





Facultad de Odontología

https://revistas.ucr.ac.cr/index.php/Odontos

BASIC RESEARCH:

Influence of the Cavity-Depth/light Tip-Material Distance on the Degree of Conversion and Physical Properties of a Nanohybrid Resin Composite Employing the Incremental Technique Influencia de la profundidad cavitaria y la consecuente distancia luz/material en el grado de conversión y propiedades físicas de una resina compuesta nanohíbrida usando la técnica incremental

Javier F. Roque Trujillo DDS¹ <u>https://orcid.org/0000-0002-7504-680X</u> Fabián Murillo-Gómez DDS, Msc, PhD² <u>https://orcid.org/0000-0002-9296-1858</u>

¹External researcher, School of Dentistry, Evangelical University of El Salvador. Operative Dentistry and Biomaterials Department, School of Dentistry, University of El Salvador. San Salvador, El Salvador.

²Dental Materials Research Lab (LIMD), School of Dentistry, University of Costa Rica. Restorative Dentistry Department, School of Dentistry, University of Costa Rica, San José, Costa Rica.

Correspondence to: Fabián Murillo-Gómez DDS, Msc, PhD - FABIAN.MURILLOGOMEZ@ucr.ac.cr

Received: 10-II-2025

Accepted: 12-III-2025

ABSTRACT: Evaluate the influence of different cavity depth and consequent light-tip/material distance, on the degree of conversion, biaxial flexural strength and microhardness of a nanohybrid resincomposite (top/bottom), employing the incremental technique. Quadrangular samples (4x4mm) with thickness variations (cavity-depth simulation: 2, 4, and 6 mm; n=10) were made using a nanohybrid resin-composite (Forma, A3, Ultradent) employing the incremental technique. Vickers Microhardness and degree of conversion were assessed on top/bottom surfaces. Biaxial flexural strength was tested on resin-composite discs (8.5mm diameter, 2mm thick) using 3D-printed molds (vertically stacked). Microhardness and Degree of conversion data were analyzed employing 2-way/ANOVA, Biaxial flexural strength with 1-way/ANOVA.For microhardness, "distance" factor plus "surface/distance" interaction resulted statistically significant (p<0.05). The 2 mm group, followed by the 4 mm group, showed the highest results (99.41 ± 52.23 and 84.1 ± 15.74 VHN), while the 6 mm group had the lowest (68.60 ± 18.69 VHN), with lower values observed on the bottom surfaces for the latter group only. Biaxial flexural strength data showed no significant differences among groups. Degree of conversion was significantly higher at the top surfaces compared to the bottom surfaces (top: 47.74±9.67%; bottom: 21.93±8.57%). At 2 and 4 mm distance, polymerization quality remained adequate (top/bottom surfaces). A 6 mm distance produced lower quality polymerization, mainly on the bottom surfaces. In such scenario, an additional photopolymerization cycle may be desirable. The current outcomes may be related only to the conditions (RBC, distances and LCU) employed in this study.

KEYWORDS: Cavity depth; Nanohybrid composites; Degree of conversion; FTIR; Microhardness; Biaxial flexural strength; Physical properties; Photopolymerization distance.

RESUMEN: Evaluar la influencia de la profundidad cavitaria/distancia de la luz fotopolimerizante y el material sobre el grado de conversión, resistencia a flexión biaxial y microdureza de una resina nanohíbrida (superficies superior/inferior), mediante técnica incremental. Se fabricaron muestras cuadrangulares (4x4 mm) con variaciones de espesores de 2, 4 y 6 mm (n=10 por medida), simulando la profundidad de una preparación cavitaria, utilizando una resina nanohíbrida (Forma, A3, Ultradent) empleando la técnica incremental. Se evaluó microdureza Vickers y grado de conversión en superficies superior/inferior. La resistencia a la flexión biaxial se probó en discos de resina (8,5 mm diámetro, 2 mm espesor) utilizando moldes impresos 3D (apilados verticalmente). Microdureza y grado de conversión se analizaron empleando ANOVA de 2 vías y resistencia a la flexión biaxial con ANOVA de 1 vía. Para microdureza, el factor "espesor" más la interacción "superficie/espesor" resultaron estadísticamente significativos (p<0.05). El grupo de 2 mm mostró resultados más altos (99.41±52.23), seguido por 4 mm (84,1±15,74 VHN), mientras que 6 mm tuvo los más bajos (68,60±18,69 VHN), observándose valores más bajos en las superficies inferiores para este último grupo. Resistencia a la flexión biaxial no mostró diferencias significativas entre grupos. El grado de conversión fue significativamente mayor en superficies superiores en comparación con las superficies inferiores (superior: 47,74±9,67 %; inferior: 21,93±8,57 %). Para los grupos de 2 mm/4 mm, la polimerización siguió siendo adecuada (superficies superior/inferior). 6 mm produjo una polimerización de menor calidad, especialmente en superficies inferiores. El grado de conversión fue mayor en superficies superiores para todos los grupos. No se observó impacto significativo en el grado de conversión o resistencia a flexión según la distancia desde la luz fotopolimerizante. Las propiedades físicas y el grado de conversión no fueron proporcionales.

PALABRAS CLAVE: Profundidad cavitaria; Resinas compuestas nanohíbridas; Grado de conversión; FTIR, Microdureza; Resistencia a flexión biaxial; Propiedades fisicas; Distancia de fotopolimerización.

