# Effect of Curing Lights and Bleaching Agents on Physical Properties of a Hybrid Composite Resin

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#### ABSTRACT

The aim of this in vitro study was to evaluate the microhardness (MH) and diametral tensile strength (DTS) of a minifill hybrid composite (Filtek Z250, 3M ESPE), polymerized with halogen lamp or second generation light-emitting diode (LED), submitted to different bleaching agents. Composite resin specimens were randomly polymerized according to experimental groups (halogen, 550 mW/cm<sup>2</sup>/20 seconds; LED, 550 mW/cm<sup>2</sup>/25 seconds) and subdivided into three subgroups (N = 8): A, without bleaching (control); H, 35% hydrogen peroxide; and C, 16% carbamide peroxide. After that, the MH test and DTS test were performed. Two-way analysis of variance (whitening  $\times$  light) and Tukey's tests ( $\alpha = 5\%$ ) were performed. For DTS, there were no statistical differences among the bleaching agents and the control group; however, the halogen group presented statistically lower DTS (p < 0.05) than the LED group. For the MH test, the carbamide peroxide group presented statistically lower MH means (p < 0.05) than the control groups, and there were no statistical differences among the lightcuring units. Sixteen percent carbamide peroxide reduced the MH of the hybrid composite tested. The second generation LED presented a performance similar to or better than the halogen lamp for hardness and DTS, respectively.

## CLINICAL SIGNIFICANCE

Repolishing of minifill hybrid composite is suggested, as the alteration caused after the contact with 16% carbamide peroxide was limited to the material surface. The second generation lightemitting diode is a good option for a curing light device when the polymerization initiator of composite resin is camphorquinone.

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#### INTRODUCTION

number of methods have  $\Lambda$  been described in the literature to improve the esthetic appearance of teeth. The most conservative one to change tooth color is the bleaching procedure. Nowadays, the frequently used bleaching techniques are in-office and home bleaching. The former makes use of a high concentration of bleaching agent. The whitening gel is applied to the teeth after protecting the soft tissues. For home bleaching, low levels of whitening agent are dispensed in a custom-made mouth guard for application to the teeth.

All tooth bleaching procedures use either hydrogen peroxide or carbamide peroxide as the whitening agent. When carbamide peroxide comes into contact with water, it breaks down into urea and hydrogen peroxide. Ten percent carbamide peroxide releases a maximum of 3.6% hydrogen peroxide.<sup>1</sup> Hydrogen peroxide, the active agent of bleach, is very unstable and undergoes dissociation, resulting in the release of free oxygen radicals, while urea decomposes, resulting in ammonia and carbon dioxide.<sup>2</sup> Although the mechanism of action of hydrogen peroxide is not well understood, it is considered to be an oxidation reaction, where the pigment molecules are broken down and the small compounds diffuse out of the tooth.<sup>1,3,4</sup> The vital tooth bleaching techniques can cause surface alterations in dental substrates and composite restorative materials, as seen in some studies.<sup>5-10</sup> In composite resin, the surface alterations may be associated with the physical properties of the material, and adequate polymerization is a crucial factor in obtaining the optimal physical performance of these materials.<sup>11</sup>

Composite resin polymerization occurs by the conversion of the monomer molecules into a polymer network, accompanied by a closer packing of the molecules, causing the composite to shrink.<sup>12,13</sup> When more intense light energy is used to polymerize a resin composite, more photons reach the camphorquinone photoinitiator molecules within the resin and more photoinitiator molecules are activated and raised to the excited state. In this excited state, camphorquinone collides with an amine, and a free radical is formed, which can then react with the carbon to carbon double bond (C==C) of a monomer molecule and initiate polymerization.<sup>14</sup>

Quartz-tungsten-halogen (QTH) lamp and light-emitting diodes (LEDs) are options for photoactivating composite resins. Halogen lamps are still the most used lightcuring units in the world. However, LED units are being increasingly used. LED units feature very narrow spectral ranges and are therefore highly efficient light sources.<sup>15</sup> Operating around 470 nm, with a bandwidth of about 20 nm, blue LEDs have all the spectral purity for highly efficient resin composite polymerization.<sup>16</sup>

The effect of 10 and 16% carbamide peroxide on the surface microhardness (MH) of restorative composite materials is controversial.<sup>17</sup> Some investigations associate the softening of composite resin with the application of bleaching gels.<sup>18,19</sup> Other investigations revealed no significant changes in hardness,<sup>20,21</sup> or increased surface hardness<sup>22,23</sup> because of the application of bleaching gels.

