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Effect of diphenyliodonium hexafluorphosphate on resin cements containing different concentrations of ethyl 4-(dimethylamino)benzoate and 2-(dimethylamino)ethyl methacrylate as co-initiators

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ABSTRACT

Objective. The present study evaluated the influence of diphenyliodonium hexafluorphosphate (DPI) combined with two different amines [ethyl 4-(dimethylamino)benzoate (EDAB) and 2-(dimethylamino)ethyl methacrylate (DMAEMA)] on the properties of model resin cements.

Methods. A comonomer base containing a 1:1 mass ratio of 2.2-bis[4-(2-hydroxy-3-methacryloxypropoxy)phenyl]propane (bis-GMA) and triethyleneglycol dimethacrylate (TEGDMA) was obtained, after which 1 mol% of camphorquinone and 0.1 mol% of hydroxyl butyl toluene were added to the comonomer blend. Concentrations of co-initiators varied at 0, 0.5 or 1 mol% for DPI and in 1 or 2 mol% for amines (DMAEMA or EDAB). Silanated Ba-Al-Si glass (60 wt%) was added as filler. The combination of each amine and DPI concentration resulted in 12 formulations, which had the following properties analyzed: degree of conversion (DC), water sorption (W_{sp}) and solubility (W_{sl}), flexural strength (FS) and flexural

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modulus (E_f). Data for DC, FS and E_f were analyzed by two-way ANOVA and Tukey's test ($\alpha = 0.05$) and W_{sp} and W_{sl} by Kruskal–Wallis and Dunn tests ($\alpha = 0.05$).

Results. EDAB promoted a higher DC than did DMAEMA; however, DPI increased DC for all materials with DMAEMA. The physical properties of resin formulations containing EDAB were significantly better than those of groups with DMAEMA; however, DPI had a positive influence on the chemical and physical properties of the model resin cement containing DMAEMA, especially with higher concentrations of amine.

Significance. EDAB proved to be more reactive than DMAEMA, being less influenced by DPI. Resins containing a 1:2 CQ/amine ratio had better properties than those with 1:1.

1. Introduction

Resin cements are low-viscosity composites that have been used to lute glass-ceramic restorations [1], and may be classified according to their initiator systems as self-cured, light-cured or dual-cured, combining both physical and chemical mechanisms [2]. Based on favorable properties such as low solubility, a wide variety of shades and good adhesion to dental and ceramic structures, these luting agents became the first choice for the cementation of metal-free restorations and prefabricated posts. Despite the fact that light-cured materials provide longer working time and show higher color stability [3,4] than dual or self-cure cements, their performance is impaired when the power of the light-source is attenuated [5], limiting the indications for these cements to clinical situations in which it is possible for the cements to be cured with high irradiance.

To improve the light-curing of resin-based materials, several methods have been evaluated, including: (i) the use of different photo-activation protocols [6–10], (ii) heating composite prior to the restorative procedure [11,12] and (iii) the use of different photoinitiator systems [13–15]. Diphenyliodonium hexafluorphosphate (DPI) is a salt that improves polymerization by forming higher quantities of free-radicals in conventional systems, such as those using camphorquinone (CQ), increasing the degree of conversion and positively influencing the properties of the polymer formed [14–16].

The reaction is improved due to the very low energy of the C–I bond present in the DPI molecule. After reacting with camphorquinone/amine, the C–I bond is irreversibly broken, providing two phenyl radicals that can further react with residual amines, abstracting a proton and creating a new free-radical [15,17]. In addition, DPI is capable of reacting with inactive CQ radicals formed during the polymerization process [12] and acting as an electron acceptor, decreasing the termination rate and enhancing initiation as new radicals are generated due to DPI fragmentation [17].

Resinous restorative materials usually have a binary initiator system. CQ, a widely used initiator in the light-curing of dental materials, cannot by itself generate high concentrations of free-radicals to promote suitable curing. Thus, amines are used as co-initiators to improve polymerization with this sensitizer [18]. In high concentrations, amines can increase composite susceptibility to oxidation, causing staining or discoloration of the restoration [19]. Conversely, low concentrations may hamper the reaction with CQ, resulting in lower monomer conversion [13].