INTRODUCTION

Resin-based composites (RBCs) are widely employed for restoring teeth, offering both structural and aesthetic benefits, in anterior and posterior regions (1). Additionally, the potential to achieve adhesion to dental tissues, when used properly along with adhesive systems, makes them a conservative choice for dental restorations (2, 3). In the United States alone, nearly 200 million composite restorations are placed annually (4). However, achieving desired outcomes with RBCs can be challenging due to their complex nature, precise light-curing demands, strict adhesive protocols, and delicate handling.

Since Ralph Bowen introduced Bis-GMA monomer (Bisphenol-A glycidyl methacrylate) in the 1960s (5), RBCs have evolved to enhance stability and predictability. They primarily consist of three components: the organic matrix, inorganic fillers, and a silane-based coupling agent that facilitates interaction between the organic and inorganic phases (6). The organic matrix combines dimethacrylates like bisphenol-A glycidyl dimethacrylate (BisGMA), urethane dimethacrylate (UDMA), triethylene glycol dimethacrylate (TEGDMA) (7) between others. This phase polymerizes and undergoes contraction, one of the greatest challenges when employing these materials. Inorganic fillers (from 4nm to 20µm in size), primarily silica, glass, and metallic oxides, enhance the material's physical and mechanical properties (8). The coupling agent, typically 3-methacryloxypropyltrimethoxysilane (MPTS), is a bifunctional methacrylated-silane molecule that binds inorganic fillers within the organic matrix (9). As RBCs are mainly categorized by particle size, the development of nanotechnology has allowed the implementation of nanometric particles as RBC fillers. Given that resin-based composites (RBCs) are primarily classified by particle size, the evolution of nanotechnology has facilitated the integration of nanometric particles as fillers in RBCs. Nanofilled and nanohybrid RBCs incorporate nanometric-sized particles (<100 nm), affording them an optimal balance between aesthetic potential and physical-mechanical properties (10, 11, 12).

During the photopolymerization of resinbased composites, they undergo a transformation from a paste-like to a solid consistency. This process is initiated by stimulating the photoinitiators within the organic matrix of the material (1, 8), using a specific light source provided by a light-curing unit (LCU) to match photoinitiators' wavelength range sensitivity. Camphoroguinone is the most commonly used photoinitiator among RBCs for dental applications. It can be activated within a wavelength range of 468-470 nm (blue light) (13). Some recent RBCs have implemented alternative photoinitiators, such as phenyl propanedione (PPD), diphenyl (2,4,6-trimethibenzoyl) phosphine oxide (TPO), and certain germaniumbased initiators (ivocerin) (14). These alternative photoinitiators have sensitivity ranges below 425 nm (violet light) and require photopolymerization using polywave LCUs emitting blue and violet light.

To effectively activate the polymerization of these composites and maximize their potential, luminous energy must reach them at the appropriate wavelength for a specific period of time (15). This ensures optimal physico-mechanical properties and proper biocompatibility. The quality of photopolymerization can be influenced by the material's composition, shade, and translucency (16). However, clinical success depends not only on the material's composition but also on an adequate photopolymerization technique performed by the clinician and the use of a high-quality LCU (17). On the LCU and technique side, factors such as the LCU's main peak(s) (intensity and wavelength), beam profile/uniformity, positioning of the light tip (distance and inclination), and exposure time can affect the material's degree of conversion (DC) and associated properties (8, 10, 18). Flaws in this process can lead to negative consequences, including biological risks, as unreacted monomers may move into surrounding tissues, causing cytotoxicity and impacting the longevity of restorations (19, 20). Zhou et al (4) reported that deficient polymerization can release monomers like bis-GMA, HEMA, and TEGDMA into the environment, leading to significant toxic effects on human pulp and gingival fibroblasts, and sometimes causing genetic mutations (in vitro).

Determining the degree of conversion (DC) through spectroscopy analysis is a reliable method to gauge the extent of polymerization in a polymeric material. It involves estimating the percentage of carbon-carbon double bonds (C=C) that have converted into carbon-carbon single bonds (C-C) during the polymerization process. This measurement provides valuable insight into an estimated number of monomers that have successfully formed polymer chains within the material. Achieving a high DC typically correlates with a previous delivery of an increased amount of radiant energy to a RBC. However, supplementary indirect methods, such as assessing microhardness, depth of cure, elastic modulus, and flexural strength, among others, have been suggested to enrich this analysis (21, 22, 23).

Manufacturers typically provide recommended photocuring techniques, including parameters like increment size or exposure time, which are derived from laboratory tests conducted under ideal conditions, often with the LCU tip positioned 0 mm away from the material surface (15). Nevertheless, when considering real clinical scenarios, such as restoring occlusal surfaces, the shortest achievable distance between the LCU tip and the first material increment for example, is approximately 3 mm, accounting for anatomical cusp height and cavity depth. For instance, the situation becomes more challenging when restoring proximal boxes, where the distance to the gingival floor can extend up to 8 mm (20). In such cases, the amount of incident light reaching the restorative material may decrease, potentially impacting its final properties (15, 19, 21, 24, 25, 26). Furthermore, certain LCU designs may make it impractical to achieve the recommended light-tip/material proximity, even on occlusal surfaces. Previous studies in this area have often utilized methodological setups that inadequately simulate realworld scenarios. Most of these studies evaluate the degree of conversion (DC) and related properties solely in the bottom increment (built alone). neglecting the actual process of constructing a resin-based composite (RBC) restoration using the incremental technique, and not considering the intermediate increments in this evaluation (27, 28, 29, 30). In this technique, residual irradiance from the curing process may reach the previous composite increment while curing the subsequent one, creating a cumulative effect. Furthermore, certain mechanical properties, such as flexural strength, are sometimes assessed by applying separate photocuring acts to build-up the specimen (12-25 mm length bars), introducing bias and deviating from clinical reality (31).

