Light-Curing Units, Photoinitiators System, and Monomers on Physico-Mechanical Properties of Experimental Composite Resins

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ABSTRACT

This study evaluated the physico-mechanical properties of experimental composite resins made with different resin matrix and light-curing units. Experimental composite groups were divided according to monomers (BisGMA + TEGDMA-BT and BisGMA + BisEMA + UDMA + TEGDMA-BBUT) and photoinitiator system (camphorquinone-CQ and 1-phenyl-1,-2-propanedione-PPD). A quartz tungsten halogen (QTH) or light-emitting diode (LED) were used to light cure all materials. Knoop microhardness (n = 10) was determined using Knoop microhardness test. Compressive strength, diametral tensile strength, and Young modulus (n = 7) were obtained using a universal testing machine at crosshead speed of 1.0 mm/min. The data were submitted to a three-way ANOVA and the Tukey post-hoc test ($\alpha = 0.05$). QTH presented the highest total irradiance values and similar total radiant exposure to LED. For the compressive strength test, BT-PPD light-cured with LED group showed the lowest mean value. BT-CQ light-cured with LED group exhibited the lowest diametral tensile strength results. The BBUT-composite resins presented lowest Young modulus values, with no statistical difference between light-curing units (QTH and LED) and photoinitiators system (CQ and PPD). Application of QTH or LED in BT-based composite resins with PPD photoinitiator generated suitable results regarding the physico-mechanical properties.

Keywords: Biomaterials; blends; dental composites; polymerization; shrinkage

1. INTRODUCTION

The physicochemical properties of composites resins may be influenced by several factors, such as light-curing unit (LCU), time, irradiance, emitted light spectrum as well as increment thickness and composite resin shade [1]. Incomplete polymerization may increase the adverse effects affecting, besides the esthetics of the restoration, the postoperative sensitivity, marginal staining, and premature failure of the restoration [2 - 4].

LCUs composed of quartz tungsten halogen (QTH) lamps consists of a tungsten filament enclosed in a crystalline quartz casing, filled with a halogen-based gas, and connected to electrodes. The electricity flows through the filament generating light and heat [5]. The QTH lamps emit white light, i.e. they are considered broad-banded in its spectral emission. Thus, QTH LCU require filters to limit the wavelength range. Thus, only the blue spectral region is selected for composite resin light-curing, since it is the camphorquinone (CQ) absorption peak [4, 6].

On other hand, the light-emitting diodes (LEDs) units does not require filters to limit the wavelength ranges, since they are composed of doped semiconductors that emit light directly in the blue region of the spectrum (440-490 nm) that falls within the CQ absorption spectrum [6]. This property allows total use of the emitted light, decreasing heat generation, differing from QTH devices. Moreover, the use of LEDs apparatus in dentistry is also justified because they have higher lifespan than QTH and low degradation caused by operating temperatures which can results in reduction of the light-curing effectiveness [7 - 9].

The composite resins have been also studied aiming to improve their physicochemical properties. Thus, nowadays

the dentist has a wide variety of resin-based materials with different monomers and photoinitiator system [10 - 13]. The majority of commercial composite resins use bisphenylglycidyl dimethacrylate (bisGMA)/triethylene glycol dimethacrylate (TEGDMA) mixture as the base monomer of the resin matrix. The TEGDMA has low molecular weight (286 g.mol^{-1}) and viscosity (0.05 Pa.s) reducing the viscosity of the bisGMA/TEGDMA mixture and increasing the degree of monomer conversion. Furthermore, this diluent monomer is used to allow incorporation of the inorganic filler in composite resins. But, the incorporation of TEGDMA causes a clinically undesirable increase in polymerization shrinkage, such as decreases general mechanical properties, increases water sorption, and hinders color stability. Other diluent monomers with low viscosity and high molecular weight, such as ethoxylated bis-phenol A dimethacrylate (BisEMA; 540 g.mol^{-1} and 3 Pa.s), have been developed and introduced in commercial composite resins to overcome the limitations of TEGDMA [2, 8, 10, 11, 14].

Despite the high clinical acceptance of the CQ, alternative photoinitiators, such as 1-phenyl-1,2-propanedione (PPD), have been suggest substituting or act synergistically with CQ. These photoinitiators aim solve the inconveniences related final esthetic of the restorations in bleached teeth related with undesirable yellowing promoted by CQ [12, 15]. The PPD has an absorption peak within the ultraviolet spectrum (~410 nm) and extends slightly into the visible region. This molecule can improve the polymerization kinetics and to reduce the yellowing effect provided by CQ [12, 13, 16]. However, alternative photoinitiators for composite resins must not only have acceptable initial and long-term esthetical aspect, but also suitable physicochemical properties.

