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GÉSSICA DANDARA MEDEIROS DE SOUZA

THE USE OF ALTERNATIVE PHOTOINITIATORS AND POLYWAVE LED TO IMPROVE THE DEGREE OF CONVERSION AND YELLOWING EFFECT OF EXPERIMENTAL ADHESIVE SYSTEMS

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Géssica Dandara Medeiros de Souza

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Trabalho de Conclusão de Curso apresentado à disciplina de Trabalho de Conclusão de Curso II do Curso de Odontologia da Universidade Federal do Rio Grande do Norte como parte dos requisitos para obtenção do título de Cirurgião-Dentista.

Orientador**:** Prof. Dr. Boniek Castillo Dutra Borges

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DEDICATÓRIA

"Tudo o que um sonho precisa para ser realizado é alguém que acredite que ele possa ser realizado." Assim dedico esse trabalho de conclusão de curso àqueles que mais acreditaram nos meus sonhos, minha família, a vocês essa conquista.

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“O futuro pertence àqueles que acreditam na beleza dos seus sonhos.”

Eleanor Roosevelt

ABSTRACT

Objetives: The aim of this study was to evaluate the degree of conversion (DC) and yellowing effect of five experimental simplified adhesive systems formulated with camphorquinone (CQ), phenyl-propanedione (PPD), and bis-alkyl phosphine oxide (BAPO) using mono- or polywave light emitting diodes (LEDs).

Methods: A model simplified adhesive system was formulated by mixing monomers (60 wt% BisGMA and 40 wt% HEMA) to 30 wt% ethanol. Five materials were formulated by inclusion of the photoinitiators CQ, CQ/PPD, CQ/BAPO, PPD, and BAPO (1 mol% or 0.5/0.5 mol%) (1 mol%). DC was measured using Fourier transformed infrared spectroscopy. The yellowing effect (*b values) was determined after 30-day water storage with a spectrophotometer. ANOVA and Tukey post-hoc test were used to analyze the data ($p<0.05$).

Results: Except for CQ, higher DC was found for other systems adhesives photoactivated by the polywave LED ($p<0.05$). At 30 days water storage, the photoactivation with the polywave LED provided less yellow color (lower b* values) to all adhesive systems ($p<0.05$).

Conclusion: The use of alternative photoinitiators and polywave LED improved the degree of conversion and decreased yellowing effect of experimental adhesive systems.

Clinical significance: Adhesive system formulated with PPD associated to CQ may have improved clinical performance as regard to esthetic appearance.

Key-words: Photoinitiator; Light curing-unit; Degree of Conversion; Adhesives; Color stability.

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SUMÁRIO

1. Introduction

A satisfactory aesthetic appearance the short and long term is influenced directly by characteristics and properties of materials used. Polymers materials are known to be susceptible to various degrees of discoloration on exposure to oral environment [1, 2]. This alteration has been attributed to the structural changes in the material due to aging, the formation of colored degradation products, the change in the surface morphology and the extrinsic staining [3,4]. The natural degradation of chemical additives presents in composite formulation, such as ultraviolet filters, initiators, co-initiators and inhibitors [5] is especially important due the yellowing effect caused by some. As the composite resin, the adhesive systems also have intrinsic color change, which can, in turn, influence the final esthetic outcome of the composite resin restorations [6].

The most contemporary adhesive systems are light activated by light within the blue band of the spectrum (400-500 nm), using generally the camphorquinone (CQ) as photoinitiator [7]. The CQ is a solid yellow compound with an unbleachable chromophore group, compromising its aesthetic performance, since photoinitiators degrade over the course of time [7, 8]. CQ can absorb light in the spectral range of approximately 400-500 nm, and has a peak near 470 nm [9-10].

This characteristic yellow hue of CQ has led to researches testing differents types of photoinitiators, such as PPD (Phenyl Propanodione) and BAPO (Bis-alkyl Phosphine Oxide), with intention of replacing CQ or acting in conjunction with it, diminishing its concentration in the monomers matrix ever further [9,11]. In consequence, reduce the yellowing effect.

However, the most alternative photoinitiators, differently of CQ, have an absorption peak in the ultraviolet region and extends slightly into the visible light spectrum (380–420 nm) [7, 12]. The spectrum emitted by the light source and the capacity of absorption by the photoinitiator have an effect on the polymerization process of composites influencing their properties [7, 12, 13]. Thus, the cure efficiency is compromised when using narrowband light-emitting diodes, which do not have light emission in this violet wavelength range [7, 12, 14].