Ethyl 4-(dimethylamino)benzoate (EDAB) and 2-(dimethylamino)ethyl methacrylate (DMAEMA) are conventionally used in CQ-amine systems, and their structures are very different. EDAB is an aromatic amine capable of donating a H^+, even at low concentrations [20,21], while DMAEMA has an aliphatic structure and a tendency to bond with O_2, inducing formation of oligomers [22]. Due to these differences, evaluating the behavior of these compounds in a ternary initiator system containing DPI is important to determine which amine is the better choice for the system, as well as to identify a concentration suitable for improving the polymerization process as well as the properties of resin composites with CQ as an initiator.

Therefore, the aim of the present study was to evaluate the influence of amines (EDAB and DMAEMA) at two concentrations (1 mol% and 2 mol%), and the interaction of these agents with different concentrations of DPI (0, 0.5 and 1 mol%), on model resin cements containing CQ as the initiator. The properties evaluated were degree of conversion, flexural strength and modulus, water sorption and solubility.

2. Materials and methods

2.1. *Preparation of the model cements*

Experimental resin materials were prepared at a 1:1 mass ratio of 2.2-bis[4-(2-hydroxy-3-methacryloxypropoxy) phenyl]propane (Bis-GMA) and triethyleneglycol dimethacrylate (TEGDMA) (Esstech Inc., Essington, PA, USA). Camphorquinone (1 mol%) and butylated hydroxytoluene (BHT) (Esstech Inc.) (0.1 mol%) were added (BHT as inhibitor), blended and homogenized for 1 h at room temperature with a magnetic stirrer. The model blend was divided into 12 fractions, labeled RC1 to RC12, and groups were formed according to the concentrations of diphenyliodonium hexafluorphosphate (0, 0.5 and 1 mol% – DPI, Sigma–Aldrich, Milwaukee, WI, USA), ethyl 4-(dimethylamino)benzoate (1 and 2 mol% – EDAB, Sigma–Aldrich) and 2-(dimethylamino)ethyl methacrylate (1 and 2 mol% DMAEMA, Sigma–Aldrich) in the formulations. Each formulation was loaded with 60 wt% of 0.7-µm average size silanated barium borosilicate glass fillers (Esstech Inc.). All chemicals were used without further purification.

2.2. Degree of conversion

To measure the degree of conversion (DC) of the model resin cements ($n = 5$), we prepared five specimens of each group using a circular stainless steel mold (4.5 mm diameter and 1 mm thick). The mold was filled with the resin cement and light-cured by means of a light-emitting diode (LED, Bluephase G2, Ivoclar-Vivadent, Schaan, Liechtenstein) at an irradiance of 1200 mW/cm^2 for 20 s. After 24 h, the measurements were performed by Fourier transform infrared spectroscopy (FTIR, Spectrum 100 Optica, PerkinElmer, Billerica, MA, USA) equipped with an attenuated total reflectance (ATR) device with a diamond crystal (Pike Technologies, Madison, WI, USA). Prior to the reading of the polymerized materials, the baseline was recorded with the unreacted monomers with a wave-number range of 1665–1580 cm^{-1}, 32 scans and resolution of 4 cm^{-1} and Happ-Genzel apodization in absorbance mode. DC was calculated according to a baseline technique [18] based on band ratios of 1638 cm^{-1} (aliphatic carbon-to-carbon double bond) and 1608 cm^{-1} (aromatic component group) as an internal standard between the polymerized and unpolymerized samples, based on the following expression: DC = 100 × [1 − (polymerized cement/unpolymerized cement)].

2.3. Flexural strength and modulus

Bar-shaped specimens (7 mm length, 2 mm width and 1 mm height) were prepared for three-point bending measurements ($n = 10$) [9]. The reduced specimen dimensions were necessary to allow the specimens to be light-cured in a single step (20 s). The specimens were light-cured by light-emitting diodes (LED, Bluephase G2, Ivoclar-Vivadent).

Flexural strength test was measured 24 h after irradiation on a universal testing machine (Instron 4411, Canton, MA, USA) with a span width of 5 mm at a crosshead speed of 0.5 mm/min. Flexural strength (FS, in MPa) and modulus (E_f, in GPa) were monitored by Blue Hill 2 software (Instron), and the σ and E_f values were calculated from the load–displacement trace. The tests were not performed for the cements containing 1 mol% of DMAEMA without DPI or with 0.5 mol% DPI, since the specimens fractured during removal from the silicone matrix, possibly due to the low conversion of the experimental resins.