Given these considerations, there exists a knowledge gap concerning how variations in lighttip/material distances may affect the degree of conversion and related properties of nanohybrid RBCs at both their top and bottom surfaces, particularly when restoring cavities of varying depths, while considering the incremental technique's actual conditions. Therefore, this in vitro study aims to evaluate the influence of different cavity depth configurations and subsequent light-tip/ material distances on the biaxial flexural strength (BFS), degree of conversion (DC), and microhardness (MH) of a nanohybrid resin-based composite employing the incremental technique, considering both top and bottom surfaces as well, for the last two variables.

The null hypothesis posited is that cavity depth and subsequent variations in light-tip/ material distance will not significantly affect the degree of conversion and associated physical properties of a nanohybrid resin composite's top and bottom surfaces when employing the incremental technique.

MATERIALS AND METHODS

SAMPLE PREPARATION

To prepare samples, quadrangular and circled molds were designed (3D MatterControITM Design Program Version 2.20.1.10422) and 3D printed (DLP 3D printer MAX UV, Asiga, NSW Australia) employing a dental-specific 3D printing resin-based material (Freeprint® denture, Detax Ettlingen). Quadrangular molds were used to create RBCs' specimens (n=10) for the microhardness (MH) and degree of conversion (DC) tests. Three different dimensions were employed to simulate varying restoration depths: A) 2mm (4x4mm by 2mm in height), B) 4mm (4x4mm by 4mm in height) and C) 6mm (4x4mm by 6mm in height). Circular molds measuring 2mm (thick) by 8.5mm (diameter), were used for the biaxial flexural strength (BFS) test as seen in Figure 1. Additionally, 1mm thick discs and squares were built to work as spacers between the main mold and the LCU.

To assess MH (Vickers) and DC by means of Fourier Transformed Infrared Spectroscopy (FTIR), 30 nanohybrid resin composite specimens (material's characteristics shown in Table 1) were built-up, following the manufacturer's instructions for the incremental technique and the associated light-curing process (details provided below). These specimens were designed to simulate three different depths as well (n=10): 2, 4, and 6 mm (refer to Figure 2).

In the case of BFS test, the described circled molds were stacked one on top of the other, separated by a celluloid mylar band to achieve the desired thickness of the system according to each group depth (2, 4 or 6mm). Each increment (disc) was photopolymerized separately (in its adequate position within the stack), with the light curing unit positioned at the maximum top distance according to each group's requirements, and if applicable, maintaining each previous increment-disc on its position while photocuring the subsequent increments on top of the first one (and so on). This procedure closely mimicked a real incremental technique used in cavity restorations of varying depths (Figure 3). After completing the incremental technique process for each stack, only the discs at the very bottom (2, 4 and 6mm) were collected to be submitted to the BFS test (n=15). All other discs (in the middle, when applicable) were not considered for the BFS test.

Regarding the photopolymerization conditions, a regular clinical-use (not new) LCU (Valo corded, Ultradent, South Jordan, UT, USA) was used in standard mode delivering an irradiance of 875 mW/cm². Each increment was cured for 20 seconds (Radiant energy: 17.5 J/cm²), placing the previously designed 1mm spacer between the surface of the mold and the LCU.

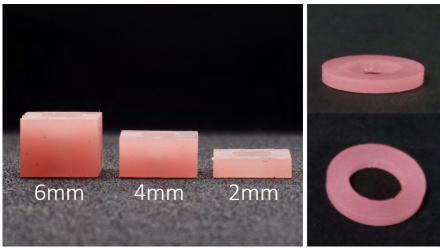


Figure 1. Molds (Die) for hardness and Degree of conversion cubical samples elaboration and discs for biaxial flexural strength test.

Commercial name	Manufacturer	Description	Information/composition	Batch number
Forma Zirconia Nano-Hybrid compo- site	Ultradent	Nanohybrid Resin based composite shade A3	Bis-GMA; Bis-EMA; TEGDMA; BHT; PEGDMA; UDMA; ytterbium trifluoride; fillers based on silane-treated ceramic, silane-treated silica, silane-treated silica-zirconium oxide, and barium glass. Filler size/content: 0.7 µm (mean particle size) (67 wt%; vol% not disclosed by manufacturer)	DOHAO
Valo corded curing light	Ultradent	Broadband spectrum curing lamp corded	Curing lamp with utilizable wavelength range: 385-515 nm; peak wavelengths: 395-415 nm and 440-480 nm. Lens size: 9.75mm. Irradiance: Standard power 1000 mW/cm ² (+/- 10%) High power plus 1400 mW/cm ² (+/- 10%) Xtra power 3200 mW/cm ² (+/- 20%) Power supply: Output 9VDC at 2A	

Table	1.	Materials	and	equipme	ent used	in	this	study	Ι.
-------	----	-----------	-----	---------	----------	----	------	-------	----

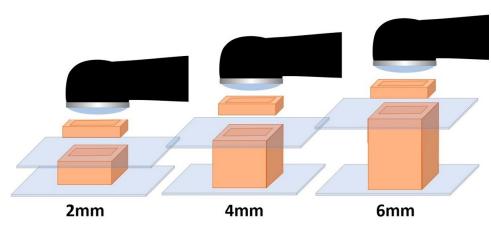


Figure 2. Micro hardness / Degree of conversion samples' preparation scheme, according to each depth group: 4x4x2 mm, 4x4x4 mm and 4x4x6 mm cubes, celluloid band on the external surface and the spacer (1mm thick) to place the lamp.