The conventional light emitting diodes (LEDs) have an emission band in the visible region, so they limited in terms of activating photoinitiators that respond to ultraviolet light. Therefore, the polywave LEDs emit dual peaks, an additional light near approximately 405 nm [15, 16], presenting the ability of activating photoinitiators, such as PPD and BAPO.

The aim of this study was to evaluate the degree of conversion (DC) and yellowing effect of five experimental simplified adhesive systems formulated with camphorquinone (CQ), phenyl-propanedione (PPD), and bis-alkyl phosphine oxide (BAPO) using mono- or polywave light emitting diodes LEDs. The following research hypothesis was tested: the adhesives systems formulated with photoinitiators alternatives when photoactivated with a polywave LED would show increased DC and decreased yellowing effect.

2. Materials and methods

2.1. Experimental design

The response variables of this *in vitro* study were degree of conversion (DC) and yellowing effect. For DC, a 5 x 2 factorial design was designed since photoinitiators at five levels CQ, CQ/PPD, CQ/BAPO, PPD, and BAPO (Table 1) and type of LED at two levels (Table 2) were tested. For yellowing effect, a 5 x 2 x 2 was designed since photoinitiators at five levels CQ, CQ/PPD, CQ/BAPO, PPD, and BAPO (Table 1), type of LED at two levels (Table 2), and evaluation periods (baseline and 30-day water storage) were tested.

2.2. Formulation of the experimental simplified adhesive systems

A model simplified adhesive system was formulated through the intensive mixture of bisphenol A glycidyl dimethacrylate (BisGMA) and hydroxyethyl methacrylate (HEMA) (Sigma–Aldrich Inc., St. Louis, MO, USA) (60:40 wt%) and ethanol (30 wt%) [19]. Five different photoinitiator systems were added at 1 mol% (CQ, PPD and BAPO) or 0.5/0.5 mol% (CQ/PPD and CQ/BAPO). Ethyl 4-(dimethylamino) benzoate (EDMAB) (Sigma–Aldrich Inc., St. Louis, MO, USA) (1 mol%) was added to all formulations as co-initiator.

2.3. DC evaluation

The evaluation of DC was made by Fourier transform infrared/attenuated total reflectance (Spectrum 100, PerkinElmer, Shelton, CT, USA) at 24ºC under 64% relative humidity. One hundred samples (5 mm in diameter x 1 mm thickness) were fabricated (n=10). One drop of each model adhesive system was inserted into a silicon mold, the solvent was evaporated for 10 s, a thin glass plate (1 mm thickness) was placed on the material and it was photoactivated for 10 s using each LED. The absorption spectra of nonpolymerized and polymerized model adhesive system were obtained from the region between 4000 and 650 cm^{-1} with 32 scans at 4 cm^{-1}. The aliphatic carbon-to-carbon double-bond absorbance peak intensity (located at 1638 cm^{-1}) and that of the aromatic component (located at 1608 cm^{-1}; reference peak) were obtained. The DC (%) was calculated using the following equation:

$$DC\ (\%) = 100\ x \left[1 - \left(\frac{R\ polymerized}{R\ nonpolymerized}\right)\right],$$

where R represents the ratio between the absorbance peak at 1638 cm^{-1} and 1608 cm^{-1}.

*2.4. Yellowing effect (*b values)*

After the DC analysis, the samples were placed in distilled water at 37° C in total darkness 24 hours. After this period, the initial color values (Ti) were measured directly on the photoactivated surface through a spectrophotometer (Easyshade, VITA, Zahnfabrik, Bad Säckingen, Germany) [20]. The specimens were dried with absorbent paper, and kept on a standard white background. The observation pattern simulated for color values followed the CIE L*a*b* system (Comission Internationale de I'Eclairage). This system consists of two axes, a* and b*, that have right angles and represent the dimension of tonality or color. The third axis is luminosity L*. This is perpendicular to the plane a* b*. For the purpose of this study, only the values of *b were measured.

After 30 days of water storage (Tf), the color values were obtained again. Five measurements were repeated on each specimen and surface, at the same assessment time and the average value was calculated. To avoid any interference of external light, a silicon barrier fabricated around the interface between the specimen and the spectrophotometer tip. Further, the silicon barrier allowed that the tip was placed on the same specimen area in both color measurements. Yellowing effect was established by comparing b* value before and after 30 days of water storage [21].

2.5. Statistical analysis

For DC evaluation, two-way ANOVA and Tukey post-hoc test were used ($p<0.05$). For b* color evaluation, three-way ANOVA with repeated measurements and Tukey post-hoc test were used ($p<0.05$).

