



PHYSICOCHEMICAL EVALUATION OF A HIGH-OPACITY COMPOSITE RESIN POLYMERIZED WITH DIFFERENT LED LIGHT-CURING UNITS

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ABSTRACT

Aims: This study aimed to evaluate the physicochemical properties of a high-opacity microhybrid composite resin in shade DA3.5, photoactivated using different LED light-curing units (LCUs). **Study design:** An experimental in vitro study was conducted to compare the effects of different generations of LED LCUs on the mechanical and chemical behavior of a composite resin. **Place and Duration of Study:** Research Laboratory of Dental Materials, Graduate Program in Dentistry (PPGO), Federal University of Ceará (UFC) **Methodology:** Three experimental groups were formed based on the type of LED LCU used: two second-generation devices, G1 (Kondentech) and G2 (Radii-Cal), and one third-generation device, G3 (Valo Cordless Grand). Physical properties were evaluated through flexural strength tests using resin bars (n=10; 12 × 2 × 2 mm), photoactivated according to each group. Knoop microhardness was assessed on cylindrical specimens (n=8; 1 mm thick × 15 mm diameter). Chemical properties were determined by water sorption and solubility tests (n=8). Data were analyzed using one-way ANOVA ($\alpha=0.05$) followed by Tukey's post hoc test. **Results:** No statistically significant differences were found among groups for flexural strength (p=0.183). Microhardness values showed a significant difference only between G3 and G1 (p=0.019). Water sorption (p=0.260) and solubility (p=0.414) did not differ significantly among groups **Conclusion:** The different LED light-curing units evaluated demonstrated satisfactory performance. No significant differences were observed in the physicochemical properties of the high-opacity microhybrid composite resin tested, indicating consistent polymerization performance across devices.

Keywords: Composite resins. Physical Properties. Mechanical Tests. Curing Lights. Dental.

INTRODUCTION

Advances in dental materials and the growing emphasis on conservative dentistry, which prioritizes the preservation of healthy tissue and the longevity of

restorations, have led to the widespread replacement of dental amalgam by composite resins (CRs). CRs fulfill both mechanical and aesthetic requirements (1). Despite their many advantages, the long-term success of composite resin restorations (CRRs) depends on various factors, including material selection (shade and type), insertion techniques, light curing, and finishing and polishing protocols. A substantial portion of CRR failures can be avoided by following appropriate clinical procedures (2).

CRs consist of an organic matrix—comprising monomers, inhibitors, pigments, and an initiator/activator system—bonded via coupling agents to an inorganic matrix filled with reinforcing particles (3). These components are essential for ensuring the physicochemical and mechanical stability of the composite, from the onset of polymerization to the completion of the restoration (4). Polymerization is the process by which monomers link to form polymer chains and can be initiated chemically, by light, or through a dual-curing mechanism (3).

Currently, light-cured composite resins (LCCRs) are the most commonly used in restorative dentistry. Camphorquinone (CQ) is the predominant photoinitiator and plays a critical role in enhancing the mechanical and biological properties of composites (5). When exposed to blue light at approximately 470 nm, CQ reacts with a co-initiator—typically a tertiary amine—to produce free radicals that initiate polymerization by breaking carbon double bonds and forming polymer chains (3). However, CQ imparts a yellowish tint proportional to its concentration, which can be a disadvantage in highly aesthetic restorations. This limitation has prompted the development of alternative photoinitiators (6).

Light-curing units (LCUs) using light-emitting diode (LED) technology have undergone significant advancements. Third-generation (multi-wavelength) LEDs emit a broader light spectrum, activating both CQ and photoinitiators that absorb at shorter wavelengths, such as those in the violet range. In contrast, second-generation (single-wavelength) LEDs, considered conventional, emit light primarily within CQ's absorption spectrum (7;8).

The effectiveness of the curing process delivered by LCUs is critical for achieving successful and durable restorations. Key parameters include irradiance,

total energy output, beam uniformity, and exposure time (9). Additionally, the composition of the restorative material influences photopolymerization, particularly the type and concentration of inorganic fillers used to control translucency (10).

