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Multi-peak light sources can improve the physical and mechanical properties of a zirconia- and diatomite-based dental resin: an in vitro study

Fontes de luz de múltiplos picos podem melhorar as propriedades físicas e mecânicas de uma resina odontológica à base de zircônia e diatomita: um estudo in vitro

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ABSTRACT

Objective: This study aimed to evaluate the physicomechanical properties of three resin composites—Zirconfill (Maquira), Applic (Maquira), and Opallis (FGM)—photoactivated with single-peak (Demi Plus; Kerr) and multi-peak (Valo Cordless Grand; Ultradent; Bluephase N G4; Ivoclar Vivadent) light-curing units. The resins differed in filler composition: Applic contains micronized barium-alumino-silicate glass and nanometric silica; Opallis includes silanized barium-alumino-silicate glass and silicon dioxide nanoparticles; and Zirconfill incorporates diatomite, silica, and zirconium mixed oxide. **Material and Methods:** A total of 270 specimens were prepared to evaluate the degree of conversion (DC), flexural strength (FS), elastic modulus (ME), water sorption (SOR), and solubility (SOL). Morphological and compositional characterization was performed using scanning electron microscopy (SEM) and energy-dispersive spectroscopy (EDS). Data were analyzed using two-way ANOVA followed by Tukey's post hoc test (5%), after verification of normality (Shapiro-Wilk test). **Results:** Zirconfill, when photoactivated with the Bluephase unit, exhibited the highest FS and ME values and the lowest SOR and SOL rates, highlighting the potential of innovative fillers to enhance restoration durability. Although Opallis showed high DC values across all groups, its mechanical properties were inferior. **Conclusion:** It can be concluded that the resin composition and the selection of a light-curing unit with a spectrum and power compatible with the composite's photoinitiator system are essential to optimize the clinical performance and longevity of resin-based restorations.

KEYWORDS

Composite resins; Curing lights dental; Dental materials; Flexural strength; FTIR.

Resumo

Objetivo: Este estudo teve como objetivo avaliar as propriedades físico-mecânicas de três resinas compostas — Zirconfill (Maquira), Applic (Maquira) e Opallis (FGM) — fotoativadas com unidades de luz de pico único (Demi Plus; Kerr) e de múltiplos picos (Valo Cordless Grand; Ultradent; Bluephase N G4; Ivoclar Vivadent). As resinas diferem na composição das cargas: Applic contém vidro de bário-alumino-silicato micronizado e sílica nanométrica; Opallis, vidro de bário-alumino-silicato silanizado e nanopartículas de dióxido de silício; e Zirconfill, diatomita, sílica e óxido misto de zircônio. **Material e Métodos:** Foram preparados 270 espécimes para avaliar grau de conversão (DC), resistência à flexão (FS), módulo de elasticidade (ME), sorção (SOR) e solubilidade (SOL). A caracterização morfológica e composicional foi realizada por microscopia eletrônica de varredura (MEV) e espectroscopia por dispersão de energia (EDS). Os dados foram analisados por ANOVA de dois fatores e teste de Tukey (5%), após verificação da normalidade (Shapiro-Wilk). **Resultados:** A resina Zirconfill, fotoativada com Bluephase, apresentou os melhores resultados em FS, ME e os menores índices de SOR e SOL, evidenciando o potencial de cargas inovadoras para aumentar a durabilidade restauradora. Embora a Opallis tenha mostrado altos valores de DC, suas propriedades mecânicas foram inferiores. **Conclusão:** Conclui-se que a composição da resina e a escolha de uma unidade de fotoativação com espectro e potência compatíveis com o sistema fotoiniciador do compósito são essenciais para otimizar o desempenho clínico e a longevidade das restaurações.

PALAVRAS-CHAVE

Resinas compostas; Lâmpadas de polimerização dentária; Materiais dentários; Resistência à flexão; FTIR.

INTRODUCTION

Dental caries remains a global public health issue, especially in low- and middle-income countries, due to biological, behavioral, and socioeconomic factors, as well as limited access to dental services [1,2]. Secondary caries, which occur around existing restorations, can complicate treatment and increase costs for both patients and health systems [3,4]. Therefore, the longevity of restorations depends on the quality of the restorative materials and the training of dental professionals [3-5].

The strength of resin composites is directly associated with the composition of their fillers and the type of light-curing unit (LCU) employed [6-9]. To improve their mechanical properties, particles such as diatomite and zirconia have been incorporated into certain formulations [10-14]. Diatomite is a porous silicate derived from diatomaceous algae, characterized by high porosity, large surface area, low density, and good thermal stability [10-12]. Its lower cost compared to other filler particles makes it an economically viable alternative for dental composites. Additionally, its porous structure facilitates monomer penetration and interlocking with the polymer matrix, contributing to a higher degree of conversion and enhanced chemical stability [10-12].

The incorporation of diatomite into dental composites was first reported around 2011 in studies conducted by Wang et al. [12]; however, these investigations primarily focused on the effect of porous diatomite on filler content and fracture morphology of resin-based composites, rather than on evaluating their chemical and mechanical properties. Despite its advantages, diatomite presents some limitations. The most notable is that its highly porous structure, although beneficial for monomer infiltration, may reduce the mobility of the surrounding polymer network, thereby limiting the degree of conversion under certain conditions [10-12].

Zirconia, in contrast, is a widely used filler particle in dentistry due to its high mechanical strength, flexural resistance, surface hardness, and thermal stability [13,14]. Its inclusion in composites significantly contributes to the structural reinforcement of the material, enhancing resistance to wear and fracture [13,14]. However, since zirconia lacks porosity, it does not substantially contribute to chemical interlocking with the polymer matrix, serving predominantly as a physical reinforcement agent [13,14].

Furthermore, advancements in light-curing units (LCUs) play a crucial role in the quality of photopolymerization [6,15,16]. Single-peak LCUs (445–480 nm) efficiently activate camphorquinone but exhibit limitations in polymerizing alternative photoinitiators such as TPO, BAPO, MAPO, and Ivocerin, which may result in a lower degree of conversion (DC). In contrast, multi-peak LCUs (380–550 nm) provide a broader emission spectrum, leading to a higher DC and improvements in properties such as mechanical strength, water sorption, and solubility [17-19]. The degree of conversion, which reflects the extent of monomer conversion into polymer, is essential for the mechanical performance and biocompatibility of resin-based materials, influencing fracture resistance, hardness, and chemical stability. A low DC can accelerate material degradation by increasing water uptake and reducing the longevity of restorations [17-22].