Some studies have shown that composite resins with PPD produces similar or superior physico-chemical performance compared with the CQ systems when a QTH is used for light-curing [12, 13, 15]. But the efficiency of these composite resins with LED unit still needs to be evaluated. Therefore, the aim of the present investigation was to evaluate the influence of the monomer composition and photoinitiator system in physico-mechanical properties (Knoop microhardness, compressive strength, diametral tensile strength, Young modulus) of experimental composites resins light-cured by QTH and LED units. The hypotheses tested were (i) PPD- and (ii) BisEMA-based experimental composite resins light-cured for both QTH and LED units will promote similar or superior physico-mechanical properties than CQ- and BisGMA-based materials, respectively.

2. MATERIAL AND METHODS

2.1 Experimental composite resin preparation

Two experimental composites resins were developed with organic matrix based on (1) BisGMA and triethyleneglycol dimethacrylate (TEGDMA) or (2) BisGMA, ethoxylated bis-phenol A dimethacrylate (BisEMA), urethane dimethacrylate (UDMA), and TEGDMA. The photoinitiator systems were composed of camphorquinone (CQ) or 1-phenyl-1,2-propanedione (PPD) with 2-(dimethylamino)ethyl methacrylate (DMAEMA). After monomer and photoinitiator system were blended, inorganic fillers were added in all groups. The inorganic content was composed of barium-aluminum-silicate (BaAlSi) (FGM, Joinville, SC, Brazil) and fumed silica (SiO$_2$) (FGM) silanized fillers (Table 1).

Table 1: Chemical composition for each experimental composite resin evaluated.

MATRIX	PHOTOINITIATOR	MATRIX (35% WT)	FILLERS (65% WT)
BT-CQ	CQ (0.4% wt) + DMAEMA (0.4% wt)	BisGMA (50% wt) + TEGDMA (50% wt)	80% (BaAlSi) / 20% (SiO$_2$)
BT-PPD	PPD (0.8% wt) + DMAEMA (0.8% wt)		
BBUT-CQ	CQ (0.4% wt) + DMAEMA (0.4% wt)	BisGMA (25% wt) + BisEMA (34.5% wt) + UDMA (34.5% wt) + TEGDMA (6% wt)	
BBUT-PPD	PPD (0.8% wt) + DMAEMA (0.8% wt)		

Average size: barium-aluminum-silicate (BaAlSi) 0.5 µm; silicon dioxide (SiO2) 0.04 µm. All materials were obtained from Sigma-Aldrich. BisGMA: bisphenylglycidyl dimethacrylate; BisEMA: ethoxylated bis-phenol A dimethacrylate; UDMA: urethane dimethacrylate; TEGDMA: triethylene glycol dimethacrylate; CQ: camphorquinone; DMAEMA: 2-(dimethylamino)ethyl methacrylate; PPD: 1-phenyl-1,2-propanedione.

All materials were measured using an analytical balance (±0.0001 g) (AX 200, Shimadzu, Tokyo, Japan) and the experimental composite resins were mechanically blended using a centrifugal mixing device (SpeedMixer DAC 150.1 FVZ- K, Hauschild Engineering, Hamm, North Rhine-Westphalia, Germany).

2.2 Light-curing units

The samples were light-cured using a quartz-tungsten-halogen (QTH; XL2500, 3M ESPE, St Paul, MN, USA) and a blue light-emitting diode (LED; UltraBlue IS, DMC, São Carlos, SP, Brazil). The diameter of the fiberoptic portion of the distal light guide end was measured using a digital caliper (Mitutoyo, Tokyo, Japan). The power (mW) of the light source was measured using a laser power thermopile sensor (10A-V2-SH, Ophir Optronics, Har Hotzvim, Jerusalem, Israel) connected to a microprocessor (Ophir Optronics, Har Hotzvim).