Different polymerization modes can lead to the resulting polymer having different structures, even though the degree of monomer conversion is the same.<sup>24</sup> The effect of bleaching agents on the polymer chains and filler particles of resinous materials has not yet been completely elucidated.

The bleaching agents may act on organic or inorganic structure of the composites. It is speculated that the high oxidative capacity of bleaching agents in contact with organic molecules would be able to damage the polymeric linkages that form the composite structure, making the composite more susceptible to degradation. Moreover,

TABLE 1. LIGHT-CURING UNITS (XL 3000-3M ESPE, ST. PAUL, MN, USA; Radii II-Sdi, Bayswater, Australia).				
Light curing unit	Light curing potency	Irradiation time		
QTH, XL 3000-3M ESPE	280 mW	20 seconds		
LED, Radii II-SDI	550 mW	20 seconds		
QTH = quartz-tungsten-halogen; LED = light-emitting diode.				

alterations in the inorganic phase could lead to a reduction in material properties, such as MH and wear resistance. De Alexandre and colleagues<sup>25</sup> reported that 10% carbamide peroxide bleaching agent altered the surface hardness of filled restorative materials, which could lead to increased wear and surface roughness.

Therefore, the aim of this in vitro study was to evaluate the effect of bleaching agents on MH and diametral tensile strength (DTS) of a hybrid composite, polymerized with QTH or LED. The null hypothesis was: there would be no difference in MH and DTS in specimens light polymerized with QTH or LED and submitted to different bleaching treatments.

## METHODOLOGY

For this study, a minifill hybrid composite resin Filtek Z250 (3M ESPE Dental Products, St. Paul, MN, USA) was used. According to the manufacturer's instructions, Filtek Z250 Universal Restorative is an esthetic composite specifically designed for use in both anterior and posterior restorations. Ninetysix cylindrical specimens were prepared in Teflon ring molds (6.0 mm in internal diameter and 3.0-mm depth). The molds were filled in three 1.0-mm increments and randomly polymerized according to the two polymerization mode experimental groups (Table 1). The potency measurements of the lightcuring units were made using an optical power meter (Broadband Power Energy Meter-13PEM001, Melles Griot, Carlsbad, CA, USA). The potencies of the light-curing units are described in Table 1. After the third increment was placed, the mold was held between two glass slabs separated by mylar matrix strips, and then pressed with a 500 g load for 30 seconds before photoactivation.

Only the specimens submitted to the MH test were finished with 400, 600, and 1,200 grit SiC paper and polished with 6-, 3-, 1-, 0.25-µm grit diamond pastes using a polishing cloth (Arotec S/A, Cotia, SP, Brazil). This protocol was defined in a pilot study. Each sample was ultrasonically cleaned in distilled water for 20 minutes, between different diamond paste grits to remove any remaining debris.

Each group was divided into three subgroups (N = 8): A, without bleaching (control); H, 35% hydrogen peroxide (Whiteness HP-FGM Produtos Odontológicos, Joinville, SC, Brazil); and C, 16% carbamide peroxide (Whiteness Perfect-FGM Produtos Odontológicos). The compositions of the bleaching agents are presented in Table 2.

# Subgroup A

The specimens were stored in relative humidity (100%) at 37°C for 14 days.

# Subgroup H

The specimens were submitted to treatment with 35% hydrogen peroxide. Two bleaching sessions were performed with a 7-day interval between them. Each session consisted of three applications of 30 minutes each of the bleaching agent on the top surface of the sample. After that, the samples were rinsed under running distilled water for 1 minute, dried with a soft absorbent paper, and stored in relative humidity at 37°C for 7 days.

