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Incorporation of a polymerization catalyst for improved mechanical behavior and physicochemical characteristics of a light-cured pulp capping material

Incorporação de um catalisador de polimerização para aprimorar propriedades mecânicas e fisíco-químicas de um protetor pulpar fotoativável

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ABSTRACT

Objective: To evaluate the influence of the incorporation of a polymerization catalyst to a light-cured pulp capping material on mechanical behavior and physicochemical characteristics. **Material and Methods:** Different percentages (2 wt%, and 4 wt%) of diphenyliodonium hexafluorophosphate (DPI) were incorporated into the Ultra-Blend Plus, a resin-modified calcium-based cement. The material without incorporation of DPI (0 wt%) served as control. Degree of Conversion (DC), Flexural Strength (FS), Elastic Modulus (EM), Water Sorption (WSp), Solubility (Sl), and pH of eluate at 24-h, 72-h, and 7-day storage times were measured. One-way ANOVA/Tukey posthoc tests were used to analyze the data ($p < 0.05$). **Results:** For DC, FS, and EM, materials with different % of DPI showed statistically significant differences, so that 0% provided the lowest values and 2% the highest values. Materials with 0% and 2% of DPI provided statistically the lowest WSp, whilst material with 0% of DPI showed statistically the highest Sl. **Conclusion:** All materials provided statistically similar pH to eluates regardless of storage time, although only materials with DPI at 2% and 4% maintained pH of eluates statistically similar from 72 h to 7 days storage times.

KEYWORDS

Light-cured pulp capping material; Polymerization catalyst; Pulp capping.

RESUMO

Objetivo: avaliar a influência nas propriedades mecânicas e físico-químicas da incorporação de um catalisador de polimerização a um protetor pulpar fotoativável. **Material e Métodos:** foram adicionadas diferentes porcentagens em massa (2% e 4%) de hexafluorofosfato de difeniliodônio (DPI) ao Ultra-Blend Plus, um cimento à base de hidróxido de cálcio modificado por resina. O material sem a adição do DPI (0%) serviu como controle. Foram avaliados: Grau de Conversão (DC), Resistência Flexural (FS), Módulo de Elasticidade (EM), Sorção (WSp), Solubilidade (SI) e o pH do eluato nos tempos de 24h, 72h e 7 dias de armazenamento. ANOVA 1-way com pós-teste de Tukey ($p < 0.05$). Foi utilizado para avaliar os resultados estatisticamente. **Resultados:** Os materiais com diferentes % de DPI apresentaram diferenças significativas para os testes de DC, FS e EM. A porcentagem em massa de 0% de DPI mostrou valores inferiores a todos os testados e os materiais com adição 2% apresentaram a melhor performance. Materiais com 0% e 2% de DPI apresentaram valores inferiores de WSp; a porcentagem de 0% proporcionou valores estatisticamente maiores para SI. **Conclusão:** Todos os materiais testados apresentaram pH semelhante nos eluatos independente do tempo de armazenamento, contudo, apenas os materiais com 2% e 4% mantiveram o pH dos eluatos estatisticamente similares nos tempos de estocagem de 72h a 7 dias.

PALAVRAS-CHAVE

Catalisador de polimerização; Proteção pulpar; Protetor pulpar fotoativável.

INTRODUCTION

In the treatment of deep caries lesions, maintaining the vitality of the dentin-pulp complex is essential. The pulp requires protection against bacterial invasion, thermoelectric conduction, and chemical protection of the overlying restorative materials. In this way, the alkaline pH of calcium hydroxide and its antimicrobial activity through the release of hydroxyl ions promote enzymatic inhibition of microorganisms and make it an excellent material for indirect and direct pulp capping [1,2]. However, the poor mechanical properties of pulp-capping materials may negatively affect their resistance to fracture during placement of final restorative material or while supporting an overlying restoration over time [3].

Due to the poor handling, weak physical strength, high solubility, and/or gradual resorption of chemically activated calcium hydroxide cements, light-cured resin-modified materials were developed [4]. These materials consist of components associated with traditional pulp therapy (e.g. calcium hydroxide), in addition to methacrylates such as diurethane dimethacrylate (UDMA) and triethyleneglycol dimethacrylate (TEGDMA), together with camphorquinone/amine photoinitiation system and additives that enable light curing [5]. On the other hand, the addition of resin and photoinitiation system implies that adequate polymerization of light-cured calcium hydroxide cements occurs, since the unsatisfactory polymerization of materials causes decreased mechanical strength, greater water sorption/solubility, the formation of cracks between the material and dentin, and increased release of residual monomers that can increase cytotoxicity [4-9]. Since the degree of conversion rate of light-cured calcium hydroxide cements is unsatisfactory [4], efforts should be made to enhance the degree of conversion and, consequently, biological and mechanical behavior.