In this context, the present study aimed to evaluate the physicomechanical properties of three resin composites containing different types of filler particles, polymerized using both single-peak and multi-peak LCUs. The evaluated properties included degree of conversion, flexural strength, modulus of elasticity, water sorption, and solubility. The experimental hypotheses tested were as follows: (i) polymerization with multi-peak LCUs results in a higher degree of conversion and superior mechanical properties compared to single-peak LCUs; and (ii) composites containing diatomite exhibit lower water sorption and solubility values compared to conventional composites.

MATERIALS AND METHODS

A total of 270 specimens were prepared for this in vitro study. Of these, 90 were used for the degree of conversion test, 90 for the flexural strength and elastic modulus test, and 90 for the sorption and solubility test (Figure 1). Three commercially available resin composites were evaluated, and for each combination of composite and light-curing unit, 10 specimens were fabricated (n = 10). The materials, compositions and other relevant characteristics are summarized in Table I. The specimens were prepared in standardized 2 mm increments. Light curing was performed using three different light-curing units: Valo Cordless Grand (Ultradent; 1250 mW/cm²), Bluephase N G4 (Ivoclar Vivadent; 1237 mW/cm²), and Demi Plus (Kerr; 1200 mW/cm²), each applied for 20 seconds.

Scanning electron microscopy and energy dispersive spectroscopy

Surface and charge characterization were performed using Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS). Three samples of each resin were prepared in 2 × 2 mm dimensions using silicone molds. Each group was

Table I - Resin composites used and their characteristics

RESINS	COMPOSITION	LOT
(APPLIC; MAQUIRA) (Maringá, Brasil)	Dimethacrylate groups, catalysts, organic filler, silicon dioxide, sodium fluoride, ethanol, Carbopol, BHT and iron oxide	266423
(OPALLIS; FGM) (Joinville, Brasil)	Monomeric matrix containing Bis (GMA), Bis (EMA), UDMA and TEGDMA. Inorganic Content: silanized Barium-Alumino silicate glass and silicon dioxide nanoparticles, camphorquinone as photoinitiator, accelerators, stabilizers and pigments.	20921
(ZIRCONFILL; MAQUIRA) (Maringá, Brasil)	Bis – GMA, Bis-EMA, TEGDMA and UDMA, Photoinitiator, Diatomite, Silica, Mixed Zirconia and Silica Oxide and Pigments.	410623

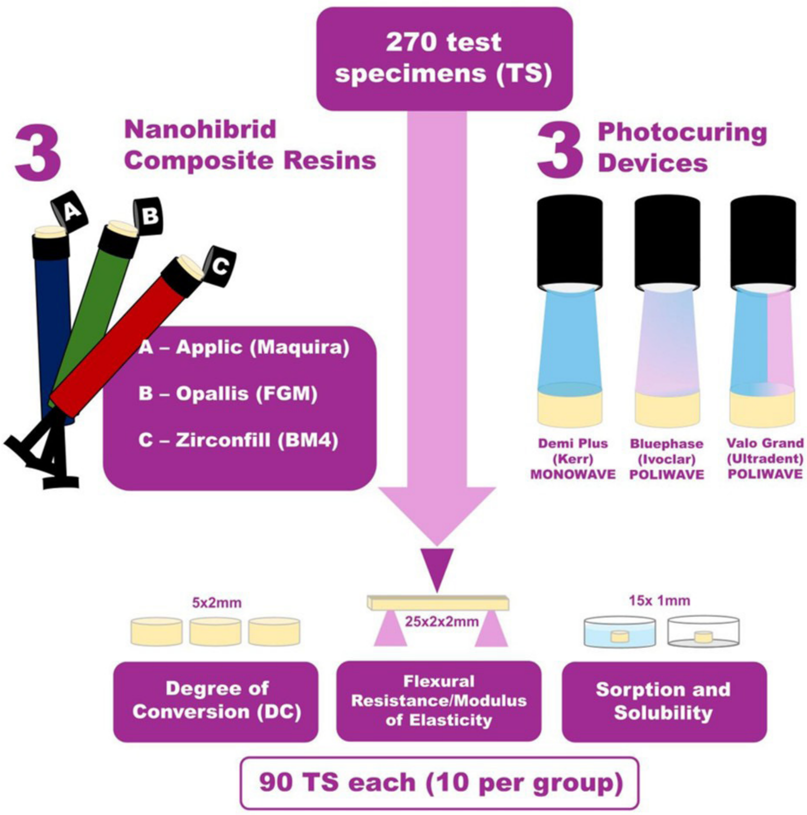

Figure 1. Schematic representation of materials, equipment used, and experimental tests performed.

light-cured for 20 seconds with the respective light-curing units (LCUs) used in this study, maintaining a 2 mm distance between the LCU tip and the sample surface.

Samples were mounted on metal stubs, sputter-coated with a ~10 nm layer of gold, and analyzed using a scanning electron microscope (Tescan Mira 3; Tescan Orsay Holding) operating at 15 kV. Images were acquired at 200× magnification, with resolution automatically adjusted by the Tescan Essence™ software. No filters or brightness/contrast modifications were applied.

Additionally, one sample of each resin, prepared with the same dimensions, was sputter-coated with carbon and mounted on a stub for elemental analysis using EDS, conducted on the same microscope under identical operational conditions. All images were analyzed under controlled conditions (22 ± 1 °C; 50-60% humidity) using Tescan Essence™ software for morphological interpretation and elemental distribution.

Degree of conversion

A total of 90 specimens were prepared and divided into nine groups. The specimens were molded using Teflon molds (5 mm in diameter × 2 mm in height), with a polyester strip placed on a glass slide beneath the mold. The mold was filled with resin, covered with another polyester strip and a glass coverslip, and light digital pressure was applied to the coverslip to ensure a smooth surface [15,18,19].