# Subgroup C

The specimens were submitted to treatment with 16% carbamide

TABLE 2. BLEACHING AGENTS EVALUATED IN THE STUDY.			
Manufacturer	Composition (as disclosed		
	by the manufacturer)		
FGM Produtos	Hydrogen peroxide at 35%,		
Odontológicos	thickening, red colorant,		
	humectant (glycol) and water		
FGM Produtos	Carbamide peroxide at 16%,		
Odontológicos	neutralized carbopol,		
	potassium nitrate, sodium		
	fluoride, humectant (glycol)		
	and deionized water		
	AGENTS EVALUATED Manufacturer FGM Produtos Odontológicos FGM Produtos Odontológicos		



Figure 1. Indentations design.

peroxide. Fourteen bleaching sessions were performed. In each session, the bleaching agent was applied 4 hours a day, according to the manufacturer, covering the entire top surface. The specimens were placed in a vacuum-formed custom tray and stored in  $95 \pm 5\%$  relative humidity (100%) at 37°C. After that, the samples were rinsed under running distilled water for 1 minute, dried with a soft absorbent paper, and stored in relative humidity at 37°C for 20 hours. After the specimens received the bleaching treatments, the following tests were performed.

#### MH

The hardness at the top of each specimen was measured using a Knoop hardness test (FM, Future Tech Corp., Japan) under a 50 g load for 15 seconds, defined in a pilot study. Nine indentations were made at the approximate center of the specimen with distance among indentations of  $150 \,\mu\text{m}$  (Figure 1), immediately measured, and converted to a Knoop hardness number.

# DTS Test

The specimens were placed on their sides between parallel plates and loaded continuously at a crosshead speed of 10 mm/minute (Instron universal testing machine, model 4411, Instron Co, Canton, MA, USA) to breaking point. DTS values were calculated in MPa. The

TABLE 3. MEAN AND SD IN PARENTHESIS OF DIAMETRAL TENSILE STRENGTH (MPa).					
Whitening	Curing unit		Tukey		
	Halogen	LED			
Hydrogen	55.4 (12.0)	59.8 (8.6)	а		
Carbamide	57.6 (8.0)	66.6 (6.0)	а		
Control	56.7 (7.9)	62.5 (5.6)	а		
Tukey	В	А			

LED = light-emitting diode.

Mean values followed by different letters differ among them for the Tukey test (p < 0.05). Capital letters compare means of different curing units, and lower case letters compare means of different bleaching agents. p (whitening × curing unit interaction) = 0.7183.

#### TABLE 4. MEAN AND SD IN PARENTHESIS OF HARDNESS (KNOOP HARDNESS NUMBER) Whitening Curing unit Tukev **QTH** LED Hydrogen 94.3 (10.40) 101.8 (11.9) ab Carbamide 89.5 (7.92) 93.0 (10.40) b Control 102.5 (6.80) 102.4 (10.05) a Tukey А А OTH = quartz-tungsten-halogen; LED = light-emitting diode.

Mean values followed by different letters differ among them for the Tukey test (p < 0.05). Capital letters compare means of different curing units, and lower case letters compare means of different bleaching agents. p (whitening × curing unit interaction) = 0.5445.

MH and DTS test results were analyzed by two-way analysis of variance (ANOVA) and Tukey's test at the 5% significance level.

#### RESULTS

The DTS and MH results are listed in Tables 3 and 4, respectively. For the DTS test, ANOVA showed significant differences only between light-curing units. No significant differences were detected among the whitening agents and the control group. The Tukey test was applied to individual comparisons (p < 0.05), and it showed that the halogen media groups presented lower DTS and differed statistically (p < 0.05) from the LED media. For the MH, ANOVA test showed no significant differences between whitening agents. No significant differences were detected among the light-curing units. The Tukey test (p < 0.05) showed that the carbamide group presented the lowest MH means and differed statistically (p < 0.05) from the control group. The hydrogen group presented intermediary means and did not differ statistically from the carbamide or control groups.

#### DISCUSSION

The carbamide peroxide groups showed lower MH means than the hydrogen peroxide and control groups for both light polymerization modes; however, the bleaching treatments did not alter the DTS. The light polymerization modes presented differences only in relation to the DTS test, and the LED groups presented higher means than the QTH groups. Thus, the null hypothesis was rejected.

The increase or decrease in surface MH of resin composite materials after the contact with carbamide peroxide depends on the material composition.<sup>5,18,19</sup> The Z250 resin used in this study presents diure-thane dimethacrylate (UDMA) and bisphenol-A ethoxylated dimethacrylate (bis-EMA) in its composition that are considered high molecular weight resins. This, according to the manufacturer,

results in less shrinkage, reduced aging, and a slightly softer resin matrix.