In an attempt to improve polymerization efficiency and mechanical behavior of methacrylate-based dental materials, the addition of diphenyliodonium hexafluorophosphate salt (DPI) to act as polymerization catalyst of camphorquinone/amine photoinitiator system was suggested [10]. DPI can act as a catalyst to photoinitiation systems, decreasing the activation energy of photoinitiation and bringing advantage in polymerization kinetics [10]. The inclusion of DPI in experimental resin cements [11] and commercially available dual-polymerizing self-adhesive resin cements [12] improved the degree of conversion and mechanical properties such as flexural strength and modulus. However, there are no data regarding the impact of the addition of DPI on the degree of conversion and mechanical behavior of photocurable calcium hydroxide cement.

Accordingly, the present work proposes to evaluate the effect of different concentrations of DPI on the degree of conversion (DC), flexural strength (FS), elastic modulus (EM), water sorption (WSp), solubility (Sl), and pH of eluate (pH) at different storage times of a light-cured pulp capping calcium-based cement. The null hypotheses tested were that: (1) the incorporation of DPI to the material would not influence the DC, FS, EM, WSp, and Wl; and (2) different concentrations of DPI incorporated into the material and storage times would not influence the pH of the eluate.

MATERIALS AND METHODS

Study design

The present study was characterized as a laboratory *in vitro* investigation. The following response variables were included: degree of conversion (DC), flexural strength (FS), elastic modulus (EM), water sorption (WSp), solubility (Sl), and pH of the eluate (pH). For DC, FS, EM, WSp, and WSl, the factor under study was the concentration of DPI (0%, 2%, and 4%). For pH, the concentration of DPI and the storage time (24h, 72h, and 7 days) were the factors under study. Figure 1 shows a schematic representation of the methods.

Materials’ preparation

The DPI salt (Sigma-Aldrich, St. Louis, MO, USA, lot BCBP1499V) was incorporated into a commercially available light-cured pulp capping calcium-based cement Ultra-Blend Plus – dentin shade (Ultradent Products, Inc., South Jordan, UT, USA) (Table I) according to different concentrations: 2 wt%, and 4 wt%. The material without incorporation of DPI (0%) was defined as the control group. After weighing the cement and DPI salt, they were mixed and homogenized using