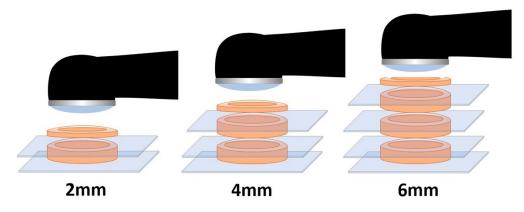


Figure 3. Biaxial flexural strenght samples' preparation scheme, according to each depth group: 8.5mm diameter by 2mm thick discs, stacking them one on top of the other until reaching the desired depth (4 or 6mm), each separated by a celluloid band and placing the 1mm thick spacer on top to place the lamp.

MICROHARDNESS EVALUATION (MH)

MH was analyzed employing a Vickers microhardness tester (Micromet 2001 durometer model 1600-4980 from Buehler®, IL USA). Three superficial indentations (200kgf for 15 seconds) were applied to each sample, both at the top and bottom surfaces. The indentations were subsequently measured with the help of the tester's attached microscope (in µm). The resulting data were recorded for the calculation of MH (VHN) following the guidelines outlined in ISO 6507-1³². After transforming data (Box-Cox, λ: 0.0233439) to meet parametric assumptions (Normality: Anderson-Darling (p=0.051), Homoscedasticity: Levene (p=0.062)), a two-way ANOVA with repeated measures in the "surface" factor (linked to the specimen) was conducted, followed by a Tukey post-hoc test (α =0.05).

BIAXIAL FLEXURAL STRENGTH (BFS)

For BFS assessment, a modified ball-onthree-balls biaxial flexural test configuration was employed. Discs were carefully positioned into a customized jig matching specimens' dimension. The entire setup was affixed to a universal testing machine (Electropuls E3000, Instron) operating at a crosshead speed of 5mm/min until the specimen failed. For each specimen, the dimensions and the corresponding failure load at the point of fracture (N) were meticulously collected and recorded in a data sheet. These values were later used to calculate the BFS (σ max) in MPa for each sample, following the methodology outlined by Staudacher *et al.* and the given formula (33):

a
$$\sigma max = f \frac{P}{t^2}$$

Where *f* is a dimensionless function considering materials' properties and its geometrical dimensions (28), P represents the failure load (N) and t, the specimen thickness (mm). As BFS data proved normality (Anderson-Darling, p=0.644) and homoscedasticity (Levene, p=0.105), they were statistically analyzed employing a 1-way ANOVA (α =0.05).

DEGREE OF CONVERSION (DC)

DC was evaluated using a Fourier Transformed Infrared Spectroscope (FTIR-ATR) (Nicolet 6700, Thermo Scientific). This system was equipped with a liquid-nitrogen-cooled MCT photovoltaic detector and operated at a resolution of 4 cm-1. A diamond crystal was utilized for ATR measurements. Spectra profiles spanning the range of approximately 4000 to 400 cm-1 were acquired from all samples, both, before and after polymerization on all surfaces (top and bottom) (n=5). All obtained spectra were subjected to analysis, with specific attention to identifying peaks corresponding to aliphatic methacrylate carbon-carbon double bonds (C=C, around 1640cm-1), and aromatic C=C double bonds (around 1610cm-1). The heights of these peaks were measured and meticulously recorded. The DC was calculated by assessing the difference in peak height of the absorbance intensity before and after light-curing:

 $\% DC = \left[1 - \frac{(1634 cm^{-1}/1608 cm^{-1}) Peak hight before light curing}{(1634 cm^{-1}/1608 cm^{-1}) Peak hight after light curing}\right]$

The obtained results were recorded in a data sheet, then statistically analyzed using a 2-way ANOVA (distance vs. surface, assessing repeated measures in the surface factor linked to specimen) and Tukey post-hoc test, after proving parametric assumptions (Normality: Anderson-Darling (p=0.493), Homoscedasticity: Levene (p=0.281)).

RESULTS

MICROHARDNESS EVALUATION (MH)

ANOVA revealed that the factor "distance" plus "surface/distance" interaction resulted statisti-

cally significant (p=0.004 and p<0.0001, respectively). All mean values, standard deviations, and statistical differences (Tukey, p<0.05) are displayed in Table 2.

Regarding the distance from the light tip to the resin-based material, 2mm group showed the highest results (99.41 ± 52.23 VHN), being not statistically different from the 4mm group (84.10 ± 15.74 VHN), and this last, not statistically different from the 6mm group (68.60 ± 18.69 VHN). When interacting both factors, group 2mm/bottom showed the highest values (126.94 ± 61.23 VHN), being not statistically different from 4mm/bottom group (89.2 ± 18.72 VHN) and this last being not statistically different from all other groups. 6mm/ bottom group obtained the lowest results among all (56.46 ± 18.07 VHN) (Table 2).

BIAXIAL FLEXURAL STRENGTH (BFS) AND DEGREE OF CONVERSION

ANOVA revealed that BFS data were not influenced by the light-tip/composite distance (p=0.194; 2mm: 120.96 MPa, 4mm: 131.56 MPa, 6mm group: 122.63 MPa).

Degree of conversion results revealed only statistical differences within the "surface" factor (p=0.001), showing statistically higher degree of conversion at the top surfaces ($47.74\pm9.67\%$) than at the bottom surfaces ($21.93\pm8.57\%$) (Table 3). "Distance" factor plus its interaction with the "surface" factor resulted not significant (p>0.05) according to 2-way ANOVA.

