LED device utilized was the Valo Cordless (Ultradent, South Jordan, USA) in two radiant exposures: 20 J/cm² with the device operating at Standard Mode, with 1000 mW/cm² , for 20 seconds; and 16.8 J/cm² with the device operating at High Power Mode, with $1400 \,\mathrm{mW/cm^2}$, for 12 seconds. These exposure times were used according to the LED device manufacturer's instructions.

For SO resin composite specimens were prepared in a metallic mold with dimensions of 5 mm in diameter and 2 mm in thickness for each experimental group ($n = 10$). The material was placed in a single increment and at the top of the mold a polyester strip was fitted along with a glass slide for microscopy to press the resin composite to avoid blistering and remove any surplus material. Then specimens were

polymerized with a broadband LED device (Valo Cordless, Ultradent, South Jordan, United States) following the light-activation protocol according to the radiant exposure/irradiance level of each group. The specimens were then individually stored in a desiccator containing silica gel at 37 °C \pm 1°C for 24 hours.

After storage, the specimens were weighed on a precision weighing scale. Using a digital caliper (Digmatic Caliper, Mitutoyo Sul Americana, São Paulo, Brazil) the measurements of the specimen volume calculated in mm3 were obtained. Subsequently, the specimens were stored with 5 mL of distilled water at 37 °C \pm 1°C for 7 days. The samples were weighed daily on an analytical scale until a constant mass (m1) was obtained. The specimens were then returned to the desiccator containing silica gel at 37 °C \pm 1°C and weighed daily until a constant mass (m2) was obtained. The water sorption (SO) values in μ g/mm³ were calculated with the following formula:

$$
SO = m1-m2/v \tag{1}
$$

Where $m1 = i$ s the mass after immersion in water, m2 = is the mass after the second desiccation cycle and $v =$ is the volume of the specimen in mm³.

As for the color stability, the color was evaluated according to the CIEDE2000 (ΔE_{∞}) equation after aging in different immersion solutions. The specimens were manually polished with a sequence of polishing discs from coarse to super fine (Sof-Lex, 3M Espe. St. Paul, USA), placed in ultrasonic water bath for 2 minutes, then stored into distilled water at 37 °C \pm 1°C for 24 hours, after this period they were placed inside of individual vials containing 5 mL of one of the solutions: distilled water, Coke soft drink (Coca-Cola, Atlanta, Georgia, United States) or beer (Heineken Lager Beer, Amsterdam, Netherlands) for 28 days with the solution renewed every 2 days. The color was evaluated with a spectrophotometer (Easyshade Advance, VITA Zanhnfabrik Bäd Sackingen, Baden-Württemberg, Germany) on a flat matte white standardized acrylic background, under standardized illumination. The color variation (ΔE_{00}) was calculated according to the following formula:

$$
\Delta E_{00} = \left[\left(\frac{\Delta L^{\prime}}{K_L S_L} \right)^2 + \left(\frac{\Delta C^{\prime}}{K_C S_C} \right)^2 + \left(\frac{\Delta H^{\prime}}{K_H S_H} \right)^2 + RT \left(\frac{\Delta C^{\prime}}{K_C S_C} \right) \left(\frac{\Delta H^{\prime}}{K_H S_H} \right) \right]^{1/2} (2)
$$

In which $\Delta L'$, $\Delta C'$ and $\Delta H'$ represent luminosity, chroma and hue respectively, RT refers to the interaction between chroma and hue in the blue region, SL, SC and SH are weighting functions to adjust total color difference in L*, a* and b* coordinates and KL, KC and KH are correction terms for experimental conditions.

SO data were analyzed by Two-way analysis of variance, while the ΔE_{00} data were evaluated through three-way ANOVA, both were submitted to Tukey's HSD test, adopting a significance level of 5%.

RESULTS

Mean values, standard deviations and statistical differences among groups analyzed for SO are shown in Table I. For SO there were significant differences between resin composites ($p = 0.000$) and radiant exposures $(p = 0.022)$. The interaction effect was not significant ($p = 0.984$). Charisma Diamond with the radiant exposure of 1000 mW/cm² showed the lowest SO values. In general, the experimental groups that used the resin composite Charisma Diamond showed the lowest water sorption values, and when considering all the resin composites evaluated, the groups that received radiant exposure of 20 J/cm² with 1000 mW/cm², for 20 seconds showed the lowest water sorption values.

For ΔE_{00} there were significant differences between resin composites ($p = 0.000$), radiant exposures ($p = 0.000$), and immersion solutions $(p = 0.000)$. All interaction effects were found to be significant ($p < 0.05$). Mean values, standard deviations and statistical differences are shown in Table II and Figure 1. The lowest

 ΔE_{00} values were observed by the experimental groups containing the resin composite Charisma Classic that received the radiant exposure of 16.8 J/cm² with 1400 mW/cm², for 12 seconds after immersion in beer, followed by the same material polymerized using the radiant exposure of 20 J/cm² with 1000 mW/cm², for 20 seconds after immersion in the same solution. Further analysis shows that when all resin composites and immersion solutions are considered, groups that received the radiant exposure of 16.8 J/cm^2 , with 1400 mW/cm² , for 12 seconds showed greater color stability with lower ΔE_{00} values. When all resin composites and radiant exposures are considered the immersion solution that was least pigmenting was distilled water, followed by beer and Coke soft drink.