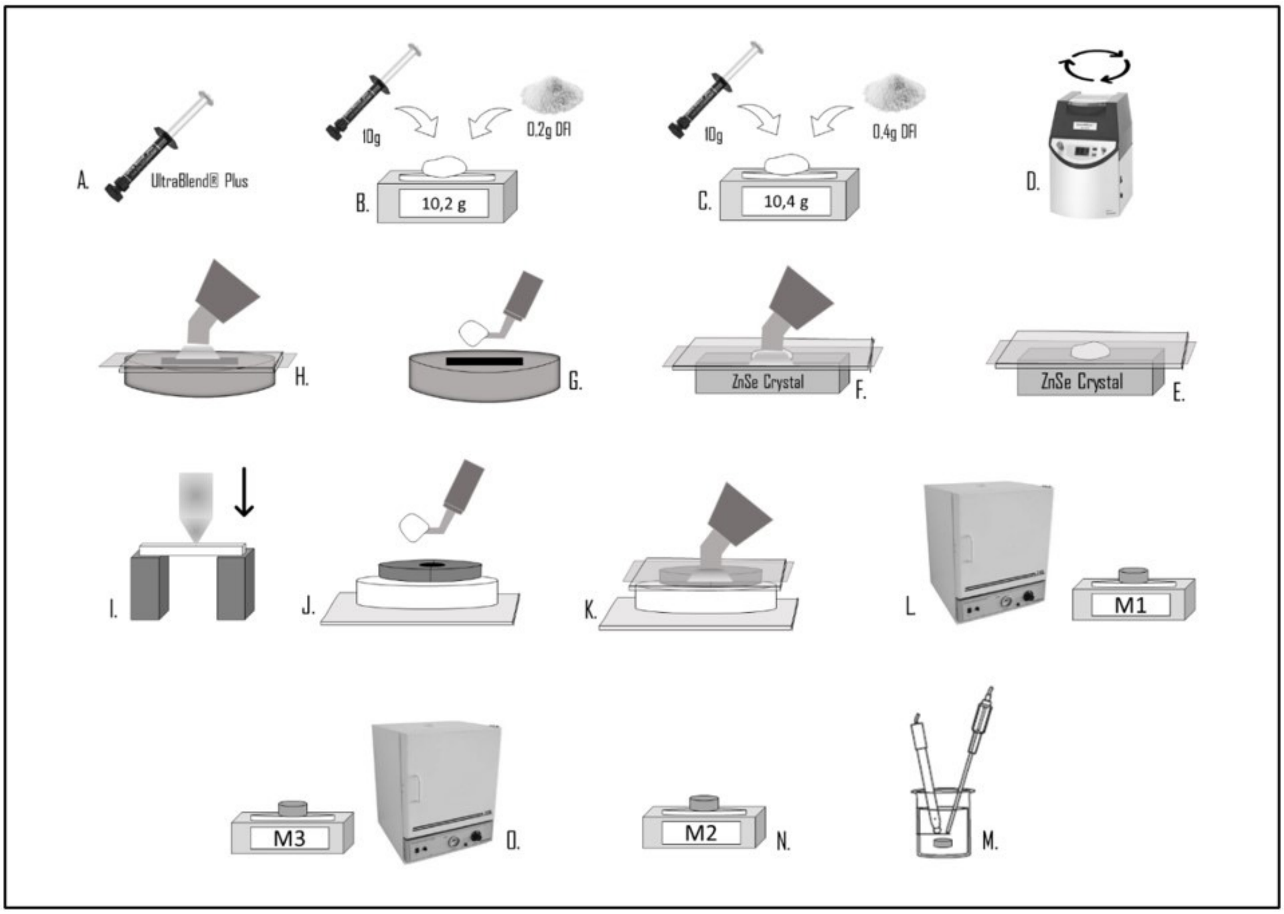

Figure 1 - Schematic representation of the methods: The light-cured pulp capping calcium-based cement Ultrablend® Plus (A) was mixed with 2 wt% (B) and 4 wt% (C) diphenyliodonium hexafluorophosphate (DPI) salt and homogenized using a centrifugal mixing device (D). Material without DPI served as control. Then, a drop of each material was dispensed on the ZnSe crystal of an FTIR/ATR device, and non-polymerized (E) and polymerized spectra after photoactivation (F) were obtained to measure the degree of conversion. Each material was inserted in a bar-shaped mold (G) and photoactivated (H) to produce specimens for flexural strength and modulus analyzes through a three-point bending test (I). Disk-shaped specimens were produced after filling a mold with each material (J) and photoactivation (K). After dehydration and mass stabilization, M1 was obtained (L) and specimens' volume was calculated. Then, they were soaked in distilled water (M) for 7 days, the pH of the eluate was recorded, and M2 obtained (N). After dehydration, and mass stabilization, M3 was obtained (O) to measure water sorption and solubility.

Table I - Chemical composition of the light-cured pulp capping calcium-based cement used in this study

Material	Manufacturer	Chemical Composition	Lot Number
Ultra-Blend® Plus	Ultradent Products Inc., South Jordan, UT, USA	Calcium hydroxide, Barium sulfate, Diurethane dimethacrylate	D059A

Data from the manufacturer.

a centrifugal mixing device (SpeedMixer, DAC 150.1 FVZ-K; Hauschild Engineering, Hamm, Germany) at 3500 rpm for 1 min. The cement homogeneity was checked after mixing and before specimens' preparation for each analysis.

DC analysis

DC was evaluated using Fourier Transform Infrared Spectroscopy (FTIR) coupled with Attenuated Total Reflectance (ATR) (IR Prestige 21, Shimadzu, Kyoto, Japan), as previously described [13]. A droplet of each material (20 μl) was dispensed on the zinc selenide crystal (n = 10) and covered with a Mylar strip. Spectra in the range between 1,800 and 1,500 cm^{-1} of unpolymerized material were obtained with 64 scans at a resolution of 4 cm^{-1}, and mirror dislocation speed of 2.8 mm/s. Then, photoactivation was performed for 10 s with a light-emitting diode (LED) unit (Coltolux LED, Coltène-Whaledent, Altstätten, Switzerland - 1200 mW/cm^2) maintaining the standardized distance of 1 mm between the tip of the curing light and the specimen, and spectra of polymerized material were obtained. The analysis was performed under controlled temperature (23 ± 2° C) and humidity (50 ± 10%) conditions. DC was calculated considering the stretching vibration intensity for carbon double bonds (C=C), in 1,635 cm^{-1} frequency. Carbonyl (1,720 cm^{-1}) was used as an internal standard for polymerized and unpolymerized specimens. To calculate the degree of conversion the formula below was used.