Prior to photopolymerization, the uncured specimens were analyzed by Fourier Transform Infrared Spectroscopy (FTIR; Perkin Elmer) in attenuated total reflectance (ATR) mode, with a resolution of 4 cm^{-1} over a spectral range of 650–4000 cm^{-1}. The analysis monitored the conversion of carbon–carbon double bonds (1638 cm^{-1}) into single bonds (1608 cm^{-1}) [18].

Photopolymerization was then performed while the specimens remained in contact with the ATR crystal. The light-curing units (LCUs) were pre-calibrated using a radiometer (RD-7; ECEL) to ensure accurate light output. Table II presents the light intensity, irradiation sequence, and the specific photopolymerizer used in all tests.

The LED units were positioned 2 mm above and perpendicular to the horizontal platform containing the ATR crystal. A spectrum collector (Spectrum; Perkin Elmer) was used to automatically acquire spectra during the polymerization process. The degree of conversion (DC) was calculated using the following equation, based on the ratio of absorbance bands before and after light polymerization:

$$DC(\%) = 1 - \left\{ \frac{\frac{(1638\,cm^{-1})}{(1608\,cm^{-1})}\;cured}{\frac{\left(1638\,cm^{-1}\right)}{\left(1608\,cm^{-1}\right)}\;uncured} \right\} x100 \quad (1)$$

Flexural strength and modulus of elasticity

Ten samples were prepared for each group (n=90) using rectangular Teflon molds (25×2×2 mm), in accordance with ISO standards [23,24]. The resin was inserted in a single increment, covered with a transparent polyester strip and a glass coverslip, and light-cured at three points (two at the top surface and one at the bottom) for 20 seconds each. A scalpel blade was used to remove surface irregularities without damaging the samples.

Table II - LCU devices, power, energy density studied groups and number of tested specimens

Photopolymerizer	Device power	Energy density (J/cm^2, 20 s)	Resin groups	Number of specimens
(Demi Plus; Kerr)	1200 mW/cm^2	24 J/cm^2	Applic-Demi (AD)	10
			Applic-Bluephase (AB)	10
			Applic-Valo (AV)	10
(Bluephase N G4; Invoclar Vivadent)	1237 mW/cm^2	24.74 J/mc^2	Opallis-Demi (OD)	10
			Opallis-Bluephase (OB)	10
			Opallis-Valo (OV)	10
(Valo Cordless Grand; Ultradent)	1250 mW/cm^2	25 J/cm^2	Zirconfill-Demi (ZD)	10
			Zirconfill-Bluephase (ZB)	10
			Zirconfiil-Valo (ZV)	10

The irradiance of each LCU was measured using a radiometer (values in Table II). The specimens were individually stored in Eppendorf tubes containing distilled water and kept in an oven at 37 °C for 24 hours prior to testing. Mechanical properties were evaluated using a three-point flexural test performed on a universal testing machine (EMIC DL-2000MF; Instron Brazil) at a crosshead speed of 0.5 mm/min with a 10 Kgf load cell. Flexural strength was calculated based on the fracture load, and the modulus of elasticity was derived from the load-deflection curves.

Sorption and solubility

To evaluate sorption and solubility, ten specimens from each of the nine groups were prepared (n=90) in accordance with ISO 4049:2019 [21,24]. The resin was inserted into the mold in a single increment, covered with a polyester strip and a glass coverslip, and light-cured for 20 seconds on the upper surface. After demolding, the bottom surface was polymerized for the same duration. The irradiance of each LCU was verified using a radiometer.

Sample thickness was measured with a digital caliper (0.01 mm accuracy) to calculate volume (mm^3). The specimens were stored in Eppendorf tubes inside a desiccator containing freshly dried silica gel and weighed after 24 hours using an analytical balance (XP204; Mettler Toledo, 0.00001 g accuracy). This cycle was repeated until a constant mass (M1) was obtained. The specimens were then immersed in 2 ml of distilled water at 37°C for 7 days. Every 24 hours, they were dried with absorbent paper, weighed (M2 - sorption), and returned to the water. After 28 days, they were dried in the desiccator and weighed daily until reaching a constant mass (M3 – solubility) [21,25].

Water sorption (SOR) and solubility (SOL) were calculated using the following equations:

$$SOR = \frac{(M2 - M3)}{V} \quad (2)$$

$$SOL = \frac{(M1 - M3)}{V} \quad (3)$$

Experimental design

The experimental design followed a 3 × 3 factorial scheme, considering two independent variables: the type of resin composite (Applic, Zirconfill, and Opallis) and the type of light-curing unit (Demi Plus; Kerr, Valo Cordless Grand; Ultradent, and Bluephase N G4; Ivoclar Vivadent), resulting in nine experimental groups. The sample size of 10 specimens per group was determined based on previous studies in the literature that employed similar experimental designs for evaluating the physicomechanical properties of resin composites [20-25]. These studies demonstrated that this sample size is adequate to detect statistically significant differences in continuous variables when analyzed using two-way ANOVA, as applied in the present study.

The Figure 1 summarizes the experiments in a schematic representation of materials, equipment used, and the tests performed.

Data analysis

After testing the data for normality using the Shapiro–Wilk test, inferential analysis was performed using two-way analysis of variance (ANOVA) with two factors (resin composite and light-curing unit). Tukey's post hoc test was applied for multiple comparisons at a significance level of 5%.

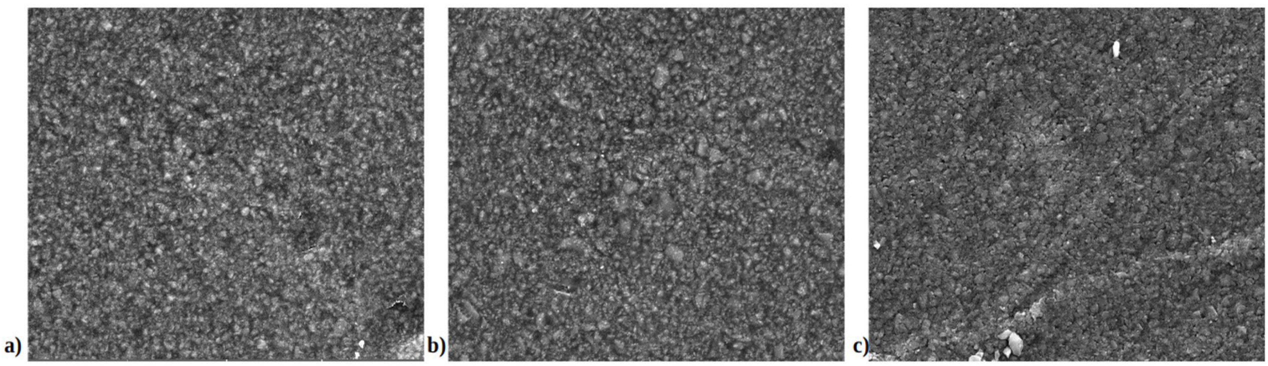

Figure 2. The magnifications considered were (a) x200 for Applic resin; (b) x200 for Opallis resin; and (c) x200 for Zirconfill resin.