2.4. Water sorption and solubility

Five specimens of each group were prepared in a manner similar to that used for the degree of conversion, in a circular stainless steel mold (4.5 mm diameter and 1 mm thick) and cured for 20 s. The specimens were individually dry-stored in Eppendorf tubes at 37 °C and weighed daily by means of an analytical balance (Discovery DV215CD, Ohaus Corporation, Pine Brook, NJ, USA) with an accuracy of 0.01 mg, until the weight stabilized (m1). The specimens were then immersed in distilled water for 7 days. After immersion, the excess water was removed with absorbent papers, and the specimens were gently dried with air for 10 s and weighed (m2). The specimens were then dry-stored and weighed again as initially described (m3).

The diameters and thicknesses of the specimens were measured by means of a digital caliper with 0.01-mm accuracy for calculation of the volume (V) of each disc, in mm^3. The values of water sorption (W_{sp}) and solubility (W_{sl}) were calculated according to the formula:

$$W_{sp} = \frac{(m2 - m3)}{V - W_{sl}} = \frac{(m1 - m3)}{V}.$$

As observed for the flexural test, water sorption and solubility of the cements containing 1 mol% of DMAEMA without DPI or with 0.5 mol% DPI were not analyzed, since the specimens fractured during removal from the matrix.

2.5. Statistical analysis

The normality and variance homogeneity of the data were analyzed. The results of W_{sp}/W_{sl} were subjected to Kruskal–Wallis and Dunn's post hoc tests ($\alpha = 0.05$). The data of FS, E_f and DC were subjected to two-way ANOVA and Tukey's post hoc test ($\alpha = 0.05$).

3. Results

3.1. Degree of conversion

The experimental resin cements without DPI, with EDAB as co-initiator, gave higher results than DMAEMA materials, and cements with 1 mol% DMAEMA gave the lowest values (Table 1). DPI improved DC for all materials with DMAEMA. For cements with EDAB, DPI reduced DC with the concentration of 1 mol%, and the effect of this co-initiator on the resins with 2 mol% of EDAB was not observed. The co-initiator EDAB promoted higher values of conversion compared with DMAEMA.

3.2. Flexural strength

The materials with DMAEMA as co-initiator had significantly increased FS with DPI, mainly in the materials containing 1 mol% of the amine (Table 1). For EDAB, DPI influenced only the materials containing 2 mol% of this amine, with 1 mol% DPI decreasing the FS. When the amines were compared, EDAB promoted the highest FS in materials without DPI. After the addition of 0.5 mol% of DPI, the resins containing 1 mol% DMAEMA had the lowest values, with the other cements presenting statistically similar values. For the cements with 1 mol% of DPI, the materials with 2 mol% DMAEMA and 1 mol% EDAB gave the highest FS, with the resins containing 1 mol% DMAEMA and 2 mol% EDAB presenting the lowest. As occurred for sorption and solubility, the three-point bending tests were not performed for cements containing 1 mol% of DMAEMA without DPI or with 0.5 mol% DPI, since the specimens fractured during removal from the silicone matrix, possibly due to low monomer conversion.

3.3. Flexural modulus

The E_f of cements with DMAEMA was positively influenced by DPI, in contrast to cements with EDAB, which were not influenced by the iodonium salt (Table 1).

Table 1 – Results of DC (in %), FS (MPa) and E_f (GPa) of the resin cements.

Test	DFI	1 mol% DMAEMA	2 mol% DMAEMA	1 mol% EDAB	2 mol% EDAB
DC	0 mol%	13.71 (2.30) Cc	44.95 (2.41) Bb	72.71 (2.54) Aa	76.79 (2.5) Aa
	0.5 mol%	31.60 (2.49) Db	58.46 (2.25) Ca	67.39 (2.35) Bb	77.26 (1.25) Aa
	1 mol%	54.2 (2.31) Ca	54.37 (2.28) Ca	68.02 (1.75) Bb	76.43 (1.52) Aa
FS	0 mol%	–	95.66 (10.88) Bb	118.78 (17.17) ABa	140.58 (14.91) Aa
	0.5 mol%	–	122.96 (23.38) Aa	130.78 (15.46) Aa	136.46 (14.09) Aa
	1 mol%	109.44 (17.7) Ba	120.64 (20.84) Aa	130.73 (14.61) Aa	102.19 (26.94) Bb
E_f	0 mol%	–	2.72 (0.36) Bc	3.82 (0.19) Aa	3.92 (0.27) Aa
	0.5 mol%	–	3.38 (0.37) Bb	3.84 (0.36) Aa	3.87 (0.28) Aa
	1 mol%	3.45 (0.23) Aa	3.92 (0.33) Aa	3.79 (0.33) Aa	3.71 (0.37) Aa

Means of each test followed by different capital letters (row) and small letters (column) differed significantly from Tukey's post hoc test ($p < 0.05$).