The output intensity was determined as follows:

$$\text{Irradiance (mW/cm}^2\text{)} = \text{Radiant power (mW)} / \text{Area (cm}^2\text{)} \qquad \text{(Eq. 1)}$$

The distributions of the spectra emitted by the LCU tip were obtained with a spectrometer (USB 2000, Ocean Optics, Dunedin, FL, USA), with a cosine corrector coupled to computer. Absorption spectra were recorded for each photoinitiator separately. Thus, the irradiance and the emitted spectra values were transferred to software (Origin 6.1, OriginLab Corp. Northampton, MA USA) to obtain irradiance values in the following regions of the spectrum through integral area calculations: wavelengths below 470 nm - PPD absorption light curve, and wavelengths between 450-490 nm - CQ absorption light curve.

2.3 Knoop microhardness

All experimental composite resins were inserted in a circular metallic matrix (2 mm height x 2 mm diameter) in a single increment to obtain 40 specimens (n = 10). A polyester strip was placed on surface of the specimen and pressed manually to decrease the oxygen inhibition and remove composite resin excesses in order to obtain a flat surface. For each sample, a new polyester strip was used in order to prevent any change in light intensity. Composite samples were light-cured using QTH and LED units with of the LCU tip in contact with matrix/material assembly following the specified exposure times. The samples were handled in environment with controlled temperature (25±1ºC).

The samples were stored dry in dark for 24 h at 37ºC. Then both top and bottom surfaces were wet-polished with 1200-grit silicon carbide (SiC) using a metallographic polishing machine (APL-4, Arotec, Cotia, SP, Brazil). After polishing procedure, the specimens were submitted to Knoop microhardness test (HMV-2000, Shimadzu, Tokyo, Japan) under a load of 50 g for 15 s. A Knoop diamond indenter was used to make five indentations on both surfaces of each specimen. The direction of indentations was changed from one sequence to another (1 mm). The mean of the five microhardness values was used as Knoop microhardness value.

2.4 Compressive strength / Diametral tensile strength / Young modulus

Matrices with 2 mm diameter and 4 mm height were placed onto a glass plate, filled in a single increment with experimental composite resins, and covered with a polyester strip. A new polyester strip was used for each sample. The experimental composite resins in the mold were light-cured according to light-curing times to provide a consistent radiant exposure between QTH and LED units. The top surface was subjected to wet-polished with 600- and 1200-grit SiC papers in a metallographic polishing machine (APL-4, Arotec). Thus, the samples were dry storage for 24 h in light-proof containers at 37°C (n = 7 for each test). The compressive strength measurements were done using a universal testing machine (Instron 1144, Canton, OH, USA) at crosshead speed of 1.0 mm/min. The Young modulus was calculated from the elastic portion of the strain / strain plot.

2.5 Statistical analysis

After being tested for homoscedasticity and normality, the data were submitted to three-way ANOVA, followed by Tukey's test, with a global significance level of 95% ($\alpha = 0.05$).

3. RESULTS

The QTH showed the highest total irradiance values and similar total radiant exposure to LED. Furthermore, QTH and LED presented the highest irradiance as well as radiant exposure values in wavelength range of 450-490 nm and < 470 nm, respectively (Table 2). The spectra of the LUCs used in this study are shown in Figure 1. The curve obtained by the QTH and LED units showed the emission peak at 484nm and 456nm, respectively.

Table 2: Irradiance emitted by LCUs (total and wavelength range <470 nm, 450-490 nm) and radiant exposure applied.

	IRRADIANCE (mW/cm²)			RADIANT EXPOSURE (J/cm²)		
	Total	< 470 nm	450-490 nm	Total	< 470 nm	450-490 nm
QTH	935	472.2	547.9	35.5	17.9	20.8
LED	597	466.3	428.1	35.8	28.0	25.7
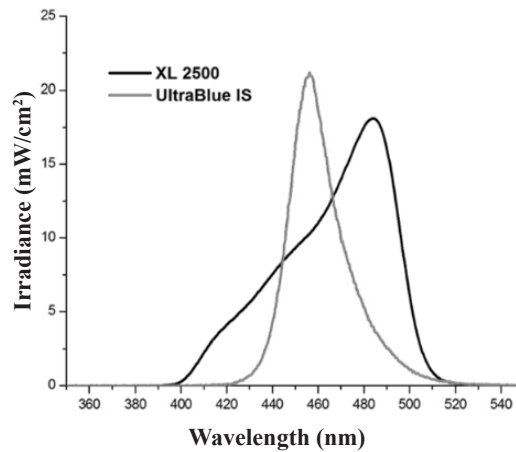
Figure 1: Emission spectra of LCUs.