Statistical analyses were conducted using GraphPad Prism software (version 6.0, 2010; La Jolla, CA, USA).

RESULTS

SEM and EDS

Figures 2 and 3 present the SEM and EDS results for the evaluated resin composites. In Figure 2, the micrographs reveal distinct surface morphologies among the materials. Zirconfill exhibited a more compact and homogeneous surface, with reduced porosity and a uniform distribution of filler particles, compared to Applic and Opallis, which showed particle agglomerates and topographical irregularities. These morphological features help to explain the superior mechanical performance observed for Zirconfill.

Figure 3 displays elemental mapping by EDS, confirming the presence of silicon (Si) and barium (Ba) in all formulations, while zirconium (Zr) was identified exclusively in the Zirconfill samples. The even dispersion of elements observed in the mapping supports the structural uniformity of the material. The inclusion of these imaging analyses is justified by their relevance in interpreting the filler composition and its potential correlation with the physical-mechanical properties evaluated.

According to the two-way ANOVA, the resin composite had a statistically significant effect on the degree of conversion (%), with p = 0.0001 and F = 37.36. Photopolymerization also had a significant impact (p = 0.0001; F = 268.7), as did the interaction between factors (p = 0.0001; F = 79.3). For flexural strength, both resin composite (p = 0.0001; F = 19.06) and photopolymerization (p = 0.0009; F = 13.68) showed statistically significant differences.

Additionally, the interaction between factors was also significant (p = 0.0001; F = 21.26). Regarding the modulus of elasticity, the effects of resin composite (p = 0.0003; F = 17.82) and photopolymerization (p = 0.0001; F = 16.57) were statistically significant, as well as their interaction (p = 0.0089; F = 5.641). Water sorption exhibited statistically significant differences for both resin composite (p = 0.0174; F = 4.260) and photopolymerization (p = 0.0001; F = 10.86). However, the interaction between factors was not statistically significant (p = 0.0591). For water solubility, resin composite (p = 0.0137; F = 4.529) and photopolymerization (p = 0.0001; F = 10.49) had statistically significant effects. However, the interaction between factors was not significant (p = 0.1052).

Degree of conversion

Table III presents the mean values and standard deviations of the degree of conversion (DC%) for the resin composites evaluated, along with the statistical analysis performed using the Tukey test. It was observed that the Zirconfill resin, when photoactivated with the Demi device, exhibited the lowest degree of conversion (35.4 ± 4.3%), which was significantly lower than the values obtained with the third-generation devices Bluephase (59.1 ± 1.8%) and Valo (60.3 ± 2.6%) (p<0.05). For the Applic resin, a similar behavior was noted, with a significantly lower DC when cured with the Demi device (47.5 ± 1.9%) compared to the Bluephase (60.0 ± 1.1%) and Valo (57.9 ± 1.1%), between which no statistically significant difference was observed. In contrast, the Opallis resin demonstrated high DC values across all light-curing units, with no statistically significant differences among them (p>0.05).

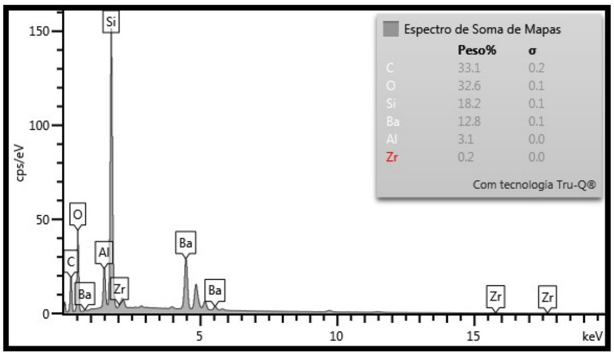

Figure 3. Elementary characterization of the EDS. Particle sizes ranged from ~10 μm to up to 5 μm and the elemental distribution showed the presence of barium, silicon, zirconium, aluminum, carbon and oxygen.

Table III - Mean (SD) and Tukey Test for degree of conversion (%), of the resin composites evaluated

	Demi	Bluephase	Valo
Zirconfill	35.4 (4.3) Cb	59.1 (1.8) Aa	60.3 (2.6) Aa
Applic	47.5 (1.9) Bb	60.0 (1.1) Aa	57.9 (1.1) Aa
Opallis	60.9 (1.7) Aa	59.8 (1.0) Aa	59.9 (1.0) Aa

Capital letters refer to columns; lowercase letters refer to lines; different letters present statistically significant differences (p<0.05).

Table IV - Result of the 2-way ANOVA test for conversion degree (%)

Source	DF	SS	MS	F	p
Resin	2	567.2	283.6	37.36	0.0001*
Photoactivation	2	1340	669.9	268.7	0.0001*
Interaction	4	1093	273.1	79.3	0.0001*
Residue	16	55.11	3.444		

*Statistically significant differences (p<0.05). Note: DF (Degrees of Freedom), SS (Sum of Squares), MS (Mean Square), F F-value and p (p-value).

The two-way ANOVA analysis (Table IV) revealed statistically significant differences between the different resin composites (p=0.0001) and between the different photoactivation protocols (p=0.0001). Moreover, a significant interaction between the factors "resin type" and "light-curing unit" was detected (p=0.0001), indicating that the degree of monomer conversion varied according to the resin composite evaluated.