However, these materials gave higher E_f compared with cements with DMAEMA, except for resins with 1 mol% of DPI, which had similar E_f regardless of the amine used.

3.4. *Water sorption and solubility*

The addition of 1 mol% DPI increased the W_{sp} of cements containing 1 mol% of EDAB but had no effect on the other experimental concentrations (Fig. 1).

The cement with 2 mol% DMAEMA had the highest solubility compared with the cements containing EDAB without DPI. The addition of DPI had a positive influence on the W_{sl} of cements with 2 mol% DMAEMA, with this cement behaving similarly to the resins with EDAB (Fig. 2). DPI had no effect on the solubility of cements with EDAB as co-initiator.

4. Discussion

Resin cements are widely used in dentistry to bond indirect restorations such as inlays/onlays, full crowns, pre-fabricated posts and laminate veneers. Nevertheless, the light-curing resin cements should be used when thickness, type or opacity of the ceramic allows for suitable light transmission during photopolymerization, to promote adequate conversion as well as better properties of the material. The greater thickness of indirect restorations, except for laminate veneers, as well as the extension of the root canal, decreases the amount of light irradiance reaching the cement, making the use of a dual-cure resin cement necessary in these situations. But dual-cure resins have the disadvantage of reduced color stability due to the higher amount of amine in this system [5,23,24], as well as reduced working time, which, in some cases, can compromise

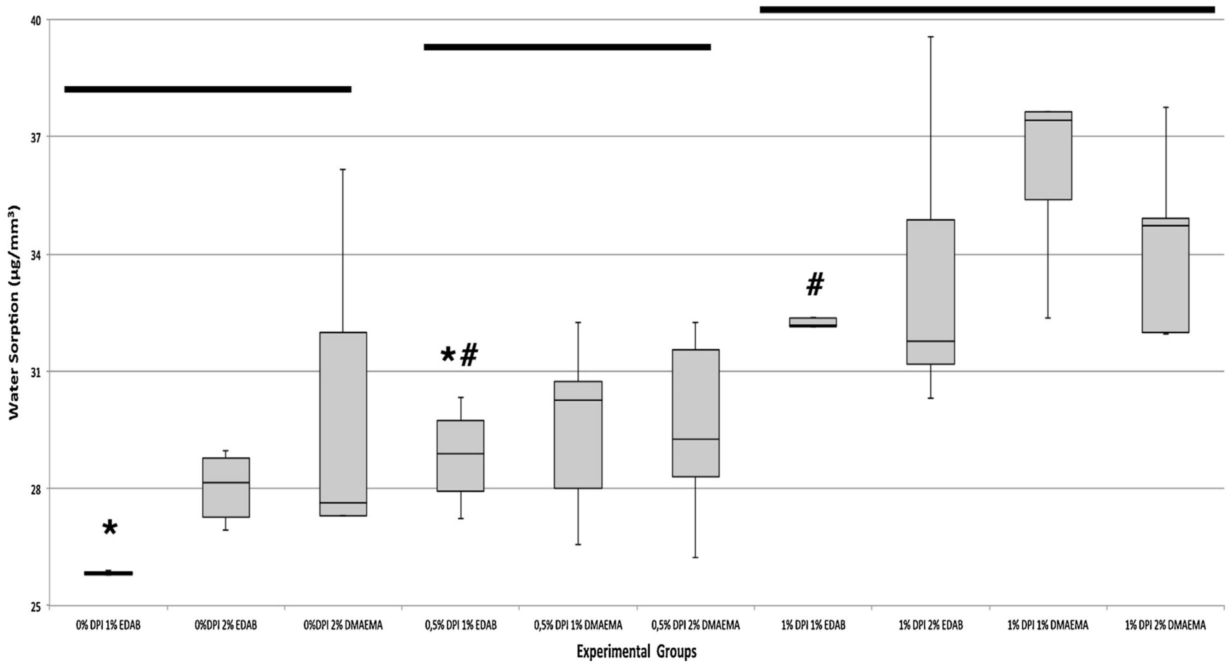

Fig. 1 – Median values of W_{sp} (µg/mm^3) analyzed by Kruskal–Wallis and Dunn's post hoc tests ($\alpha = 0.05$). Horizontal lines compare amines within each DPI concentration. Different symbols (*#) indicate statistically significant differences of DPI concentrations within amines.