DISCUSSION

The null hypothesis that there would be no difference in the SO and color stability values in the different resin composite types as a function of radiant exposures was rejected because significant differences were found in SO and color stability tests.

However, the findings of the current study do not support the previous research where different power densities of LED light did not affect the degree of conversion of composite resins and the microleakage values of restorations in small Class II cavities [25]. However, both pieces of evidence demonstrate the relevance of better understanding the behavior of light-cured materials in extreme environments such as the mouth.

The degree of conversion is a very important feature of resin-based materials,

Table I - Mean values, standard deviations, and statistical differences of water sorption (in µg/mm³). Different superscript letters for each column means statistically significant differences (p < 0.05)

Resin Composite	Radiant Exposure	Water sorption $(\mu g/mm^3)$
Charisma Classic	1000 mW/cm ²	$14.8(1.9)$ ^{ab}
	1400 mW/cm ²	$17.1 (4.2)^{b}$
Charisma Diamond	1000 mW/cm ²	11.1 (2.9) ^a
	1400 mW/cm ²	$12.5(4.4)$ ^{ab}
Filtek Bulk Fill	1000 mW/cm ²	$15.6(4.1)$ ^{ab}
	1400 mW/cm ²	$17.8(4.4)^{b}$
Filtek Z350 XT	1000 mW/cm ²	$15.6(2.5)$ ^{ab}
	1400 mW/cm ²	$17.6(5.0)^{b}$

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Table II - Mean values and standard deviations of ∆E₀₀. Different superscript letters mean statistically significant differences (p < 0.05)

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as it is significantly correlated with several other important characteristics of the material, such as mechanical properties, polymerization shrinkage stress, wear resistance and elution of monomers [8,13,14,20]. Therefore, the degree of conversion of the resin-based materials can vary from 40% to 75% depending mainly on the material and the photopolymerization conditions and is often measured to evaluate the efficiency of the polymerization and the most common method is by spectroscopy techniques that infer the amount of remaining double bonds, such as the Fourier transform infrared spectroscopy (FTIR) method [26,27].

However, it is known that the greater the number of unreacted monomers in the polymer matrix, the greater the solubility of the resin composite [19].

In the present study, the radiant exposures evaluated are the ones recommended by the LED manufacturer for curing the final layer of the resin composite restorations. The light-activation with different radiant exposures, however, is not in agreement with the concept of reciprocity, or the Exposure Law of Reciprocity was not followed. According to this idea, the same radiant energy (in Joules) reaching the surface of a resin composite $(i.e.,$ radiant exposure measured in $J/cm²$) would have the same effect on the material's properties despite the irradiance (in mW/cm²) and exposure time (in seconds) combinations. It has been suggested that such an equivalence is materialand property-dependent [22].

The large variations in the sorption values of the different materials can be explained by the difference in the composition of each material. In general, when the weight percentage of the filler particles increases, the polymer matrix decreases with the consequent decrease of water sorption [6,8-10] and different polymers have different tendencies to absorb different amounts of water, based on their microstructure and molecular aspects [28].

The water sorption behavior of resin composites is primarily determined by the hydrophilicity of the monomers in their formulation. This hydrophilicity is influenced by the presence of hydrophilic functional groups such as hydroxyl, ether, or amine groups—within the monomer's chemical structure. Monomers like triethylene glycol dimethacrylate (TEGDMA) have a higher tendency to absorb water due to their

polar nature and ability to form hydrogen bonds with water molecules. In contrast, monomers like bisphenol A-glycidyl methacrylate (Bis-GMA) exhibit lower water uptake because their less polar aromatic structures reduce water affinity [8,15,20] It should be noted that the ISO 4049 standard which establishes methods and criteria for evaluating this property was not strictly followed in this study either in terms of the size of the specimen to be evaluated or the period of accommodation in the dissector at room temperature, to reduce the amount of material required and to lower the initial barriers to starting the project. While the standard provides a useful framework for generating widely accepted evidence, the methodology was adjusted to better align with the specific objectives and practical considerations of our study. Although this approach may introduce some differences compared to standardized results, it is expected to still provide valuable insights relevant to our research goals. Resins primarily composed of hydrophobic monomers can still show increased water sorption if they include more hydrophilic monomers or additives designed to improve handling and flow characteristics [8-10]. Studies comparing bulk-fill composites with conventional composites indicate that bulk-fill resins typically have similar or slightly higher water sorption, depending on their specific formulations and curing protocols. This aligns with our findings, which showed no statistically significant differences in water sorption between bulk-fill resins and other composite resins evaluated [7].