Surface/ Distance	2mm		n		4mm		n		6mm			n
Тор	71.89	±17.02	b	10	79.06	±10.72	b	10	80.78	±9.08	b	10
Bottom	126.94	±61.53	а	10	89.2	±18.72	ab	10	56.46	±18.07	b	10
Tukey "Distance"												
(p<0.05)	99.41	±52.23	А		84.1	±15.74	AB		68.6	±18.69	В	

Table 2. Vickers microhardness (VHN) mean values, standard deviations and group comparisons of
--

Different letters represent significant differences among groups (capital: factor CDS; lowercase: interaction).

Table 3. Degree of conversion (%) mean values, standard deviations and group comparisons outcomes.

Surface/ Distance	2mm		n	4mm		n	6mm		n	Tukey ("surface", p<0.05)
Тор	45.63	±5.63	10	52.13	±3.45	10	45.47	±13.83	10	47.74±9.67 A
Bottom	14.65	±11.12	10	26.06	±4.79	10	25.07	±4.26	10	21,93±8,57 B

Different letters represent significant differences among groups.

DISCUSSION

Performing efficient photopolymerization is essential to bestow the composite material with adequate physicomechanical properties. Inadequate photopolymerization can significantly impact the long-term prognosis of restorations, especia-Ily given the challenging conditions they face during both the operative procedure and functional use34. One notable challenge that clinicians routinely encounter is the variation in the distance between the light tip of the LCU and the composite material, especially at different depths within cavities. Literature is still not conclusive on how these varying distances may affect materials' properties (at the top/bottom surfaces) and the consequent success of a RBC restoration. This study thus sought to investigate this issue. Although microhardness (MH) and the degree of conversion (DC) were notably affected by certain variables such as distance and surface location. biaxial flexural strength (BFS) remained unaffected. As a result, the null hypothesis, was partially rejected. The findings from this study indicate that, with increasing cavity depth, light-tip/material distance and, consequently, restoration thickness from 2mm to 6mm, the microhardness of the RBC material exhibited in general, a significant reduction. Moreover, at this greater thickness (6mm), the bottom surfaces of the restoration displayed even lower MH values than their top surfaces.

De Mendoca Et Al³⁵ obtained similar results on Bulkfill composites (a reduction in MH as increasing the thickness of the restoration). Thus, it appears that the distance between the light tip and the composite, and the respective final thickness of the restoration, may exert a negative influence on the quality of RBCs' polymerization. In such cases, the light must travel such distance from the light tip to reach the edge of the increment, and penetrate through, to its bottom surface. This adverse impact seems to be directly proportional to the thickness of the increments and the light-tip/ composite distance. This phenomenon is evident considering the lower MH values observed at the bottom of the deeper cavity preparations (6mm group), likely attributable to a reduced crosslinking rate within the resulting polymeric network. One plausible explanation for this is an insufficient energy flux reaching the material at its final destination, leading to lower activation of the photoinitiators within the material (21, 26, 36, 37). These outcomes agree with Lima et al.(38) who established that distances greater than 6mm in interproximal molar boxes, could result in a 50% reduction in radiant exposure reaching their bottom region. Interestingly, an inverse scenario unfolded in the 2mm group, where greater microhardness (MH) values were recorded at its bottom surfaces. This phenomenon is possible, probably because the light beam at the very edge of the light tip, may produce lower effective energy compared to the portion of the light beam a couple of millimeters downstream, due to light scattering and reflection within the material, producing greater hardness at 2mm than at 0mm (39). This disparity persists even when the light must travel through the bulk of the material, as long as the increment remains thin (2mm). However, the results produced by this phenomenon may vary significantly (Table 2), as thinner samples may exhibit greater variability in light distribution, leading to non-uniform polymerization and hardness. On the other hand, with thicker materials (e.g., 6mm), although light distribution may be more uniform in some circumstances, the continuity of light is likely disrupted while traveling through a thicker bulk of material, resulting in reduced energy reaching the material's bottom and subsequently leading to such decline in properties (39).

The current BFS outcomes did not reveal any statistically significant differences between the study groups. Similar results were obtained in another study (40), where also no statistical differences were observed in micro-flexural strength among various nanohybrid RBC's at 0, 2, and 8 mm light-tip/material distances. However, contrasting results were reported by EI-Askary *et al.* (41), who observed significantly higher values at shorter light-tip/material distances (0, 2, 4, and 6mm). Probably, variations in study setup may have contributed to the diverse outcomes reported in the literature on this topic. One key factor that might influence these discrepancies is the choice of the flexural strength testing setup. Most studies adhere to ISO standard 4049 (31), which suggests a uniaxial 3-point bending setup to test 25mm-long bars. However, some concerns have been raised regarding this specimen dimension, as it necessitates multiple photopolymerization cycles to complete each specimen. Therefore, when testing variables related to photopolymerization protocols, this approach may produce biased results, reason why alternative flexural strength testing setups have been proposed (biaxial approach) as more suitable and less variable alternatives for evaluating the flexural strength of RBCs (22, 42, 43).

Related to this biaxial flexural strength approach, an innovative ball-on-three-balls test was employed (33) as it allows the construction of each specimen in a single polymerization cycle, closely mimicking a real clinical scenario in an incremental technique. Another particularity in the current experiment setup (Figure 3), is that the bottom increment was not removed while photopolymerizing the upper increments to complete stacks of 4mm and 6mm. This design was intentional to simulate the actual amount of energy reaching the bottom portion of the restoration in such scenarios (2, 4, and 6mm light-tip/ material distances), trying to mirror the real clinical process. In clinical practice, these bottom portions typically receive residual energy while placing the upper increments to achieve the full thickness. This setup may have resulted in a consistent amount of energy reaching the bottom discs of all groups' stacks, potentially compensating for any negative effects of distance and consequently producing similar BFS values within the groups. This compensatory effect is likely achievable when using a high-quality light-curing unit characterized by uniform beam distribution and minimal light dispersion, as the one employed in this study.