Flexural strength and modulus of elasticity

The flexural strength and modulus of elasticity of the evaluated resin composites were significantly influenced by both the type of resin and the light-curing unit used (Table V). Zirconfill exhibited the highest flexural strength and modulus of elasticity values, particularly when photoactivated with the Bluephase device. The Applic resin demonstrated reduced flexural strength when cured with Bluephase compared to the Demi unit, whereas Opallis showed the lowest values for mechanical properties among the composites, regardless of the light-curing unit employed.

Sorption and solubility

Table VI presents the mean values and standard deviations for the percentage of water sorption and solubility observed in the studied groups. The highest sorption and solubility values were recorded in the Opallis group (microhybrid) when polymerized with the Bluephase device, leading to more significant changes compared to the Valo device, which demonstrated greater stability. This suggests that the light-curing unit influenced polymerization efficiency by promoting the formation of longer polymer chains and reducing intermolecular spaces within the matrix.

Some degree of polymerization was achieved with the Demi device. However, the curing was insufficient to convert most of the monomers, resulting in a higher number of residual monomers, which, in turn, increased water absorption. Notably, the presence of zirconia particles in the Zirconfill resin appeared to contribute to maintaining its properties, regardless of the light-curing unit used.

Table V - Mean (SD) and Tukey Test for flexural strength and modulus of elasticity, of the resin composites evaluat

Flexural strength			
Zirconfill	149.79 (11.5) Ab	176.02 (9.2) Aa	136.01 (7.3) Ab
Applic	158.51 (14.2) Aa	120.65 (10.9) Bb	143.69 (11.5) Aa
Opallis	135.51 (7.83) Ba	136.58 (15.8) Ba	127.53 (11.9) Aa
Modulus of elasticity			
Zirconfill	3.42 (0.22) Ab	3.73 (0.28) Aa	3.11 (0.26) Ac
Applic	2.85 (0.45) Bb	3.53 (0.37) Aa	3.04 (0.24) Ab
Opallis	2.84 (0.25) Bb	3.16 (0.19) Ba	3.07 (0.23) Aa

Capital letters refer to columns; lowercase letters refer to lines; different letters show statistically significant differences (p<0.05).

Table VI - Mean (SD) and Tukey Test for water sorption and solubility (μg/mm³) of the resin composites evaluated

Sorption			
Zirconfill	0.157 (0.01) Aa	0.159 (0.008) Aa	0.153 (0.01) Aa
Applic	0.163 (0.01) ACa	0.157 (0.005) Aab	0.152 (0.01) Ab
Opallis	0.169 (0.01) BCa	0.171 (0.01) Ba	0.151 (0.01) Ab
Solubility			
Zirconfill	0.157 (0.01) Aa	0.158 (0.008) Aa	0.152 (0.01) Aa
Applic	0.162 (0.01) ACa	0.156(0.005) Aa	0.151 (0.01) Aa
Opallis	0.168 (0.01) BCa	0.170 (0.01) Ba	0.151 (0.01) Ab

Capital letters refer to columns; lowercase letters refer to lines; different letters show statistically significant differences (p<0.05).

DISCUSSION

The selection of resin composites for restorative procedures should prioritize their physical-mechanical properties in order to optimize the quality and durability of the restorations [26]. The type of filler used in the resins directly influences their handling, aesthetic appearance, and mechanical properties [15,17]. In this study, the characterization of the resin components identified zirconium, barium, and silicon, with barium glass and diatomite being the main constituents of the filler in Zirconfill resin. The type and amount of filler influence the degree of conversion (DC), flexural strength (FS), sorption, and solubility of the resin composites [10,15,17,27-30].

The findings of this study indicate that resin composites containing diatomite, when photoactivated using second-generation light-curing units (LCUs), exhibit a degree of conversion (DC) below 40% (Table III). The presence of diatomite and zirconia appears to hinder effective light penetration, thereby reducing energy delivery and monomer conversion — an effect that contrasts with findings reported in previous studies [10,12,14]. Although specific studies evaluating the direct effect of diatomite or zirconia on light transmission are limited, it is well established that highly opaque fillers and materials with elevated refractive indices can impede light propagation, negatively affecting polymerization efficiency [18,29,30]. Additionally, the alternative photoinitiators incorporated in these formulations are not efficiently activated by second-generation LCUs [9,10,27]. In contrast, third-generation LCUs, which are characterized by higher energy density, were capable of overcoming these limitations and promoting improved polymer conversion.

In addition to classifying light-curing units (LCUs) based on their emission spectra (single-peak and multi-peak), it is essential to consider the energy density delivered by each device. Energy density, defined as the product of irradiance (mW/cm^2) and exposure time (s), represents the radiant exposure received by the resin and plays a critical role in determining polymerization efficiency [28-30]. In this study, the calculated energy densities were 24 J/cm^2 for Demi, 24.74 J/cm^2 for Bluephase, and 25 J/cm^2 for Valo. These values are within the range of 20–30 J/cm^2, which is considered adequate for effective polymerization of 2-mm composite layers according to previous studies [20,28-30]. Despite the similar energy densities provided by the different devices, differences in emission spectra and the compatibility with the photoinitiator systems impacted the degree of conversion and other physical-mechanical properties observed. Therefore, evaluating only irradiance or exposure time individually is insufficient — both the total energy density and spectral compatibility must be considered to accurately interpret polymerization outcomes [20,28,30].

Diatomite has been reported to enhance the physical and mechanical properties of resin composites, thereby increasing the longevity of restorations [10,12]. The literature suggests that resins containing diatomite can achieve a DC of approximately 60% immediately after multi-peak light-curing, increasing to around 80% after seven days [10,11]. Most studies have focused on evaluating DC using multi-peak units [10,12].

This study showed that Opallis resin achieved a DC greater than 58%, regardless of the type of LCU used. This elevated degree of conversion may be attributed to the smaller filler particle size (~0.5 μm), lower filler volume, and the composition of the organic matrix, all of which facilitates light penetration [28,29]. The combination of UDMA and TEGDMA promotes the formation of a higher number of crosslinks and results in a greater DC compared to systems based on BisGMA and TEGDMA, contributing to the superior performance observed in Opallis.