Even if a material's composition ensures good mechanical properties, incorrect polymerization can lead to material failure. It is important to consider that the emission and absorption properties of photoinitiators can vary significantly [7], and the mechanical properties of resin materials are directly correlated with the types of photoinitiators used and the power density of light activation [12,29] In this study, we standardized the light exposure times based on the manufacturer's recommendations for the broadband LED device rather than using the curing times suggested by each material's manufacturer. This approach allowed us to compare the performance of different materials under consistent light exposure conditions, as the recommended curing times for various materials can differ widely. By following the LED device's specifications, we aimed to provide a uniform

baseline for evaluating material responses under similar photoactivation conditions.

This study evaluated the color stability of dental composites, which is influenced by water absorption and other physical-mechanical properties. Composites with lower water absorption are generally more resistant to discoloration because water can cause the leaching of unreacted monomers, pigments, and other components, leading to changes in optical properties [4,5,15,18]. The study observed varying degrees of color change among different materials after immersion in distilled water, carbonated soft drinks, and beer, highlighting the effects of humidity, acidity, and pigmented beverages on dental aesthetics. These findings are consistent with previous studies that have identified fluid absorption and matrix degradation as key factors contributing to color change in dental composites [12,23]. This is further demonstrated by the significant color alterations observed in Filtek Z350 XT after 28 days of exposure to staining solutions [20].

A spectrophotometer was used to measure color across different evaluation periods, selected for its precision and versatility in accommodating a wide range of experimental designs in research on resin composites for dental applications. The data obtained from spectrophotometry can be effectively translated into clinically relevant information and can detect color variations that are imperceptible to the human eye [29,30].

In addition to evaluating color changes, the study also assessed perceptibility and acceptability thresholds, which consider non-uniformities in human color perception. Although there is some disagreement in the literature regarding the perceptibility threshold for acceptable color change, this study referenced the thresholds of ΔE_{on} for perceptibility at 0.8 and acceptability at 1.8 [31] of the 24 experimental groups evaluated, 21 exhibited perceptible color changes, and 13 showed color changes that were both perceptible and unacceptable after 28 days of immersion in staining solutions. These findings emphasize the need for materials with greater resistance to discoloration and a deeper understanding of how photoactivation protocols can influence the physical and chemical properties critical to their clinical application. In conclusion, the polymerization of dental composite resins is a crucial factor in the success

and longevity of dental restorations. This process is influenced by several variables, including lightcuring protocols and, specifically, the radiant exposure [21,22,26,32].

High irradiance with shorter exposure times accelerates the polymerization process, resulting in a rapid increase in cross-link formation. However, this rapid process can limit the time available for the material to relax, leading to shrinkage stresses [33,34]. Consequently, microvoids and internal stresses may form, creating structural weaknesses within the composite. These defects can serve as pathways for water ingress, increasing water sorption and potentially causing discoloration over time [35,36].

These findings suggest that the curing protocol directly affects the kinetics of the polymerization reaction, potentially leading to incomplete monomer conversion, the formation of suboptimal cross-links, and the development of internal defects within the composite structure. The impact of polymerization and degree of conversion on water sorption is evident in the results of this study, where increased radiant exposure combined with reduced curing times elevated water sorption levels, making the material more susceptible to hydrolytic degradation. From a practical and clinical perspective, protocols that excessively increase radiant exposure while reducing curing times, although efficient in appearance, may compromise the structural and physical properties of composite resin.

For future studies, we recommend not only determining the degree of water sorption but also directly measuring the degree of conversion. Studies that consider multiple causal variables provide significant insights into the factors that affect the clinical performance and longevity of dental materials.

CONCLUSION

It can be concluded that both the type of resin composite and the radiant exposure significantly influence water sorption (SO) and color stability (ΔE_{00}) of the materials tested. The light-curing protocol of 16.8 J/cm2 at 1400 mW/cm2 for 12 seconds provided the best color stability, while 20 J/cm² at 1000 mW/cm² for 20 seconds was most effective in reducing water sorption. Therefore, the optimal light-curing protocol depends on

whether the primary goal is minimizing color change or water sorption. The combination of the appropriate resin composite and radiant exposure is most effective in minimizing water sorption and color change. The immersion solution also significantly impacted color stability, with distilled water causing the least discoloration, followed by beer and Coke soft drink.

Author's Contributions

PHM, LCO, AYF: Conceptualization, Methodology. PHM, AFS, FAPR, AYF: Writing – Review & Editing, Formal analysis. PHM, AFS, LCO, AYF: Data Curation. PHM, AYF: Investigation, Funding Acquisition, Project Administration. LCO: Resources. AFS: Writing – Original Draft Preparation, Investigation, Supervision.

Conflict of Interest

The authors have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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Regulatory Statement

Not applicable.

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