The aspect of the CQ absorption curve showed a broad range of visible spectrum (blue spectral region) absorbed by this photoinitiator. The light absorption analysis of photoinitiator showed that CQ exhibited lowest absorption values in the UV region, with maximum absorption wavelength (Absmax) at 470 nm, while PPD initiates the curve in the UV region extended into the visible region (~ 485 nm), with Absmax at 398 nm (Figure 2).
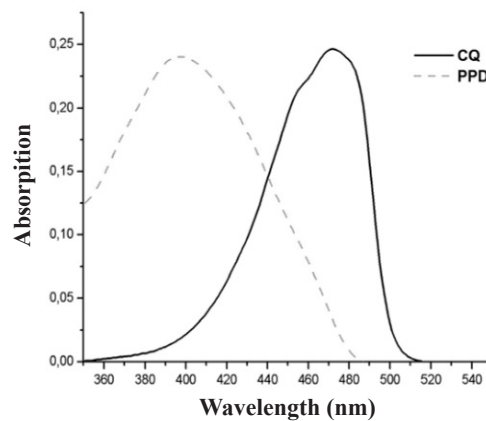
Figure 2: The spectra of the photoinitiators.

The compressive strength, diametral tensile strength, and Young modulus results are presented in Table 3. For compressive strength test the statistical analysis showed a significant effect only for photoinitiator ($\rho < 0.001$). The diametral tensile strength test showed significant effect for photoinitiator ($\rho < 0.001$) and for LCUs ($\rho < 0.001$). On other hand, Young modulus results showed a significant effect only for resin matrix ($\rho < 0.001$). However, no significant effect was found for interactions between resin matrix and photoinitiator system, resin matrix and LCUs, and photoinitiator system and LCUs, or for the interaction among all three study factors ($\rho > 0.05$).

Table 3 shows that compressive strength test revealed statistically significant difference only for BT-PPD composite resin light-cured with LED ($\rho < 0.001$), while BT-CQ light-cured using LED showed the lowest diametral tensile strength values ($\rho < 0.001$). The Young modulus analysis presented higher values for BT than BBUT ($\rho < 0.001$).

Table 3: Physico-mechanical properties mean (standard deviations) values (MPa).

MATRIX	PHOTOINITIATOR	QTH	LED
Compressive strength			
BT$^{\neq}$	CQ	344 (33.7) A,a	338 (51.4) A,a
PPD	334 (40.4) A,a	263 (36.3) B,b	
BBUT$^{\neq}$	CQ	299 (90.3) A,a	287 (47.0) A,a
PPD	347 (17.8) A,a	327 (69.4) A,a	
Diametral tensile strength			
BT$^{\neq}$	CQ	47.4 (1.3) A,a	42.7 (8.0) B,a
PPD	47.3 (7.5) A,a	53.6 (5.5) A,a	
BBUT$^{\neq}$	CQ	49.5 (8.3) A,a	46.4 (4.6) A,a
PPD	51.0 (6.0) A,a	50.7 (3.7) A,a	
Young modulus			
BT$^{\neq}$	CQ	370 (49.0) A,a	359 (45.9) A,a
PPD	357 (32.6) A,a	375 (33.8) A,a	
BBUT$^{\#}$	CQ	322 (40.5) A,a	309 (44.5) A,a
PPD	332 (33.7) A,a	327 (21.7) A,a	

Different letters indicate statistically significant difference for each test. Capital letters for comparison in columns and lowercase letters in rows (p<0.05). The Greek letters indicate comparison between resin matrix.

The statistical analysis showed no significant effect for resin matrix as well as for the study factors ($\rho > 0.05$). However, the top surface showed higher Knoop microhardness (pool mean = 28.2 KHN) than bottom surface (pool mean = 16.4 KHN) for all experimental composite resins tested ($\rho < 0.001$). Regardless of LCU, BT-PDD composite resin presented the lowest Knoop microhardness values at the top surface ($\rho < 0.001$). On other hand, the lowest Knoop microhardness mean at the bottom surface was found in BT- and BBUT-PPD groups light-cured with QTH ($\rho < 0.001$) (Table 4; Figures 3 and 4).

Table 4: Knoop microhardness mean (standard deviations) values (KNH).