In contrast, the Applic resin, when light-cured with the Demi device, exhibited a DC below 50%. Although there is no ideal DC threshold for clinical performance, the literature suggests a range of 55–65% for occlusal restorative layers [27,29,30]. The reduced DC observed in the Applic may be attributed to its higher filler content (77–79%) and larger particle size (>0.7 μm), both of which hinder monomer conversion when using low-spectral-range sources such as single-peak LCUs. Conversely, Opallis, with its lower filler content, consistently demonstrated DC values above 59%, reinforcing previous findings regarding the influence of filler content, spectral compatibility, and LCU power on polymerization efficiency [19,28].

In this study, despite the low degree of conversion observed in resins cured with the single-peak light-curing unit, the FS and ME values met the requirements of ISO 4049:2019 and were consistent with findings from previous studies [10,22,24,27]. The resin containing diatomite and a higher volume of inorganic filler exhibited elevated flexural strength and modulus of elasticity values, indicating that diatomite reinforces the mechanical properties of the resin composites, as previously reported [10-12]. The mechanical properties of resin composites are closely associated with the DC, playing a crucial role in the long-term success of restorations [6,9,17,22]. The correlation between filler content and its influence on FS and ME is well established, resulting both from stress transfer between filler particles and the resin matrix, and from the quality of adhesion between these components [9,31,32]. However, some authors have reported a low correlation between the filler volume and FS [32-34].

The high FS and ME values observed for the Zirconfill resin may be attributed not only to the greater amount of filler particles, but also to the reinforcing effect of diatomite, which can deflect cracks and generate frictional forces that enhance the material's strength [10,12]. Several studies support the relationship between filler loading and improved mechanical properties in resin-based composites [6,9,17,20,27,33]. Additionally, the composition of the organic matrix plays a significant role in mechanical performance [9,34,35]. The presence of Bis-GMA and Bis-EMA, while increasing resin viscosity, promotes higher crosslink density and contributes to enhanced mechanical properties [9,34,35].

The results of this study demonstrated that resins containing traditional fillers exhibited lower flexural strength (FS) and modulus of elasticity (ME) compared to Zirconfill, even in the presence of reinforcing monomers such as Bis-GMA and Bis-EMA. Applic resin, when cured with the Bluephase device, presented values of approximately 120.65 MPa, while Opallis resin cured with the Valo unit showed values around 127.53 MPa. Both exhibited the lowest FS values among the groups tested (Table V). Nevertheless, all tested resins complied with the ISO 4049:2019 standards [24].

The type of LCU appeared to influence only the water sorption behavior of resins with conventional filler compositions. In contrast, Zirconfill resin showed no significant differences in sorption between specimens cured with single-peak and multi-peak LCUs. This stability may be related to the size and type of inorganic particles in Zirconfill's composition. Previous studies have indicated that the incorporation of zirconia and silica particles into resin composites can enhance hydrolytic stability by reducing water sorption and limiting the diffusion of water molecules into the polymer matrix, thus contributing to the long-term preservation of mechanical properties [10,11,25]. The sorption resistance observed in Zirconfill has been reported previously, reinforcing the potential of its filler formulation to enhance the material's hydrolytic stability [10]. Since water sorption is associated with chemical degradation through the release of residual monomers, the reinforcing particles in Zirconfill may help prevent this degradation, preserving the structural integrity of the polymer matrix [10,25].

The elevated solubility values observed in the Opallis resin photoactivated with the Bluephase device may be attributed to the release of residual free monomers, additives, and filler particles. This phenomenon is likely related to the hydrophilic nature and high mobility of these components, as well as the presence of TEGDMA in the resin composite used in this study [14,25,28,31]. A low degree of conversion can also lead to a higher concentration of unreacted monomers, which are prone to dissolution in humid environments, leading to material degradation and potentially compromising the longevity of the restoration. Conversely, groups cured with multi-peak LCUs exhibited the lowest solubility values, likely due to the higher energy output of third-generation units, which deliver greater radiant exposure to the resin materials [25,28].

Based on the findings of this study, the first experimental hypothesis was generally confirmed. The Zirconfill and Applic resin composites, when photoactivated with multi-peak light-curing units, exhibited higher degree of conversion and superior mechanical properties (flexural strength and elastic modulus) compared to those cured with a single-peak unit. However, for the Opallis resin, the type of light-curing unit did not significantly influence the degree of conversion, indicating that this effect also depends on the specific composition of each composite. Regarding the second hypothesis, it was partially confirmed. Zirconfill demonstrated lower water sorption and solubility values compared to the conventional Opallis composite, especially when multi-peak light-curing units were used.

However, the curing device also affected these properties, highlighting the importance of compatibility between the photoinitiator system, the inorganic filler characteristics, and the light source employed. Overall, the results of this study emphasize that both the type of filler and the photoactivation protocol directly influence the physicomechanical behavior and potential clinical longevity of composite restorations.

These findings further underscore the need for careful selection of light-curing units according to the resin composite formulation to ensure optimal clinical outcomes. Multi-peak LCUs proved to be more effective. Multi-peak LCUs proved to be more effective in activating alternative photoinitiators, especially in composites with high inorganic filler content, such as those containing diatomite and zirconia. The high flexural strength and elastic modulus values observed in these materials, even under less favorable curing conditions, indicate their potential for use in areas subject to greater mechanical demands [10,12,18,21,25,28,31]. Moreover, the superior mechanical stability and lower sorption and solubility indices associated with functional fillers suggest additional benefits in more challenging clinical environments [10,25,26,31].

Despite these promising results, some limitations of this study should be acknowledged. First, the polymerization time of 20 seconds used for all LCUs, particularly the single-peak device, may have been insufficient to fully cure resin composites with more complex filler compositions, such as those containing diatomite and zirconia. This may have affected the degree of conversion and, consequently, the physical-mechanical properties. Additionally, although the porous structure of diatomite may facilitate monomer infiltration and interfacial bonding, the restricted mobility of the polymer network in the presence of such fillers could also limit the extent of polymerization [10,12]. It is also important to note that the findings are based on in vitro conditions and may not fully replicate the behavior of these materials in the oral environment, where thermal, mechanical, and chemical challenges are constant. Finally, the study did not consider different curing times, which may influence polymerization outcomes. Future studies should investigate this variable to confirm and expand the applicability of the present findings.