Carbamide peroxide led to a reduction in composite surface MH for both polymerization units tested. The 16% carbamide peroxide used in this study contained carboxypolymethylene polymer (carbopol) as a thickening agent. This polymer improves tissue adherence and delays the release of the whitening agent. The reduction in composite surface MH by carbamide peroxide agent happened probably because the carbopol presents the solubility parameters similar to that bleaching agent. The bisphenol A-glycidyl dimethacrylate (bis-GMA) copolymer is susceptible to softening by chemicals with a broad range of solubility parameters.<sup>26</sup>

Wattanapayungkul and colleagues<sup>27</sup> reported that hydrogen peroxide and its free radicals can have an adverse effect on the resin–filler interface, resulting in filler–matrix debonding by water uptake and stress corrosion. This might affect the surface integrity of the materials. The 35% hydrogen peroxide used in this study did not cause a reduction in the surface MH of the composite.

For the DTS, there were significant differences between polymerization modes. The LED groups presented higher DTS means than the QTH groups. The bleaching treatments did not affect the DTS. Therefore, it may be suggested that the different bleaching agents remained restricted to specimen surface, not affecting the cohesive properties of the composite specimen body. According to Price and colleagues,<sup>28</sup> LED could polymerize the composites better than a QTH lamp. The LED used in this study is considered a second generation LED, which consists of multiple emitters on the same substrate, different from the first generation, which presents low irradiance and results in an insufficiently polymerized composite. Resin composite polymerization depends on the light intensity, irradiation time, and spectral distribution of the light source.<sup>29</sup> The availability of LED emission used in this study was higher than that of the halogen lamp.

The most reactive wavelength of camphorquinone, a polymerization initiator present in the composite tested, is near 470 nm. The peak of wavelength distribution for the halogen lamp is about 700 nm, in comparison with the distribution range of the LED tested, which is narrow and presents the proportion of wavelengths near 470 nm. Therefore, the polymerization depth and degree of conversion of the composite photoactivated by this LED is superior to that of the halogen lamp.<sup>30</sup> De Alexandre and colleagues<sup>25</sup> showed that LED can achieve similar or enhanced mechanical properties compared with a QTH curing unit for composites in which the photoinitiator is camphorquinone. As the spectrum of this LED is narrow, the photoinitiator of the composite needs to coincide with the emission spectrum of the light source. If the resin composite contains other photoinitiators, in addition to camphorquinone, which do not coincide with this region, composite polymerization is compromised.<sup>31</sup> The resin composite used in this study only contains the photoinitiator camphorquinone.

Moreover, for the LED mode used in this study, the energy produced was 25 J/cm<sup>2</sup> (25 seconds at  $1,000 \text{ mW/cm}^2$ ). For the QTH mode, the energy was 12 J/cm<sup>2</sup> (20 seconds at  $600 \text{ mW/cm}^2$ ). The higher amount of energy for the LED groups may be another factor that explains the results of the present study. When more energy is used to polymerize a resin composite, more photons reach the camphorquinone photoinitiator molecules within the resin, and more photoinitiator molecules are activated and raised to the excited state. Adequate polymerization is a crucial factor in obtaining the optimal physical performance of these materials<sup>11</sup> and is related to a better clinical performance.

However, both LED and QTH groups showed no significant differences in MH means when the same bleaching protocol was performed. It can be supposed that the top surface of the specimens had a similar polymer structure with higher cross-link density, because of the high number of monomers that may have been reacted, generating several growth centers.<sup>32</sup> Even though the two light-polymerizing systems, LED and QTH, presented different quantities of energy, the hardness at the top was statistically similar, as the top surface hardness may be less dependent on total energy, compared with the deeper areas of polymerized resin.33

There are no studies in the literature that show whether the alterations on the composite surface may affect the longevity of the restoration.<sup>17</sup> Further investigations are necessary to elucidate whether these alterations have any clinical relevance.

## CONCLUSION

Under the conditions of this study, 16% carbamide peroxide reduced the MH of the hybrid composite surface, independent of the type of light source used. Both bleaching treatments did not affect the cohesive property of the composite.

#### DISCLOSURE AND ACKNOWLEDGMENT

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