Interestingly, microhardness (MH) and biaxial flexural strength (BFS) did not demonstrate identical trends in the present study, contrary to the most commonly reported outcome in previous studies (27, 28, 29, 30). A significant reduction in MH was observed at the bottom surfaces of the 6mm group compared to 2mm group. This discrepancy can be attributed to the nature of both tests. MH involves testing a specific surface at a very localized spot, leading to high variability in the compositional structures of the resin-based composite (RBC) (matrix, fillers, etc.) present at that specific testing site. Consequently, this test may be more likely to evaluate the independent responses of individual components or at least not all acting together at each indentation location, resulting in variable outcomes. In contrast, BFS assesses the mechanical behavior of the integral bulk of the material, where all the components of the RBC are a unified entity. This characteristic is likely the reason why the BFS results were less influenced by changes in distance or decreases in light irradiance and their potential effects on the organic matrix.

Although both variables appeared to have some impact on the properties of the materials, the extent of this effect could have been worse, given previous literature reports (38, 44, 45). Specifically, concerning the distance between the light tip and the material, only a 6mm distance produced a significant negative effect on material properties (MH), and only at the bottom surfaces of the specimens. This suggests that distance alone does not have a substantial impact, at least under the current study conditions, as no differences were observed in MH between groups for the top surfaces. Therefore, it appears that the combination of a sufficiently large distance (e.g., 6mm) and the thickness of the increment (starting from 2mm) represents a critical scenario for negatively affecting material properties. It is essential to emphasize that these results were obtained using a highquality light-curing unit (LCU), and it is plausible that this situation may worsen if a lower quality LCU is used.

Physical properties such as MH and BFS are considered indirect methods for assessing the degree of conversion (DC) of RBCs, as a direct approach involves spectroscopic analysis to evaluate the change in the relative amount of C=C functional groups before and after photopolymerization (22). Therefore, this methodological approach was also employed in this study. The results revealed a significant difference in DC between the top and bottom surfaces of the simulated restorations, regardless of the lighttip/material distance used. Previous studies have suggested that increasing the light-tip/material distance and the final thickness of the restoration may lead to a reduction in the irradiance received by the bottom surface (around 91%), potentially affecting RBC DC and hardness (21). However, it has been suggested that light may encounter more obstacles when passing through a solid material (such as uncured RBC, based on penetration coefficients) than when traveling a certain distance without facing solid obstacles along the way (38). When working incrementally in a deep preparation, the light-curing unit is initially positioned at a greater distance from the first increment. As subsequent increments are applied, the restorative material itself serves as a physical barrier to light transmission, particularly at greater distances such as 6mm. However, this effect may not be as pronounced at shorter distances such as 2mm and 4mm, as previously indicated for MH. This can make it challenging to achieve the desired physicomechanical properties at the bottom of the restoration at 6mm. Therefore, it is reasonable to assume that within this distance, passing through the thickness of the restorative material poses a greater hindrance to irradiance transmission than the light-tip/material distance itself. However, it is important to remember that besides being considered a direct method, the DC calculation method is performed based on a "relative number of monomers converting" (not an absolute number of monomers), as spectroscopic devices capture a signal received from the vibrations of the molecules present within the material's surface, produced by the incidence of the infrared light beam. This is why this method could also show some inconsistencies within the results reported in the literature, and thus, may not exactly match the same trend as the results derived from physical properties, as observed here.

Our results confirm some information previously found in the literature on this subject; however, new insights were obtained regarding the role of the distance from the light tip to the material. Typically, the literature has suggested that the most favorable scenario is to photopolymerize the material at the shortest possible distance without contacting it, and that on this closest surface, the material will exhibit the best properties, which are subsequently affected as the distance increases (27, 28, 29, 30). However, in this case, the current results indicate that this does not always apply. Even when applying a standardized additional distance of 1mm between the active tip of the lamp and the material for all groups (2, 4, and 6mm), higher microhardness values on the top surfaces of the samples were not consistently obtained. In the 2mm and 4mm groups, higher microhardness was observed on the bottom surfaces of the samples, suggesting that the polymerizing effect of light might be better over a certain short distance, even when it passes through the material. Additionally, in the case of the 4mm group, the additional residual irradiation captured by the bottom increment, resulting from photopolymerizing the subsequent increment on top of it, may have contributed to these results. However, this did not occur with the 6mm group, indicating that possibly this distance is indeed significantly detrimental, and this residual irradiation does not have a positive effect on the bottom surface from this distance (6mm) to further. This would suggest that the positive effect of a certain distance to make the light more efficient in activating polymerization (described above) has a saturation point between 4 and 6mm, beyond which the effect would be rather negative.

Some limitations of this *in vitro* study can be listed as follows. First, the specimens were not subjected to aging processes (thermal/mechanical), as the primary aim of this initial study was to isolate the effects of the variables under investigation. Second, it was not possible to conduct biaxial flexural strength (BFS) tests separately on the top and bottom surfaces of each increment, as was done for MH and DC. Third, 3D-printed composite geometrical molds were used to simulate cavity preparations instead of real teeth. Fourth, these results can only be extrapolated to this specific material and its specific shade (A3). In this regard, the use of less saturated shades may facilitate light transmission, thereby potentially improving microhardness (MH), degree of conversion (DC), and bond strength (BFS) compared to the current results. Lastly, a single LCU was employed. Future studies in this research area should consider additional variables, such as specimen aging, LCU angulation, irradiance measurements under all simulated scenarios, and the use of multiple LCUs to assess if they produce consistent results, among other factors.