MATRIX	PHOTOINITIATOR	QTH	LED	SURFACE
BT$^{\neq}$	CQ	29.8 (2.7) A,a	31.5 (2.4) A,a	Top*
	PPD	22.9 (3.4) B,a	24.5 (4.7) B,a	
BBUT$^{\neq}$	CQ	30.3 (3.7) A,a	28.4 (2.7) A,a	
	PPD	27.8 (3.7) A,a	30.4 (4.7) A,a	
BT$^{\neq}$	CQ	19.0 (3.7) A,a	19.6 (3.5) A,a	Bottom
	PPD	11.2 (2.0) B,b	17.0 (6.4) A,a	
BBUT$^{\neq}$	CQ	17.4 (3.3) A,a	17.9 (2.8) A,a	
	PPD	12.9 (1.7) B,b	16.5 (2.6) A,a	

Different letters indicate statistically significant difference for each test. Capital letters for comparison in columns and lowercase letters in rows (p<0.05). The Greek letters indicate comparison between resin matrix. Asterisk indicate comparison between surface.
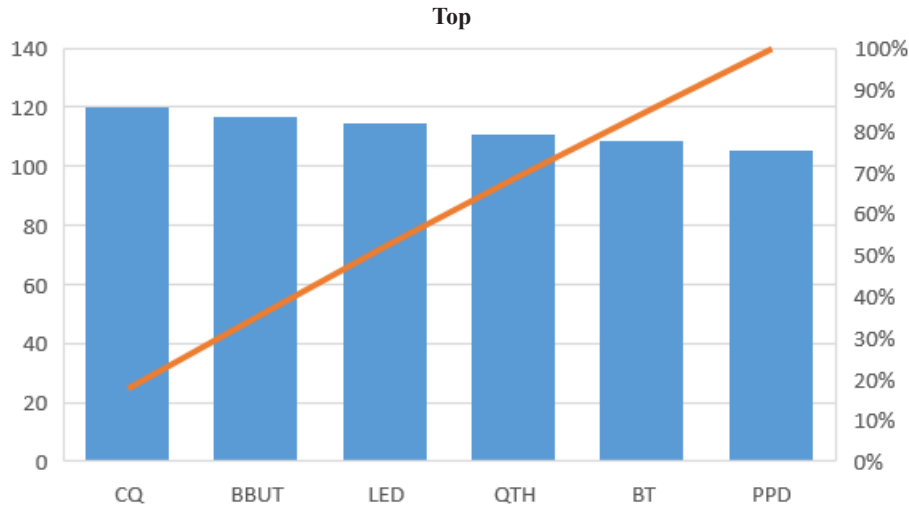
Figure 3: Pareto chart of the top surface.
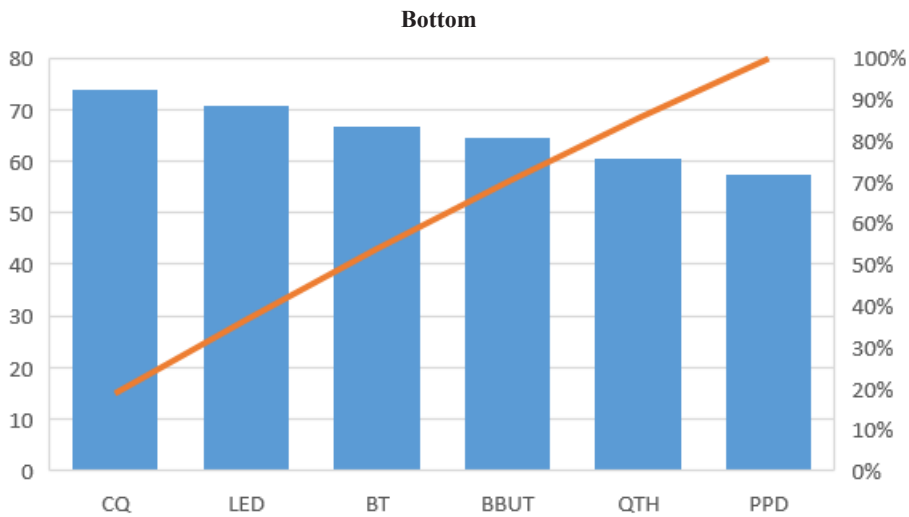

Figure 4: Pareto chart of the bottom surface.

4. DISCUSSION

The PPD photoinitiator promoted lower compressive strength and Knoop microhardness values than CQ in BT- and BBU-T-based experimental composite resins light-cured by QTH and LED, rejecting the first research hypothesis. In spite of the light-curing depends on the applied radiant exposure, composites resins with different chemical compositions (resin matrix and photoinitiator system) can present different physico-mechanical properties [1-4], even if the resin-based materials present similar degree of monomer conversion values [17].