$$\%GC = 1\frac{(1639/1609 \text{ for polymerized resin})}{(1639/1609 \text{ for non-polymerized resin})}\times 100 \quad (1)$$

FS and EM analyses

Forty bar-shaped specimens (10 mm x 2 mm x 1 mm) were produced (*n* = 10 per material), as previously described [13]. The material was inserted into a silicone (Express XT, 3M ESPE, St. Paul, MN, US) mold (width = 2.0 mm, length = 10.0 mm, height = 1.0 mm), covered with a Mylar strip and a glass slide, and photoactivated from the top using two overlapping zones for 10 s (Coltolux LED, Coltène-Whaledent, Altstätten, Switzerland - 1200 mW/cm^2). The specimens were removed from the mold, stored in distilled water at 37° ± 2°C for 24h, and submitted to a three-point bending test in a universal test machine (OM 150, ODEME, Luzerna, SC, Brazil) with 8 mm span between supports at 0.5 mm/min. The FS (MPa) and EM (GPa) were calculated using these Equations 2 and 3:

$$FS = \frac{3l\text{F}}{2wh^2} \quad (2)$$

$$EM = \frac{l^3\text{F}}{4wh^3d} \quad (3)$$

where l is the spam between supports (mm); F is the failure load (N); w and h are the width and the height of the specimen (mm), respectively; and d is the deflexion of the specimen during the elastic regimen.

WSp, Sl and pH analyses

The analyzes were performed according to what was previously described [14]. Forty disc-shaped specimens (5 mm x 1 mm) were produced (*n* = 10 per material). The material was inserted into a silicone mold (diameter = 5 mm, height = 1 mm), covered with a Mylar strip and a glass slide, and photoactivated from the top for 10 s (Coltolux LED, Coltène-Whaledent, Altstätten, Switzerland - 1200 mW/cm^2). The specimens were stored in a glass desiccator for 22 h in an incubator (37 ± 2°C), transferred into another desiccator and maintained at room temperature (23 ± 2°C) for 2 h, thus completing a 24-h cycle. Following each 24-h cycle, specimens were weighed on a 0.01 mg precision analytical balance (Aw220 Analytical Balance - 200g X 0.0001g Mars / Shimadzu) daily. The procedure was repeated until the mass loss was less than 0.1 mg and that weight was recorded as m1 (mg). Two orthogonal measures of diameter and five measures of thickness of each specimen were obtained using a digital caliper (Mitutoyo Co., Kanagawa, Japan) and averaged to calculate the volume (V) in mm^3. The specimens were soaked in deionized water (20 mL) for 7 days (37 ± 2°C), and then taken out, swabbed, waived in the air (15 s), and weighed to obtain mass recorded as m^2 (mg). Then, the specimens were desiccated by the same procedure described above until the constant weight (m3) was obtained. The WSp and Sl were calculated using these Equations 4 and 5:

$$WSp = \frac{\text{m2} - \text{m3}}{\text{V}} \quad (4)$$

$$Sl = \frac{m1 - m3}{\text{V}} \quad (5)$$

Except for specimens' dimensions, WSp and Sl analyses were conducted following the ISO 4049:2009 [15].

The pH was measured in the eluate resulting from the immersion of specimens in deionized water (*n* = 10 per material) for WSp and Sl analyses. The pH values were determined after 24-h, 72-h, and 7-day storages with a digital pHmeter (PHS-3E, Jiangsu Instruments, Jiangsu, China) previously calibrated at room temperature (25 ± 2°C) with standard buffers of pH 6.86 and 4.00.

Statistical analysis

Data from DC, FS, EM, WSp, and Sl were subjected to one-way ANOVA (% of DPI) and Tukey posthoc test ($p<0.05$). pH was analyzed using two-way ANOVA (% of DPI *x* timepoint) and Tukey posthoc test ($p<0.05$). Statistical tests were performed in the GraphPad Software (2365 Northside Dr. Suite 560, San Diego, CA 92108).