Highly opaque composite resins are particularly effective for masking discolorations and stains, yielding excellent esthetic outcomes when used in layering techniques with more translucent resins. This makes them especially valuable in anterior restorations (11). However, the higher filler content required to achieve increased opacity may compromise certain physicochemical properties compared to more translucent composites (12).

Given these factors, this study aimed to evaluate the influence of light curing—using three LED curing units from different commercial brands, including two second-generation devices and one third-generation device—on the physicochemical properties of a high-opacity composite resin. The hypothesis of this study is that there will be no statistically significant difference between the groups according to the light curing unit used.

MATERIAL AND METHODS

Experimental Desing

This in vitro, quantitative laboratory study aimed to compare the physicochemical properties of a high-opacity composite resin, Opallis (FGM, Joinville, Santa Catarina, Brazil), in shade DA3.5. Specimens were divided into three groups based on the type of LED light-curing unit used: two second-generation units—G1 (Cordless LED 6, Kondentech) and G2 (Cordless Rádi-Cal, SDI)—and one third-generation unit—G3 (Valo Cordless Grand, Ultradent, Standard Mode). A total of 15 specimens were prepared for the flexural strength and elastic modulus test, 24 for the Knoop microhardness test, and 24 for the sorption and solubility test.

Irradiance

To determine the irradiance (IR) of the light-curing devices, a digital dental radiometer (RD-7, Ecel, Ribeirão Preto, São Paulo, Brazil) was used. Prior to testing, the radiometer was calibrated according to the manufacturer's instructions. The

active tip of each light guide was positioned vertically over the center of the radiometer's photosensitive sensor. Each device was activated for one minute before measurements were taken to ensure values that reflected clinical conditions (13).

Three irradiance readings were taken for each device, with each measurement lasting 40 seconds and a 30-second interval between them. The arithmetic mean of the three values was calculated. The 40-second exposure time was based on the composite resin manufacturer's recommendations. All light-curing units were cordless and fully charged at the time of testing, and all produced irradiance values exceeding 1000 mW/cm². In addition, irradiance and battery charge were reassessed after every five specimens were photoactivated.

Flexural Strength

Bar-shaped specimens (n = 5 per group) were fabricated for each experimental group (G1, G2, and G3). The composite resin was inserted into pre-fabricated addition silicone molds (12 mm in length × 2 mm in width × 2 mm in height), following a modified version of the ISO 4049:14 flexural strength protocol, as adopted by researchers such as Yap et al. (2003)(14) and Borges et al. (2012)(15). The resin was then light-cured for 40 seconds using the respective LED light-curing unit assigned to each group. After polymerization, the specimens were stored in distilled water at 37 °C for 24 hours. The elastic modulus (EM) and mini-flexural strength were evaluated using a three-point bending test with a 500 N load cell on a universal testing machine (Instron 3345; Instron Inc., Canton, MA, USA), operating at a crosshead speed of 0.5 mm/min and a span length of 10 mm.

Knoop Microhardness

A total of 24 cylindrical specimens were fabricated (n = 8 per group), each measuring 2.0 ± 0.1 mm in height and 6.0 ± 0.1 mm in diameter, using a metal mold. The composite resin was placed into the mold in a single increment with a #1/2 Almor insertion spatula and a Hollenback condenser No. 6 (Quinelato, Schobell Industrial Ltda., Rio Claro, São Paulo, Brazil). A polyester strip (TDV Dental Ltda., Pomerode, Santa Catarina, Brazil) and a 1 mm-thick glass slide were positioned over

the mold surface, and the resin was light-cured for 40 seconds according to the respective group protocol. The base of each specimen was marked to ensure standardization.

After 24 hours of storage in distilled water at 37 °C, the specimens were embedded using a mounting press (AROTEC® PRE-30Mi, Cotia, São Paulo, Brazil). The base of each specimen was polished under continuous water irrigation using a polishing machine (BUEHLER®, Illinois, USA), first with 600-grit silicon carbide abrasive paper for 20 seconds, followed by 1200-grit paper for 30 seconds. The polished surface was then subjected to Knoop microhardness testing using a Shimadzu HMV-2 microhardness tester (Tokyo, Japan). Five indentations were made on each specimen with a 50 g load applied for 15 seconds to determine surface microhardness values.