CONCLUSION

Within the limitations of this study, it can be concluded that the type of resin composite and the light-curing unit significantly influenced the degree of conversion, flexural strength, elastic modulus, water sorption, and solubility of the evaluated materials. The Zirconfill resin composite, particularly when photoactivated with the Bluephase multi-peak device, demonstrated the most favorable physicomechanical performance, achieving the highest values of flexural strength and elastic modulus, as well as lower water sorption and solubility compared to the other composites. Although Zirconfill exhibited a lower degree of conversion when polymerized with the single-peak Demi Plus device, the use of multi-peak units effectively optimized its polymerization. The Applic resin also benefited from photoactivation with multi-peak devices, whereas Opallis resin, despite achieving high degrees of conversion regardless of the device used, exhibited lower mechanical properties. These findings highlight the importance of selecting composites with optimized inorganic fillers and using light-curing devices compatible with the photoinitiator systems and matrix characteristics to enhance the clinical performance and longevity of resin composite restorations.

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Author's Contributions

JMG: Conceptualization, Investigation, Writing – Original Draft Preparation, Validation, Visualization. TAMS: Investigation, Writing – Original Draft Preparation, Visualization. CSF: Investigation, Visualization. RPM: Investigation, Resources, Writing – Original Draft Preparation, Visualization. JPSJ: Investigation, Formal Analysis, Resources, Writing – Original Draft Preparation, Visualization. TMS: Investigation, Formal Analysis, Resources, Writing – Original Draft Preparation, Writing – Review & Editing, Visualization. SEPG: Investigation, Formal Analysis, Resources, Writing – Original Draft Preparation, Writing – Review &

Editing, Visualization. HBD: Investigation, Formal analysis, Project Administration, Resources, Supervision, Writing – Original Draft Preparation, Writing – Review & Editing, Visualization.

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

This study was conducted in accordance with

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REFERENCES

1. Moharrami M, Farmer J, Singhal S, Watson E, Glogauer M, Johnson AEW, et al. Detecting dental caries on oral photographs using artificial intelligence: a systematic review. Oral Dis. 2024;30(4):1765-83. http://doi.org/10.1111/odi.14659. PMid:37392423.
2. Crescente LG, Gehrke GH, Santos CM. Mudanças da prevalência de dentes permanentes cariados no Brasil e em países de renda média-alta nos anos 1990 e 2017. Cien Saude Colet. 2022;27(3):1181-90. http://doi.org/10.1590/1413-81232022273.46812020. PMid:35293454.
3. Bhadila GY, Baras BH, Balhaddad AA, Williams MA, Oates TW, Weir MD, et al. Recurrent caries models to assess dental restorations: a scoping review. J Dent. 2023;136:104604. http://doi.org/10.1016/j.jdent.2023.104604. PMid:37419382.
4. Nedeljkovic I, De Munck J, Vanloy A, Declerck D, Lambrechts P, Peumans M, et al. Secondary caries: prevalence, characteristics, and approach. Clin Oral Investig. 2020;24(2):683-91. http://doi.org/10.1007/s00784-019-02894-0. PMid:31123872.
5. Ávila NNM, Bottezini PA, Nicoloso GFN, Araújo FB, Ardenghi TM, Lenzi TL, et al. Prevalence of defective restorations and factors associated with re-intervention in primary teeth: a retrospective university-based study. Int J Paediatr Dent. 2019;29(5):566-72. http://doi.org/10.1111/ipd.12493. PMid:30860303.
6. Marović D, Daničić P, Bojo G, Par M, Tarle Z. Monowave vs. polywave light - curing units: effect on light transmission of composite without alternative photoinitiators. Acta Stomatol Croat. 2024;58(1):30-8. http://doi.org/10.15644/asc58/1/3. PMid:38562217.
7. Peres TS, Oliveira HLQ, Leyton C, Tereza M, Carlo HL, Price RB, et al. Effect of four different mono and multi-wave light-curing units on the Knoop hardness of veneer resin composites. Dent Mater. 2024;40(1):80-9. http://doi.org/10.1016/j.dental.2023.10.019. PMid:37919111.
8. Siqueira BDPS, Santos JN, Lima LS, Vieira TV, Lima AF, Gonçalves LS. Como as características dos LEDs de segunda e terceira geração podem influenciar a dureza de compósitos restauradores: uma revisão da literatura. Rev Fac Odontol. 2021;26(2):299-312. http://doi.org/10.5335/rfo.v26i2.13031.
9. Cilingir A, Ozsoy A, Mert Eren M, Behram O, Dikmen B, Ozcan M. Mechanical properties of bulk-fill versus nanohybrid composites: effect of layer thickness and application protocols. Braz Dent Sci. 2019;22(2):234-42. http://doi.org/10.14295/bds.2019.v22i2.1719.
10. Jordão I, Zanini M, Guarda M, Fronza B, Consani S, Pinheiro I, et al. Diatomite filler for resin composites application: a new approach for materials improvement. Res Soc Dev. 2022;11(16):e268111637738. http://doi.org/10.33448/rsd-v11i16.37738.
11. Regis M, Lima I, Pinto T, Medeiros HP, Oliveira HKC. Possibilities of the use of diatomaceous earth in the composition of dental materials: integrative review. Res Soc Dev. 2021;10(16):e521101623846. http://doi.org/10.33448/rsd-v10i16.23846.
12. Wang H, Zhu M, Li Y, Zhang Q, Wang H. Mechanical properties of dental resin composites by co-filling diatomite and nanosized silica particles. Mater Sci Eng C. 2011;31(3):600-5. http://doi.org/10.1016/j.msec.2010.11.023.
13. Shelar P, Abdolvand H, Butler S. On the behaviour of zirconia-based dental materials: a review. J Mech Behav Biomed Mater. 2021;124:104861. http://doi.org/10.1016/j.jmbbm.2021.104861. PMid:34600431.
14. Hong G, Yang J, Jin X, Wu T, Dai S, Xie H, et al. Mechanical properties of nanohybrid resin composites containing various mass fractions of modified zirconia particles. Int J Nanomedicine. 2020;15:9891-907. http://doi.org/10.2147/IJN.S283742. PMid:33328732.
15. Cardoso IO, Machado AV, Teixeira D, Basílio FC, Marletta A, Soares P. Influence of different cordless light-emitting-diode units and battery levels on chemical, mechanical, and physical properties of composite resin. Oper Dent. 2020;45(4):377-86. http://doi.org/10.2341/19-095-L. PMid:31794341.
16. Mauricio F, Medina J, Vilchez L, Sotomayor O, Muricio-Vilchez C, Mayta-Tovalino F. Effects of different light-curing modes on the compressive strengths of nanohybrid resin-based composites: a comparative in vitro study. J Int Soc Prev Community Dent. 2021;11(2):184-9. http://doi.org/10.4103/jispcd.JISPCD_423_20. PMid:34036081.
17. Boeira PO, Kinalski MA, dos Santos MBF, de Moraes RR, Lima GS. Polywave and monowave light-curing units effects on polymerization efficiency of different photoinitiators. Braz Dent Sci. 2021;24(4):1-9. http://doi.org/10.14295/bds.2021.v24i4.2661.
18. Nikhil V, Varshney I, Jha P. Effect of monowave and polywave light curing on the degree of conversion and microhardness of composites with different photoinitiators: an in vitro study. J Conserv Dent. 2022;25(6):661-5. http://doi.org/10.4103/jcd.jcd_223_22. PMid:36591587.
19. Tsuzuki F, de Castro-Hoshino L, Lopes L, Sato F, Baesso M, Terada R. Evaluation of the influence of light-curing units on the degree of conversion in depth of a bulk-fill resin. J Clin Exp Dent. 2020;12(12):e1117-23. http://doi.org/10.4317/jced.57288. PMid:33282131.
20. Farzad A, Kasraei S, Haghi S, Masoumbeigi M, Torabzadeh H, Panahandeh N. Effects of 3 different light-curing units on the physico-mechanical properties of bleach-shade resin