3 Results

3.1. DC

There were statistically significant differences in the interaction photoinitiator system x curing light ($p<0.01$). Table 3 shows the intergroup comparisons. Only the CQ adhesive system showed similar DC between samples photoactivated by Radii Cal and Bluephase G2. Bluephase G2 provided higher DC means than Radii Cal to other adhesive systems. Bluephase G2 provided similar DC means to all adhesive systems. Radii Cal provided the highest DC to CQ adhesive system and lowest DC to CQ/BAPO- and BAPO adhesive systems.

3.2. Yellowing effect (b value)*

There were statistically significant differences in the interaction storage time in water x photoinitiator system x curing light ($p<0.01$). Table 4 shows the intergroup comparisons. At 24 h water storage, only CQ/BAPO adhesive system showed similar yellowing (b* values) between samples photoactivated by Radii Cal and Bluephase G2. Bluephase G2 provided less yellow color (lower b* values) than Radii Cal to CQ, CQ/PPD, PPD, and BAPO adhesive systems. At 30 days water storage, Bluephase G2 provided less yellow color (lower b* values) to all adhesive systems. Radii Cal provided and Bluephase G2 provided the highest yellowing (highest b* value) to the CQ adhesive system, and the lowest yellowing (lowest b* value) to the CQ/PPD adhesive system. Radii Cal provided no yellowing (similar b* values) to CQ, CQ/PPD, and CQ/BAPO adhesive system and increased yellowing (higher b* values) to PPD and BAPO adhesive system after 30 days water storage. On the other hand, except to the CQ adhesive system, Bluephase G2 provided less yellowing (lower b* values) to other adhesive system after 30 days water storage.

4. Discussion

In the present study, was evaluated the use of alternative photoinitiators and polywave led to improve the degree of conversion and yellowing effect of experimental adhesive systems. Based on the results obtained, it could be affirmed that the tested hypothesis was acepted, the systems adhesives formulated with photoinitiators alternatives when photoactivated with LED lights of third generation show an increase in DC and a smaller yellowing effect.

The DC is a very important parameter used to measure photopolymerization performance, is influenced for the photopolymerization activity of photoinitiators and the wavelength and intensity of irradiations lamps [22]. The table 1 shows the results associated a DC.

Only the systems adhesives formulated with CQ showed similar DC between samples photoactivated by Radii Cal and Bluephase G2. Convencional LED lights, such as Radii Cal, have an emission band in the visible region, so that emitted one-peak in a narrow spectral band [18]. Unlike convencional LEDs, Bluephase G2 is a dual peak LED, providing additional light with nearly 405 nm [15, 16]. The CQ is light activated by lights within the visible light spectrum and has a peak of absorbance near 470 nm [9-10]. Thus, the similar DC observed in the CQ systems adhesives is due the two types of LED lights used presents an emission of light within the light band ideal for the photoactivation of CQ, the visible light spectrum.

Unlike CQ, the systems adhesives formulated with CQ/PPD, CQ/BAPO, PPD and BAPO exhibited higher DC when photoactivated by Bluephase G2 than Radii Cal. Photoinitiators alternatives such as PPD and BAPO have an absorption peak in the ultraviolet region (100 - 400 nm) [23], their peaks of absorbance are 398 and 370 nm, respectively [7]. Thus, the generation of free radicals from these compounds was more efficient when using a dual-peak LED, due the absorption spectra that overlap with the emission spectra of irradiance source.

Although its absorption peak is in the infrared light range, the PPD has the greatest DC that systems adhesives containing BAPO when photoactivated with Radii Cal. This fact is possible related to the ability of the PPD additionally absorbs light in the visible spectrum [18]. The activation of the other photoinitiators with single-peak LEDs is not possible, because this light source does not emit UV light [18].

The yellowing effect in the systems adhesives also was evaluated. After 24 hours of storage in water, only CQ/BAPO adhesive system showed similar yellowing between samples photoactivated by Radii Cal and Bluephase G2. The reason for this fact is the similar DC propitiate for two LEDs to the CQ adhesive. Both the LEDs emit light in wavelength capable to excit only CQ, while the other photoinitiators are not excited completely by the Radii, because this not emit light in infrared spectral. In contrast, Bluephase G2 promoted lower yellowing to CQ, CQ/PPD, PPD, and BAPO adhesive systems. The emission of dual-peak light of Bluephase enable higher DC and, consenquently, lower yellowing.