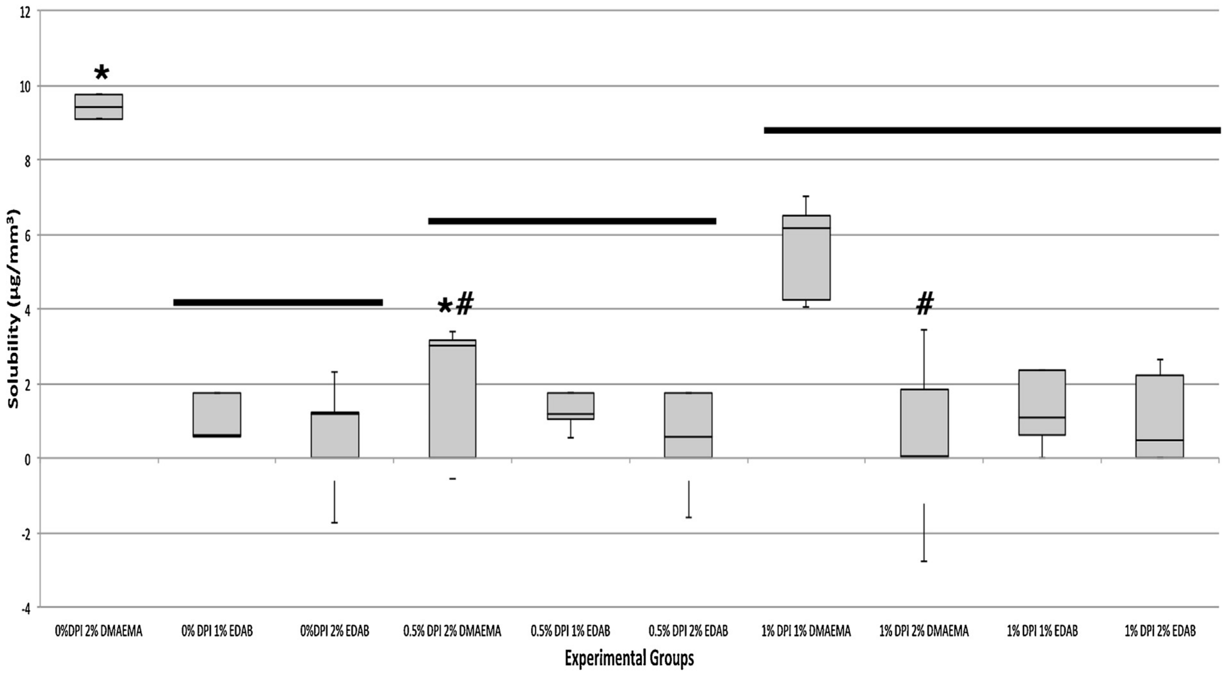

Fig. 2 – Median values of W_{sl} ($\mu g/mm^3$) analyzed by Kruskal–Wallis and Dunn's post hoc tests ($\alpha = 0.05$). Horizontal lines compare amines within each DPI concentration. Different symbols (*#) indicate statistically significant differences of DPI concentrations within amines.

the complete removal of excess resin cement after cementation. Therefore, it is important that light-curing resin cements are modified to become more reactive, improving the polymerization process and consequently increasing the degree of conversion of the material, not only to be used in one exclusive treatment, but also providing the advantages of these cements for all adhesive procedures.

The present study showed the interaction of DPI with two concentrations of different amines in a free-radical initiator system. The cements containing EDAB showed the highest DC, which can be explained by the structure of this co-initiator. DMAEMA has hydrophilic characteristics, different from those of CQ [25], which may have hindered the interaction of the two components, consequently reducing the degree of resin cure. Another aspect to be considered is the nucleophilicity, that is, the ability of a chemical compound to transfer electrons to an electrophile molecule (e.g. CQ in the triplet state) to promote a chemical bond. Nucleophilicity depends on the structure of the co-initiator, reducing the reactivity with the sensitizer according to the reduced number of C=C bonds present in the molecule [26]. EDAB has an aromatic ring on the backbone and higher nucleophilicity compared with that of DMAEMA, providing more H^+ ions to CQ, increasing reactivity of the systems and consequently improving monomeric conversion [20]. The best results obtained from the use of EDAB compared with DMAEMA are similar to those obtained in another study evaluating the curing properties of different initiator systems [26].