composites. Restor Dent Endod. 2022;47(1):e9. http://doi.org/10.5395/rde.2022.47.e9. PMid:35284327.

21. Atalı PY, Kaya BD, Özen AM, Tarçın B, Şenol AA, Bayraktar ET, et al. Assessment of micro-hardness, degree of conversion, and flexural strength for single-shade universal resin composites. Polymers. 2022;14(22):4987. http://doi.org/10.3390/polym14224987. PMid:36433113.

22. Borges ALS, Borges AB, Barcellos DC, Saavedra GDSFA, Paes TJDA Jr, Rode SM. Avaliação da resistência flexural e módulo de elasticidade de diferentes resinas compostas indiretas. Rev Pós-Grad. 2012;19(2):50-6.

23. Özduman ZC, Oglakci B, Bagis DMH, Temel BA, Dalkilic EE. Comparison of a nanofiber-reinforced composite with different types of composite resins. Polymers. 2023;15(17):3628. http://doi.org/10.3390/polym15173628. PMid:37688254.

24. International Organization for Standardization. ISO 4049:2019: dentistry: polymer-based restorative materials. Geneva: ISO; 2019.

25. Maia KMFV, Rodrigues FV, Damasceno JE, Ramos RVC, Martins VL, Lima MJP, et al. Water sorption and solubility of a nanofilled composite resin protected against erosive challenges. Braz Dent Sci. 2019;22(1):46-54. http://doi.org/10.14295/bds.2019.v22i1.1660.

26. Strini BS, Marques JFL, Pereira R, Sobral-Souza DF, Pecorari VGA, Liporoni PCS, et al. Comparative evaluation of bulk-fill composite resins: knoop microhardness, diametral tensile strength and degree of conversion. Clin Cosmet Investig Dent. 2022;14:225-33. http://doi.org/10.2147/CCIDE.S376195. PMid:35957701.

27. Tapety CM, Carneiro YK, Chagas YM, Souza LC, Souza NO, Valadas LA. Degree of conversion and mechanical properties of a commercial composite with an advanced polymerization system. Acta Odontol Latinoam. 2023;36(2):112-9. http://doi.org/10.54589/aol.36/2/112. PMid:37776508.

28. Werlang J, Dalfovo R, Neiva I, Obici A. Atenuação da intensidade de luz e profundidade de polimerização de resinas compostas. Arq Odontol. 2013;49(1):12-8.

29. Hyun HK, Christoferson CK, Pfeifer CS, Felix C, Ferracane JL. Effect of shade, opacity and layer thickness on light transmission through a nano-hybrid dental composite during curing. J Esthet Restor Dent. 2017;29(5):362-7. http://doi.org/10.1111/jerd.12311. PMid:28628735.

30. AlShaafi MM. Factors affecting polymerization of resin-based composites: a literature review. Saudi Dent J. 2017;29(2):48-58. http://doi.org/10.1016/j.sdentj.2017.01.002. PMid:28490843.

31. Silva TM, Petrucelli NF, Mendonça RP, Silva JP Jr, Campos TMB, Gonçalves SEP. Impact of photoinitiator quality on chemical-mechanical properties of dental adhesives under different light intensities. Braz Dent Sci. 2023;26(1):e3704. http://doi.org/10.4322/bds.2022.e3704.

32. Goracci C, Cadenaro M, Fontanive L, Giangrosso G, Juloski J, Vichi A, et al. Polymerization efficiency and flexural strength of low-stress restorative composites. Dent Mater. 2014;30(6):688-94. http://doi.org/10.1016/j.dental.2014.03.006. PMid:24703547.

33. Park JK, Lee GH, Kim JH, Park MG, Ko CC, Kim HI, et al. Polymerization shrinkage, flexural and compression properties of low-shrinkage dental resin composites. Dent Mater J. 2014;33(1):104-10. http://doi.org/10.4012/dmj.2013-126. PMid:24492120.

34. Frauscher KE, Ilie N. Depth of cure and mechanical properties of nano-hybrid resin-based composites with novel and conventional matrix formulation. Clin Oral Investig. 2012;16(5):1425-34. http://doi.org/10.1007/s00784-011-0647-3. PMid:22134667.

35. Moezzyzadeh M. Evaluation of the compressive strength of hybrid and nanocomposites. J Dent Sch. 2012;30(1):24-9.

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