After 30 days of storage in water, Bluephase G2 promoted lower yellowing for all systems adhesives. The Bluephase G2 being a polywave LED, it can emit light with two peaks in different ranges (UV and blue light), thus allowing different photoinitiators are able to absorb the light emitted by it and carry out an effective polymerization. Therefore, if the DC of systems adhesives were higher, the yellowing will be lower, to due the influence of DC over the yellowing. The degree of conversion is directly related to their staining capacity and chemical characteristics [24]. A low polymerization coefficient results in low mechanical strength and degradation of the unreacted molecules over the course of time [25, 26].

The CQ adhesive system showed higher yellowing for the samples photoactivated by the two types of LEDs. CQ presents characteristics inherent to its chemical composition that contribute to it to produce a higher level of yellowing compared to other photoinitiators. This typical color of CQ is due to its chomophore group, in which a set of atoms that form molecules, this when excited by light, reflect spectra in a tonality that ranges from yellow to orange [25].

Corroborating this fact, unlike others photoinitiators, such a PPD, the CQ has a low capacity to perform photobleaching, which is the ability to break the chromophoric groups, shortly after sensitization by light, thus contributing to the reduction of yellowing [25]. These factors associated meant that even when photoactivated with different LEDs lights, the CQ present the biggest b * values.

On the other hand, the CQ/PPD system adhesive showed the lower yellowing, for the two LED lights used. Studies have demonstrated that the use of PPD increases the efficiency of polymerization of monomers, and reduce the yellowing effect of CQ [25, 26]. The CQ can absorb light in a spectral range of approximately 400-500 nm, peaking at 470 nm absorption [9, 10]. Differently, the PPD have their peak absorption in the ultraviolet light range, which corresponds to approximately 398 nm [7]. The Bluephase G2 can properly excite this adhesive system for issuing peaks in the blue and ultraviolet range.

The performance of CQ in association with PPD system photoactivated for the Radii Cal can be explained by the fact PPD achieve an additional absorption of light in the visible range, unlike other photoinitiators [18], contributing a greater degree of conversion. It is known that an insufficient monomer conversion, and the presence of carbon with double bonds make the material more susceptible to degradation reactions which lead to instability in color, so that the more the degree of conversion, the lower the probability of yellowing.

The adhesive systems that had CQ in its composition alone or in combination not have an increase in yellowing after 30 days when photoactivated by Radii Cal. This is due Radii Cal not emit light at the wavelength of maximum absorption of BAPO and PPD, so that only the CQ can be turned on appropriately. In contrast, the materials that had PPD and BAPO isolated had more yellow color after 30 days of water storage. These photoinitiators which has its peak light absorption in the ultraviolet region. Thus, when a monowave LED such as the Radii Cal was used to photoactivate BAPO and PPD systems, the degree of conversion is compromised.

With the exception of CQ, all other systems adhesives showed after 30 days, a lower yellowing when photoactivated by Bluephase G2. The emitting light in blue and ultraviolet range by Bluephase provided a photoactivation adequate for all photoinitiators used. This result shows that PPD and BAPO, when photoactivated by a light source adequate, presents lower yellowing a long term, in comparison the CQ. The higher yellowing of CQ a long term is associated the formation of chomophores group and inability to perform photobleaching [25].

5. Conclusion

The use of alternative photoinitiators and polywave LED improved the degree of conversion and decreased yellowing effect of experimental adhesive systems. The association between PPD and CQ provided the lowest yellowing effect to the adhesive system.

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Tables

Table 1 - Characteristics of photoinitiators used in this study.

Name	Absorption spectrum range (nm)	Absorption intensity peak (nm) [7]	Molar extinction coeficiente (L/mol cm) [7]
CQ*	400 – 500 [7]	470	28±2
PPD**	350 – 480 [17]	398	150±10
BAPO***	365 – 416 [7]	370	300±10

*Camphorquinone; **Phenyl-Propanedione; ***Bis-Alkyl Phosphine Oxide

Table 2 - Technical details of Light Emiting Diodes used in this study.

Comercial name/ Manufacturer	Classification	Spectrum range	Intensity peaks	Irradiance
Radii Cal, SDI, Victoria, Australia.	Mowave	440 - 480 nm	460 nm	1200 mW/cm^2
Bluephase G2, Ivoclar Vivadent, Schaan, Liechtenstein	Polywave	385 – 515 nm (380 – 420 + 420 – 490)	405 nm 460 nm [18]	1200mW/cm^2

Table 3 - Degree of conversion (%) means (standard-deviation) of experimental adhesive systems according to the photoinitiator system and the curing light.