The results reported by a previous study differ from those seen in this study, showing DMAEMA with higher reactivity than EDAB [27]. This difference can be attributed to the CQ/amine ratio used (1:1 and 1:2 in this study; 1:4 in the previous study [27]). As already mentioned, amines can cause yellowing in resin materials, and large quantities of these agents can exacerbate this problem, highlighting the importance of improving polymerization, e.g., using DPI, decreasing the quantities of sensitizer and reducing agents.

However, the polymerization of cements containing DMAEMA can be improved by DPI. This iodonium salt minimizes the non-productive electron back-transfer from the ketone radical anion and the amine radical cation. DPI intercepts the ketone radical anion and prevents electron back-transfer from occurring, and irreversibly decomposes to an aryl radical and an aryl iodide, forming a new radical and improving polymerization.

Except for the resin containing 1 mol% DMAEMA without and with 0.5 mol% of DPI, which could not be tested due to the reduced degree of conversion, all cements in the present study showed W_{sp} between 25.82 $\mu g/mm^3$ and 37.41 $\mu g/mm^3$, similar to results with commercial resin cements evaluated in a previous study [28]. All model cements studied had W_{sp}/W_{sl} in accordance with ISO 4049 limits. The highest W_{sl} of resins containing 1 mol% DMAEMA + 1 mol% DPI and 2 mol% DMAEMA without DPI can be explained by factors such as DC. DMAEMA has an open chain and is less reactive than aromatic amines, therefore promoting slower polymerization when short irradiation times are applied [22]. Paradoxically, DPI increased W_{sl} of cements containing 1 mol% EDAB + 1 mol% of DPI. EDAB combined with CQ gave a high degree of resin conversion, even without DPI. Nevertheless, in cements containing the 1:1 ratio of CQ/EDAB, the concentration of amine was probably insufficient to react properly with the CQ and DPI, which can trigger a premature termination of the chains, reducing DC and negatively influencing W_{sp} (groups R7 and R11). However, the present results corroborate those of previous studies demonstrating that a high concentration of DPI can inhibit polymerization due to fast curing and premature beginning of the termination phase [14]. Thereby, the penetration of water into composite by interaction with ester, ether and hydroxyl groups, as well as porosities through the interface between organic matrix and filler [29], can be accentuated by the reduced degree of conversion.

FS and E_f were significantly improved in DMAEMA cements forming stiff and strong materials by the addition of DPI. This behavior can probably be attributed to increased crosslinking density in these cements combined with the higher DC, corroborating the results obtained in a previous study [14]. The amine EDAB, even without the use of DPI, significantly increased the DC of model cements. Resin cements with 2 mol% EDAB were less influenced by DPI, since, with the increased efficiency of amine, the maximum polymerization was probably achieved for the formulation examined. However, a significant reduction in FS was observed in 2 mol% EDAB + 1 mol% DPI. It can be speculated that using 1 mol% DPI with 2 mol% EDAB can increase not only the extent of polymerization but also primary cyclization, which can result in a polymer with a high degree of conversion but reduced strength, promoting, in this case, a reduction in FS.

Previous studies have shown that the addition of DPI to resin formulations could increase the rate of curing and degree of polymerization and improve the physical properties of resins with CQ/DMAEMA as the initiator system [14]. Based on this, the present study evaluated the different amines that can be used combined with CQ and DPI to obtain better results for the chemical and physical properties of resins in a ternary initiator system. According to the results, EDAB was highly compatible with CQ systems, improving the cure and physical properties of resins, whether combined or not with DPI. The present study showed the importance of DPI to resin materials with DMAEMA as the co-initiator, but also the efficiency and better compatibility of EDAB with CQ, promoting better chemical and physical properties even without the use of DPI. Thus, future investigations are needed to determine suitable concentrations of the components of this system and to establish the best relationship among initiators, co-initiators and the properties of different resin materials.

5. Conclusions

Based on the results obtained, it can be concluded that:

- EDAB gave significantly better results than DMAEMA in terms of the chemical and physical properties of the resins evaluated;
- DPI had a positive influence on the chemical and physical properties of the model resin cements tested, primarily those containing DMAEMA;
- DPI increased the degree of conversion of the resins containing DMAEMA, but reduced the conversion of systems containing CQ/EDAB at a 1:1 ratio; and
- the 1:2 ratio of CQ/amine can promote significantly better properties than the 1:1 ratio.

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