Since PPD is a slow photoinitiator for the polymerization of composite resins [12, 18], the polymer formed from PPD-based experimental composite resins has some physico-mechanical properties different from that CQ-based experimental composite resins. The polymers formed from the PPD photoinitiator may have lower degree of monomer conversion values [19], because PPD produces a more linear polymer, reducing the physico-mechanical properties of composite resins [20]. In general, CQ-based experimental composite resins showed the highest Knoop microhardness and compressive strength mean values for both LCUs, mainly in BT-based experimental composite resins. Thus, it can be said that BT-based experimental composite resins with PPD photoinitiator showed the lowest degree of monomer conversion. On other hand, the PPD slower reaction could produce less polymerization stress at tooth/restoration interface. So, the slow reaction is an advantage to preserve the bond strength of restorations to tooth [21].

It would be expected that PPD-based experimental composite resins presented the lowest diametral tensile strength and Young modulus mean values too. But these tests showed no difference between composite resins with PPD and CQ

photoinitiators, because in both tests several factors influence the results, besides the degree of monomer conversion, such as cross-link density [12, 13, 17]. Despite PPD photoinitiator promotes slow polymerization of composite resins, and therefore low cross-link density [20], it is considered a cross-link density agent creating more cross-linked polymer structure in composite resins [22]. This could explain the similar results of the PPD- and CQ-based experimental composite resins in diametral tensile strength and Young modulus tests.

The compressive strength, diametral tensile strength, and Young modulus tests showed no statistically significant differences between CQ and PPD photoinitiators, mainly in BBUT-based experimental composite resins. Some studies have shown that the BT-based experimental composite resins, due to the large amount of monomers with low molecular weight and monomer chain (TEGDMA), requires longer light-curing times to reach pre-gel phase [23]. Thus, BT-based experimental composite resins with PPD could promote problems in polymerization process, because the BT matrix resin has "slow" polymerization (high viscosity and molecular weight, and low flexibility – BisGMA) and PPD also needs a long light-curing time to create a resin polymer with degree of monomer conversion values similar to composite resins with CQ [18].

The second alternative hypothesis was also rejected, since BBUT exhibited the lowest Young modulus mean values. This result was already expected, as the greater amount of the TEGDMA monomer in BT-based experimental composite resins leads to a high degree of monomer conversion values [11], which increases its Young modulus results. The small size of the TEGDMA molecule and the high concentration of double bonds makes it an excellent cross-link agent, allowing its penetration between the monomer chain formation and the polymer reticulation [8, 11, 14]. According to the Knoop microhardness results, it can be observed that high Knoop microhardness where found to BT-based experimental composite resins with CQ. Thus, when PPD photoinitiator is used in BT-based composite resins a longer light-curing time should be recommended.

Furthermore, no difference was found in the properties produced in the composites by the two light sources. Despite QTH LCU has a wider spectrum of light emission than LED (Figure 1), QTH emits less amount of light into the PPD region with better absorption peak (Table 2), in agreement with other studies [3, 5-8]. However, this difference in light emission between LCUs was not enough to produce statistically significant difference in physico-mechanical properties evaluated, except for Knoop microhardness results at the bottom surface of the experimental composite resins containing PPD. The QTH LCU produced lower Knoop microhardness values than LED at the bottom of these experimental composite resins. This could be explained by the lower efficiency of QTH LCU associated with its lower correlation with PPD photoinitiator [3, 6, 8, 12, 13, 15], besides loss of energy caused by the absorption and scattering of light, which made it difficult the light-curing procedure of composite resins at 2 mm depth as compared to LED LCU [24]. Thus, the PPD photoinitiator demonstrated potential to initiate the polymerization reaction of BT- and BBUT-based composites resins, but other studies should be conducted to verify the influence of the PPD on the formation of the monomer chain.

5. CONCLUSIONS

According to the findings of this study, it was possible to conclude that LUCs did not influence the physico-mechanical properties of BT- and BBUT-based composites resins with PPD or CQ, PPD photoinitiator demonstrating potential to initiate the polymerization reaction of dental composites (similar physico-mechanical properties to CQ), PPD photoinitiator should preferably be used with resin matrix composed of high molecular weight monomers, and BT matrix has larger diametral modulus than BBUT matrix.

6. ACKNOWLEDGMENTS

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