	Curing light	
Photoinitiator system	Radii Cal	Bluephase G2
CQ	77.8 (6.8) aA	77.3 (14.1) aA
CQ/PPD	48.8 (7.4) bB	71.6 (7.7) aA
CQ/BAPO	31.5 (11.5) cB	74.1 (6.9) aA
PPD	47.2 (4.0) bB	74.2 (6.4) aA
BAPO	27.6 (3.9) cB	81.6 (6.5) aA

Means followed by different capital letters indicate statistically significant differences between curing lights for the same photoinitiator ($p<0.05$). Means followed by different lower case letters indicate statistically significant differences among photoinitiator systems for the same curing light ($p<0.05$).

Table 4 - b* value means (standard-deviation) of experimental adhesive systems according to the photoinitiator system, curing light and storage time in water.

		Curing ligth	
Storage time in water	Photoinitiator system	Radii Cal	Bluephase G2
24 h	CQ	57.8 (3.9) aA^{ns}	43.3 (4.4) aB^{ns}
	CQ/PPD	25.7 (0.6) dA^{ns}	17.3 (0.8) cB*
	CQ/BAPO	49.7 (3.4) bA^{ns}	44.3 (3.0) aA*
	PPD	33.3 (1.2) cA*	28.9 (0.1) bB*
	BAPO	51.3 (4.2) bA*	34.8 (3.3) bB*
30 days	CQ	60.9 (1.0) aA	40.0 (1.9) aB
	CQ/PPD	26.0 (2.1) dA	16.2 (1.2) dB
	CQ/BAPO	51.8 (0.6) bA	34.6 (3.0) bB
	PPD	36.5 (0.2) cA	20.8 (3.6) cB
	BAPO	55.2 (4.0) bA	28.9 (3.2) bB

Means followed by different capital letters indicate statistically significant differences between curing lights for the same photoinitiator ($p<0.05$). Means followed by different lower case letters indicate statistically significant differences among photoinitiator systems for the same curing light ($p<0.05$). ^{ns}No statistically significant differences between different storage time in water for the same photoinitiator

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Abstract: should not exceed 250 words and should be presented under the following subheadings: Objectives, Methods; Results; Conclusions (For Reviews: Objectives; Data; Sources; Study selection; Conclusions). A 50 word 'Clinical Significance' statement should appear at the end of the abstract advising readers of the clinical importance and relevance of their work. These subheadings should appear in the text of the abstract. Please repeat the title of the article at the top of the abstract page.

Introduction: must be presented in a structured format, covering the following subjects, although not under subheadings: succinct statements of the issue in question, and the essence of existing knowledge and understanding pertinent to the issue. In keeping with the house style of Journal of Dentistry, the final paragraph of the introduction should clearly state the aims and/or objective of the work being reported. Prospective authors may find the following form of words to be helpful: "The aim of this paper is to ..." Where appropriate, a hypothesis (e.g. null or a priori) should then be stated.

Keywords: up to 6 keywords should be supplied.

Abbreviations and acronyms: terms and names to be referred to in the form of abbreviations or acronyms must be given in full when first mentioned.

Units: SI units should be used throughout. If non-SI units must be quoted, the SI equivalent must immediately follow in parentheses. The complete names of individual teeth must be given in the test. In tables and legends for illustrations individual teeth should be identified using the FDI two-digit system.

Statistics: Statistical methods should be described with enough detail to enable a knowledgeable reader with access to the original data to verify the reported results. When possible, findings should be quantified and appropriate measures of error or uncertainty (such as confidence intervals) given. Details about eligibility criteria for subjects, randomization and the number of observations should be included. The computer software and the statistical method(s) used should be specified with references to standard works when possible (with pages specified). See http://www.icmje.org/manuscript_1prepare.html for more detailed guidelines.

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Lynch CD, Frazier KB, McConnell RJ, Blum IR, Wilson NHF. State-of-the-art techniques in Operative Dentistry: contemporary teaching of posterior composites in UK and Irish dental schools. British Dental Journal 2010; 209: 129 - 36.

Wilson NHF, Mjör I. The teaching of class I and class II direct composite restorations in European dental schools. Journal of Dentistry 2000; 28: 15-21.

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Books

Lynch CD. Successful posterior composites. London: Quintessence Publishing Co., 2008.

Book chapters

Phillips SJ, Whisnant JP. The role of dentine under restorations. In: Laragh JH, Brenner BM, editors. The science of restorative dentistry. 2nd ed. Oxford: Elsevier; 2003. p.266-78.

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