Water Sorption and Solubility

Twenty-four cylindrical composite resin specimens ($n = 8$ per group) were fabricated using a metal mold with dimensions of 1.0 ± 0.1 mm in thickness and 15.0 ± 0.1 mm in diameter, following ISO 4049:2009 guidelines. Since the specimen diameter exceeded the light-curing unit tip size, polymerization was performed by irradiating the center of the specimen followed by four additional irradiations around the periphery.

After 24 hours of storage at 37 °C, all specimens were measured and weighed. One diameter and one thickness measurement were recorded for each specimen using a digital caliper (Stainless Hardened, China), and mass was determined using a precision balance (Shimadzu, Japan). Initially, the specimens were placed in a glass desiccator inside an oven at 37 °C for 24 hours, then weighed. This cycle was repeated until a constant mass (m_1) was achieved.

Subsequently, the specimens were immersed in distilled water and stored at 37 °C for 7 days. After this period, they were removed from the water, gently blotted with absorbent paper for 1 minute, transferred to a second dry paper towel, and weighed to obtain the mass after water absorption (m_2). The specimens were then

returned to the desiccator, and the drying cycle was repeated until a second constant mass (m_3), referred to as the final dry mass, was obtained.

Water sorption was calculated using the following formula:

$$w_{sp} = \frac{m_2 - m_3}{v}$$

Water solubility values was calculated using the following formula:

$$w_{sl} = \frac{m_1 - m_3}{v}$$

Statistical Analysis

Statistical analysis was conducted using Jamovi software (Computer Software, Sydney, Australia). Data normality was assessed using the Shapiro–Wilk test, and homogeneity of variance was evaluated using Levene’s test; both confirmed a parametric distribution ($p > 0.05$). One-way analysis of variance (ANOVA) was performed to detect significant differences among the groups, followed by Tukey’s post hoc test to identify specific pairwise differences. A significance level of 5% ($p < 0.05$) was adopted for all analyses.

RESULTS

The results of the study are displayed in Table 1, organized according to the tests conducted. No statistically significant differences were found among the groups in the flexural strength test ($P = .183$), based on mean and standard deviation values. In the Knoop microhardness test, group G3 exhibited significantly higher microhardness values compared to group G1 ($P = .019$). However, no significant differences were observed between group G2 and either G1 ($P = .883$) or G3 ($P = .054$). Additionally, there were no statistically significant differences among the groups in the water sorption ($P = .260$) or solubility ($P = .414$) tests.

DISCUSSION

The high-opacity composite resin evaluated in this study was polymerized using devices with irradiance levels greater than 1000 mW/cm². Mechanical and physicochemical properties were assessed through flexural strength, Knoop microhardness, sorption, and solubility tests. Statistically significant differences were found only in the Knoop microhardness test. Thus, the null hypothesis—that there would be no statistically significant difference between the groups cured with different light-curing units (LCUs)—was rejected, as one of the properties showed a difference.

Irradiance is a critical factor for the curing efficiency and long-term performance of composite resins. It can be influenced by the light-source-to-resin distance, resin composition, and filler content, as demonstrated by Prochnow et al. (2022), who observed significant differences between low- and high-power LCUs (16). In this study, all curing devices were fully charged, which may explain the favorable test outcomes. Clinically, this emphasizes the importance of ensuring maximum battery charge and routinely monitoring irradiance throughout the day.

Another relevant factor is the light-curing distance: the greater the distance between the LCU tip and the resin surface, the lower the curing efficiency. This can be affected by the angle of the curing tip, exposure time, and the position of the tooth being restored (17). Among the LCUs tested, the Valo unit (G3) offers superior ergonomics, allowing better alignment and positioning. In contrast, the Kondentech (G1) and Rádi-Cal (G2), due to their straight-tip design, may pose access limitations. This is in line with Okuse et al. (2024), who concluded that light beams projected parallel to the composite resin and at minimal distances promote more effective curing and increased longevity (18).