CONCLUSIONS

In light of the current evidence and despite the limitations of this in vitro study, the following conclusions can be drawn. The cavity depth and light-tip/material distance negatively affected polymerization quality (microhardness). This adverse effect was particularly pronounced on the bottom surfaces at 6 mm distance. For 2 and 4 mm distances, polymerization quality remained adequate on both the top and bottom surfaces; however, the degree of conversion was lower in general for the bottom surfaces. Nonetheless, no significant impact on the degree of conversion or flexural strength was observed based on the distance from the light tip, indicating that some physical properties and degree of conversion may not always be proportional. The current outcomes may only be related to the specific photopolymerization conditions reported here.

CONFLICT OF INTERESTS

There are no conflict of interests. The authors do not have any financial interest in the companies whose materials are included in this article.

CLINICAL RELEVANCE STATEMENT

The current outcomes suggest that polymerization quality of an incrementally-built restoration of a nanohybrid resin composite was adequate when the cavity bottom is located 2 or 4 mm away from the light tip. However, a 6 mm distance showed a detrimental effect on the polymerization quality of the studied material under the employed conditions. In such case, an additional polymerization cycle may be necessary.

AUTHOR CONTRIBUTION STATEMENT

Conceptualization and design: F.M.G., J.F.R.T. Literature review: J.F.R.T., F.M.G. Methodology and validation: J.F.R.T., F.M.G. Formal analysis: J.F.R.T., F.M.G. Investigation and data collection: J.F.R.T. Data analysis and interpretation: F.M.G. Writing-original draft preparation: J.F.R.T. Writing-review & editing: F.M.G.

REFERENCES

- 1. Mulligan S., Hatton P.V., Martin N. Resinbased composite materials: elution and pollution. Br Dent J. 2022; 232 (9): 644-52.
- 2. Bellinaso M.D., Soares F.Z.M., Rocha R. de O. Do bulk-fill resins decrease the restorative time in posterior teeth? A systematic review and meta-analysis of in vitro studies. J Investig Clin Dent. 2019; 10 (4).
- Illie N., Hickel R. Resin composite restorative materials: Composites. Aust Dent J. 2011; 56 (Suppl 1): 59-66.
- Zhou X., Huang X., Li M., Peng X., Wang S., Zhou X., et al. Development and status of resin composite as dental restorative materials. J Appl Polym Sci. 2019; 136 (44).
- Stein P.S., Sullivan J., Haubenreich J.E., Osborne P.B. Composite resin in medicine and dentistry. J Long Term Eff Med Implants. 2005; 15 (6): 641-54.
- Zimmerli B., Strub M., Jeger F., Stadler O., Lussi A. Composite materials: composition, properties and clinical applications. A literature review. Schweiz Monatsschr Zahnmed. 2010; 120 (11): 972-86.
- Aminoroaya A., Esmaeely Neisiany R., Nouri Khorasani S., Panahi P., Das O., Ramakrishna S. A review of dental composites: Methods of characterizations. ACS Biomater Sci Eng. 2020; 6 (7): 3713-34.
- Shinkai K., Taira Y., Suzuki S., Kawashima S., Suzuki M. Effect of filler size and filler loading on wear of experimental flowable resin composites. J Appl Oral Sci. 2018; 26 (0).
- 9. Rebholz-Zaribaf N., Özcan M. Adhesion to zirconia as a function of primers/silane

coupling agents, luting cement types, aging and test methods. J Adhes Sci Technol. 2017; 31 (13): 1408-21.

- 10. Ferracane J.L. Resin composite-State of the Art. Dent Mater. 2011; 27 (1): 29-38.
- Randolph L.D., Palin W.M., Leloup G., Leprince J.G. Filler characteristics of modern dental resin composites and their influence on physico-mechanical properties. Dent Mater. 2016; 32 (12): 1586-99.
- Bompolaki D., Lubisich E.B., Fugolin A.P. Resin-based composites for direct and indirect restorations. Dent Clin North Am. 2022; 66 (4): 517-36.
- 13. Price R.B.T. Light curing in dentistry. Dent Clin North Am. 2017; 61 (4): 751-78.
- Fugolin A.P.P., Pfeifer C.S. New resins for dental composites. J Dent Res. 2017; 96 (10): 1085-91.
- Al-Zain A.O., Eckert G.J., Platt J.A. The influence of distance on radiant exposure and degree of conversion using different light-emitting-diode curing units. Oper Dent. 2019; 44 (3): E133-44.
- Rueggeberg F.A. State-of-the-art: Dental photocuring-A review. Dent Mater. 2011; 27 (1): 39-52.
- Strazzi-Sahyon H., Rocha E., Assunção W., dos Santos P. Influence of light-curing intensity on color stability and microhardness of composite resins. Int J Periodontics Restor Dent. 2020; 40 (1): 129-34.
- Alkhudhairy F. Wear resistance of bulkfill composite resin restorative materials polymerized under different curing intensities. J Contemp Dent Pract. 2017;18: 39-43.
- Felix C.A., Price R.B.T. The effect of distance from light source on light intensity from curing lights. J Adhes Dent. 2003; 5 (4): 283-91.