RESULTS

DC, FS, EM, WSp and Sl

Results from DC, FS, EM, WSp, and SL are shown in Table II. For DC, FS, and EM, the

Table II - Means ± standard deviations of DC (%), FS (MPa), EM (GPa), WSp ($\mu g/mm^3$), and Sl ($\mu g/mm^3$) according to the % of DPI incorporated into the light-cured pulp capping calcium-based cement

DPI	DC	FS	EM	WSp	Sl
0%	44.79 ± 4.64 c	58.07 ± 5.32 c	25.48 ± 3.25 c	151.35 ± 28.30 b	640.02 ± 37.47 a
2%	56.43 ± 2.09 a	212.46 ± 18.82 a	64.90 ± 6.40 a	126.43 ± 6.10 b	151.35 ± 28.30 b
4%	49.84 ± 3.48 b	188.80 ± 11.15 b	51.75 ± 4.28 b	217.45 ± 13.80 a	126.43 ± 6.10 b

DC: Degree of Conversion; FS: Flexural Strength; EM: Elastic Modulus; WSp: Water Sorption; Sl: Solubility. DPI: Diphenyliodonium Hexafluorophosphate. Different lowercase letters indicate statistically significant differences between the addition of different percentages of diphenyliodonium (p <0.05).

Table III - Means ± standard deviations of pH of the eluate according to the % of DPI incorporated into the light-cured pulp capping calcium-based cement and storage time analyzed

DPI	Storage time		
	24 h	72 h	7 days
0%	7.27 ± 0.07 Ba	7.67 ± 0.12Aa	7.16 ± 0.09 Ba
2%	6.96 ± 0.27 Ba	7.65 ± 0.16 Aa	7.22 ± 0.02 Aa
4%	6.68 ± 0.36 Ba	7.50 ± 0.06 Aa	7.27 ± 0.01 Aa

DPI: Diphenyliodonium Hexafluorophosphate. Different lower-case letters indicate statistically significant differences between the materials for the same evaluation time. Different capital letters indicate statistically significant differences between the same material between different release times.

material without incorporation of DPI presented statistically the lowest mean, whilst material with DPI at 4% showed statistically higher FS than that without incorporation of DPI and lower than that in which DPI was incorporated at 2%.

Concerning WSp, statistically similar means were obtained by materials without incorporation of DPI and with incorporation at 2%, which were statistically lower than that presented by the material with incorporation at 2%.

Materials in which DPI was incorporated at 2% and 4% presented statistically similar Sl means, which were statistically lower than that presented by the material without incorporation of DPI.

pH

There were statistically significant differences between concentrations of DPI (p <0.01) and timepoints (p<0.01). Comparisons among the groups are shown in Table III. Comparisons among materials into the same storage time showed statistically similar pH of eluates at all storage times. Materials with DPI at 2% and 4% provided the highest pH to eluates at 72-h and 7-day storage times, which were statistically similar and higher than at 24-h. On the other hand, the material without DPI (0%) provided a statistically similar pH to the eluates at 24-h and 7-day storage times, which were statistically lower than at 72-h.

DISCUSSION

Since DC, FS, EM, WSp, and Sl and of materials in which DPI was incorporated presented statistically significant differences from that without the inclusion of DPI, the first null hypothesis tested was rejected. The incorporation of DPI at 2% provided the highest DC, FS, and EM to the light-cured calcium hydroxide cement tested.

Although the material data sheet is incomplete regarding all chemical components included in the Ultrablend Plus, it was demonstrated that this material contains a binary photoinitiator system composed by camphorquinone (CQ), a Type II photoinitiator, and the tertiary amine 2-(Dimethylamino) ethyl methacrylate (DMAEMA)(5), which initiates the photopolymerization reaction. The photopolymerization of Type II photoinitiator based materials involves the absorption of visible blue light by the carbonyl group of CQ which lead it to triplet state via excitation into a singlet state. Subsequently, hydrogen abstraction from the co-initiator occurs and a radical is formed. Back electron transfer reverses the excited state of the photoinitiator and restores the donor and acceptor to their original oxidation levels which retards the hydrogen abstraction process and the generation of photoinitiator radicals [16,17]. When DPI was incorporated into the tested material, a ternary photoinitiator system was obtained, leading to remarkable improvements in DC, as well reported previously [10,12,16]. For a ternary system, the rate of radical generation may be faster due to the availability of more excited and the re-activation of inactive CQ molecules, in the way that back electron transfer is reduced(10). Thus, whilst binary CQ systems can generate only up to one active radical per molecule of CQ, ternary CQ systems can generate up to 4 active radicals per molecule of CQ [10,18].