The market offers a wide range of light-curable composite resins with diverse shades, chromas, and values. High-opacity resins, which contain greater pigment concentrations, are often used to mask discolored substrates (11). However, such resins require longer exposure times and thinner increments compared to more translucent composites (19). Pigments absorb part of the emitted light, acting as filters for certain wavelengths and limiting light penetration into thicker layers (20).

For the flexural strength test, 12 mm-long specimens were used, enabling single-shot photoactivation and standardized light incidence, as adopted by Yap et al. (2003)(14). This method produces comparable results to the 25 mm specimens recommended by ISO 4049:2019, which focus light only at the central point—potentially underexposing specimen edges and negatively affecting outcomes (15). In this study, no statistical differences were observed among groups, likely due to the standardized and minimal distance between the light source and the specimens, as well as the limited influence of pigment content on this property. These findings are consistent with Al-Zain and Platt (2021)(21), who found that flexural strength results vary significantly with curing distance.

Regarding Knoop microhardness, Group G3 (Valo) exhibited significantly higher values than Group G1 ($P = .019$). No significant differences were observed between Groups G2 and G1 ($P = .883$), or between G2 and G3 ($P = .054$). This may be attributed to Valo's collimated light guide, which emits a focused, non-divergent beam. This parallel propagation minimizes scattering and results in greater energy delivery to the resin surface, increasing hardness. Similar results were reported by Cardoso et al. (2021)(22), who observed increased hardness in specimens cured with high-irradiance LCUs and in close proximity to the sample using the Valo device.

It is well-documented that composite resins are not chemically stable after polymerization due to the organic matrix composition, exhibiting varying degrees of sorption and solubility over time (23;24). Moreover, the degree of conversion is inversely proportional to solubility, highlighting the critical role of LCUs in this process (23). This underscores the clinical importance of evaluating LCU performance. In the present study, no statistically significant differences in sorption and solubility were observed among the groups, suggesting that the risk of staining, degradation, marginal gap formation, or microleakage is likely minimized.

In conclusion, the LCUs evaluated in this study demonstrated the capability and efficiency to adequately cure the high-opacity composite resin. However, as this was a laboratory-based study, ideal and controlled conditions were established—conditions that may not be entirely replicable in clinical settings.

Table 1: Mean ($\pm$ SD) values of flexural strength, Knoop microhardness, water sorption, and solubility of the composite resin according to the LED light-curing units tested.

GROUP	FLEXURAL STRENGHT (MPA)	KNOOP MICROHARDNESS	SORPTION (μ G/MM ³)	SOLUBILITY (μ G/MM ³)
Kondentech (G1)	92.3 ($\pm$ 23.7) A	55.8 ($\pm$ 4.33) A	4.75 ($\pm$ 1.06) A	5.35 ($\pm$ 0.54) A
Radii-Cal (G2)	102 ($\pm$ 14.9) A	57.0 ($\pm$ 2.61) AB	5.27 ($\pm$ 0.52) A	5.17 ($\pm$ 0.53) A
Valo (G3)	87.1 ($\pm$ 18.9) A	63.1 ($\pm$ 6.89) B	4.81 ($\pm$ 0.64) A	4.69 ($\pm$ 1.23) A

Values are expressed as mean $\pm$ standard deviation. Different superscript letters within the same row indicate statistically significant differences according to Tukey's post hoc test ($\alpha = 0.05$).

Fonte: Autores, 2025.

CONCLUSION

The LEDs evaluated demonstrated satisfactory performance, with no difference between the physical-chemical properties of the high-opacity composite resin, thus all being favorable for its curing.

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COMPETING INTERESTS

The authors declare that they have no known financial conflicts of interest or personal relationships that could have appeared to influence the work reported in this article.

AUTHORS' CONTRIBUTIONS

Elvia Maria Sousa Campos: Writing – original draft, Methodology, Investigation, Conceptualization. Davi Iago Sousa dos Santos: Writing – original draft, Visualization, Methodology. Ketelyn Kerty Moreira de Oliveira: Writing – original draft, Visualization, Methodology. Sérgio Lima Santiago: Writing – review & editing, Writing – original draft. Celiane Mary Carneiro Tapety: Writing – review & editing. Lidiane Costa de Souza: Writing – review & editing, Writing – original draft.

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