- Durner J., Obermaier J., Draenert M., Ilie N. Correlation of the degree of conversion with the amount of elutable substances in nanohybrid dental composites. Dent Mater. 2012; 28 (11): 1146-53.
- 21. AlShaafi M.M. Factors affecting polymerization of resin-based composites: A literature review. Saudi Dent J. 2017; 29 (2): 48-58.
- 22. Ferracane J.L., Hilton T.J., Stansbury J.W., Watts D.C., Silikas N., Ilie N., et al. Academy of Dental Materials guidance— Resin composites: Part II—Technique sensitivity (handling, polymerization, dimensional changes). Dent Mater. 2017; 33 (11): 1171-91.
- Abidin T., Dennis D., Siagian J.S., Ikhsan T. Effect of different LED light-curing units on degree of conversion and microhardness of bulk-fill composite resin. J Contemp Dent Pract. 2020; 21 (6): 615-20.
- 24. Eshmawi Y.T., Al-Zain A.O., Eckert G.J., Platt J.A. Variation in composite degree of conversion and microflexural strength for different curing lights and surface locations. J Am Dent Assoc. 2018; 149 (10): 893-902.
- 25. Price R.B.T., Labrie D., Whalen J.M., Felix C.M. Effect of distance on irradiance and beam homogeneity from 4 light-emitting diode curing units. J Can Dent Assoc. 2011; 77: b9.
- 26. Shortall A.C., Price R.B.T., MacKenzie L., Burke F.J.T. Guidelines for the selection, use, and maintenance of LED light-curing units – Part II. Br Dent J. 2016; 221 (9): 551-4.
- 27. Catelan A., de Araújo L.S.N., da Silveira B.C.M., Kawano Y., Ambrosano G.M.B., Marchi G.M., et al. Impact of the distance of light curing on the degree of conversion and microhardness of a composite resin. Acta Odontol Scand. 2015; 73 (4): 298-301.

- 28. Oh S., Kim H.J., Kim H.J., Antonson S.A., Kim S.Y. Influence of irradiation distance on the mechanical performances of resin composites polymerized with high-irradiance light curing units. Biomater Res. 2022; 26 (1).
- Gonçalves L., Amaral C.M., Poskus L.T., Guimarães J.G.A., Silva E.M. Degradation of resin composites in a simulated deep cavity. Braz Dent J. 2014; 25 (6): 532-7.
- 30. Faria-e-Silva A.L., Fanger C., Nguyen L., Howerton D., Pfeifer C.S. Impact of material shade and distance from light curing unit tip on the depth of polymerization of composites. Braz Dent J. 2017; 28 (5): 632-7.
- International Organization for Standardization. Dentistry-Polymer based restorative materials, International standard ISO 4049; 5th Edition 2019-05.
- 32. International Standard ISO. Metallic materials-Vicker's hardness test-Part 1: test method (ISO 6507-1: 2018).
- Staudacher M., Lube T., Supancic P. The Ballon-Three-Balls strength test for discs and plates: Extending and simplifying stress evaluation. J Eur Ceram Soc. 2023; 43 (2): 648-60.
- McNaught A., Wilkinson A. IUPAC compendium of chemical terminology. Int Union Pure Appl Chem. 1997; 19: 458-65.
- 35. de Mendonça B.C., Soto-Montero J.R., de Castro E.F., Pecorari V.G.A., Rueggeberg F.A., Giannini M. Flexural strength and microhardness of bulk-fill restorative materials. J Esthet Restor Dent. 2021; 33 (4): 628-35.
- 36. El-Askary F.S., Botros S.A., Nassif M.S.A., Özcan M. Flexural strength of nano-hybrid resin composite as a function of light attenuation distance and specimen dimension. J Adhes Sci Technol. 2017; 31 (5): 520-9.
- 37. Musanje L., Darvell B.W. Polymerization of resin composite restorative materials:

exposure reciprocity. Dent Mater. 2003; 19 (6): 531-41.

- 38. Lima R.B.W., Murillo-Gómez F., Sartori C.G., De Góes M.F. Effect of light absence or attenuation on biaxial flexural strength of dual-polymerized resin cements after shortand long-term storage. J Esthet Restor Dent. 2019; 31 (1): 80-7.
- Jandt K.D., Mills R.W. A brief history of LED photopolymerization. Dent Mater. 2013; 29 (6): 605-17.
- 40. Al-Zain A.O., Marghalani H.Y. Influence of light-curing distances on microflexural strength of two resin-based composites. Oper Dent. 2020; 45 (3): 297-305.
- El-Askary F.S., El-Korashy D.I. Influence of shade and light-curing distance on the degree of conversion and flexural strength of a dualcure core build-up resin composite. Am J Dent. 2012; 25 (2): 97-102.
- 42. Rueggeberg F.A., Cole M.A., Looney S.W., Vickers A., Swift E.J. Comparison of manufacturer-recommended exposure durations with those determined using biaxial flexure strength and scraped composite thickness among a variety of light-curing units. J Esthet Restor Dent. 2009; 21 (1): 43-61.
- 43. Özcan C., Lestriez P., Berry-Kromer V., Thiebaud F., Sockalingum G.D., Untereiner V., et al. Misinterpretation of ISO 4049 standard recommendations: Impact on Young's modulus and conversion degree of dental composites. J Mech Behav Biomed Mater. 2020; 110: 103947.
- McNaught A., Wilkinson A. IUPAC compendium of chemical terminology. Int Union Pure Appl Chem. 1997; 19: 458-65.
- 45. Haifa B. Led Dental photopolymerization. A literature review. Int J Med Dent. 2021; 25: 53-62.