Although it has been suggested that the use of DPI to institute a ternary photoinitiator with CQ and DMAEMA in experimental methacrylate-based cements might increase the polymer crosslinking, the fraction of DPI incorporated has a limit until after no improvement in the photopolymerization is observed [11]. In this sense, the material with 4% DPI presented higher DC than the control (0% DPI), but lower DC than that with 2% DPI. Since DPI is a salt that needs to be completely dissolved into the monomeric blend of the material, it is likely that material with 4% DPI may have not provided and homogeneous bulk in comparison with that containing 2% DPI due to the increased volume of salt. The poor homogeneity of the material in which DPI was incorporated at 4% may also have contributed to increased WSp in comparison with others. On the other hand, since the organic component of the cements with 2% and 4% DPI showed increased DC than that without DPI, a more packed polymer can likely release fewer residual unreacted monomers when soaked in water, decreasing solubility [19]. Although the solubilization of calcium-based cements used as pulp-capping materials is necessary for biologic activity, it should occur in a gradual way [20]. Thus, the incorporation of DPI to the cement tested in this study could favor long-lasting therapeutic stimulus to the pulp, since the solubility was got more controlled in comparison with the material without DPI.

DC of methacrylates-based materials is also directly correlated to mechanical properties such as FS and EM [21,22], even in unfilled materials [23]. Thus, differences in DC of materials affected FS and EM of the materials tested in this study, so that incorporation of DPI at 2% promoted the highest DC, FS, and EM. Increased DC generates a higher number of cross-linkages, higher 3-D packing of polymer chains, and a reduction in the free volume of the material, increasing FS and EM [22,24,25]. Since FS and EM indicate the capacity of the cement to resist high chewing forces [26], likely, the light-cured calcium hydroxide cement with DPI 2% would present higher strength to displacement in the tooth preparation, leading probably to a long-lasting internal adaptation of the dental restorative and lower post-operative sensitivity.

Besides good physical properties and mechanical behavior, a calcium-based cement used as pulp-capping agent should present high alkalinity; this will irritate the pulp and stimulate the repair process of the pulp through the release of bioactive molecules sequestered within the dentin, such as bonemorphogenic protein-7 (BMP-7) and transforming growth factor-β1 (TBF- β1) [27]. In this way, the incorporation of DPI into the tested light-cured calcium hydroxide cement could not impair the release of hydroxyl ions to decrease pH. Since the materials with 2% and 4% DPI showed lower solubility than the control even that all of them provided similar pH to the eluates, one can assume that the release of hydroxyl ions was not impaired in materials with 2% and 4% DPI, so that decreased solubility would have been attributed to the lower release of residual unpolymerized monomers. Moreover, the fact that materials with 2% and 4% DPI were able to maintain pH of the eluate similar at 72-h and 7-day storage times indicates that the incorporation of DPI might promote more prolonged hydroxyl/calcium release in comparison with the control material. Possibly, the more packed polymer structure in materials with 2% and 4% DPI caused more entrapment of calcium hydroxide in the polymer chains, providing slight and gradual release. However, further investigations should be performed to confirm this hypothesis.

It was reported previously that the incorporation of 0.5 mol% DPI improved the physical properties of dual polymerizing self-adhesive resin cements[12]. In the present study, a preliminary investigation was performed to adjust the concentrations of DPI by weight to be incorporated into the Ultrablend® Plus based on the effect on the DC and handly characteristics. It was preferred to incorporate DPI by wt% instead of mol% into the cement tested since we were not sure of the exact quantity of monomers contained in the material, which is necessary to calculate the DPI mol%. Although this study pointed that the incorporation of DPI at 2% improved mechanical behavior and physicochemical characteristics of a light-cured calcium hydroxide cement, biological properties should be further evaluated to confirm that this material would have clinical application.

The incorporation of diphenyliodonium hexafluorophosphate intto the light-cured pulp capping calcium-based cement at 2 wt% was able to improve the degree of conversion, flexural strength, elastic modulus water sorption, solubility, and maintain its alkaline potential.

Author Contributions

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RODOLFO XAVIER DE SOUSA-LIMA: Methodology, Investigation and Writing - Original Draft.

LETÍCIA VIRGÍNIA FREITAS CHAVES: Methodology, Investigation and Writing - Original Draft.

BONIEK CASTILLO DUTRA BORGES: Conceptualization, Writing - Review & Editing and 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

The authors declare that this in vitro study did not require application to the institution's ethics committee.

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