


EVALUATION OF FLEXURAL STRENGTH AND MICROHARDNESS OF A PHOTOCURATED MICROHYBRID RESIN IN DIFFERENT IRRADIANCES <https://doi.org/10.56238/sevened2024.034-010>

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ABSTRACT

Physical-mechanical evaluation tests provide data on the stiffness, resilience and hardness of materials, as well as allow the association of these characteristics with monomeric conversion factors, an important agent that will directly influence the optimization of aesthetic and functional results. This pilot study sought to evaluate a microhybrid composite resin, used in class I and II dental restorations, through the Knoop physical-mechanical tests of flexural strength and microhardness. This provided relevant data on the material's ability to withstand deformation forces and its degree of conversion according to different photocuring methods. For the study, Fill Magic – Vigodent composite resin was used. A total of 9 specimens were made, divided into 3 groups according to the photoactivation mode. The polymerization methods used were the STANDART and HIGH modes. Photoactivation was performed in the center of the samples. The specimens were fixed in a metal device, coupled to the universal testing machine and submitted to the three-point flexural strength test. The force was applied to the center of the specimen at a speed of 1mm/min with a load cell of 500 N, until the specimen failed completely. Then, the same specimens submitted to the flexion test were evaluated in the Knoop microhardness test. The results obtained indicate that the photocuring protocol, especially the exposure time and the proximity of the

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tip, directly influences the mechanical strength and hardness of the material, with better results observed in the groups with longer photocuring time.

Keywords: Mechanical Tests. Flexural Strength. Hardness Tests. Composite Resins.

INTRODUCTION

The verification of the performance of dental materials under clinical conditions enables a prediction of the material's ability to resist deformation under load, abrasion resistance and the integrity capacity of the material's surface, which are fundamental parameters for the longevity of restorations (Heintze et al, 2017).

Physical-mechanical evaluation tests provide data on the stiffness, resilience and hardness of materials, as well as allow the association of these characteristics with monomeric conversion factors, an important agent that will directly influence the optimization of aesthetic and functional results (Heintze et al., 2011; Opdam et al., 2014).

Dental restorative/prosthetic material must present functional mechanical integrity in the oral environment, from the postoperative moment to long periods that represent the longevity of rehabilitation (Ilie N et al., 2017). To identify the minimum expected strength values in a resinous material, the flexure test develops tensile, compression, and shear stresses during its execution, which represents a strong scientific basis for clinical fracture correlations of composite resin restorations, and can guide the dentist regarding the clinical wear of the material (Heintze et al., 2017).

In this regard, the most common and highly reliable flexure test to assess the modulus of elasticity of a composite resin specimen is the three-point loading mode, according to ISO 4049 (ISO, 2019). The elastic modulus in a three-point flexure test has a good correlation with the indentation modulus in the evaluation of the strength of a resin composite, and this analysis is performed through a hardness device, which forces an indenter on the surface of the sample and the result of the test is the relationship between the force and the relevant indentation depth (Oliver, 1992; Cardoso, 2023). In this way, Knoop microhardness measures the hardness of the surface of the composite, which can be directly correlated to its degree of conversion, since it indicates not only the mechanical properties, but also the mode of chemical degradation of the material.

Therefore, this pilot study sought to evaluate a microhybrid composite resin, used in class I and II dental restorations, through Knoop's physical-mechanical tests of flexural strength and microhardness. Providing relevant data on the material's ability to withstand deformation forces and its degree of conversion according to different photocuring methods.

MICROHYBRID COMPOUND RESIN

Microhybrid composites are widely classified as "universal composites" due to their versatility, allowing use in both anterior and posterior restorations. This classification is

based on its optimized combination of mechanical strength and polishing capacity, meeting aesthetic and functional demands (Ferracane., 2011). Microhybrid composites are formulated with mixed loading systems, incorporating microfine particles with sizes ranging from 0.4µm, aiming at optimizing surface smoothness compared to smaller particle composites, while preserving their desirable mechanical properties. Thus, these materials are often classified as general-purpose composites, suitable for the restoration of areas with high mechanical stress, in which aesthetic considerations are fundamental (Rawls., 2013).

The composition of these materials is based on colloidal silica and ground glass particles that contain heavy metals, resulting in an inorganic content of approximately 75 to 80% by weight. Glass particles have an average size of 0.4 to 1.0 µm, with a consistent trend of particle size reduction as improvements are integrated into formulations. It is estimated that about 75% of the ground particles are less than 1.0 µm, while colloidal silica accounts for between 10 to 20% of the total weight of the filler content. This combination of components contributes to the superior mechanical and aesthetic properties of microhybrid resins in dental applications (Anusavice; Shen; Rawls., 2013).

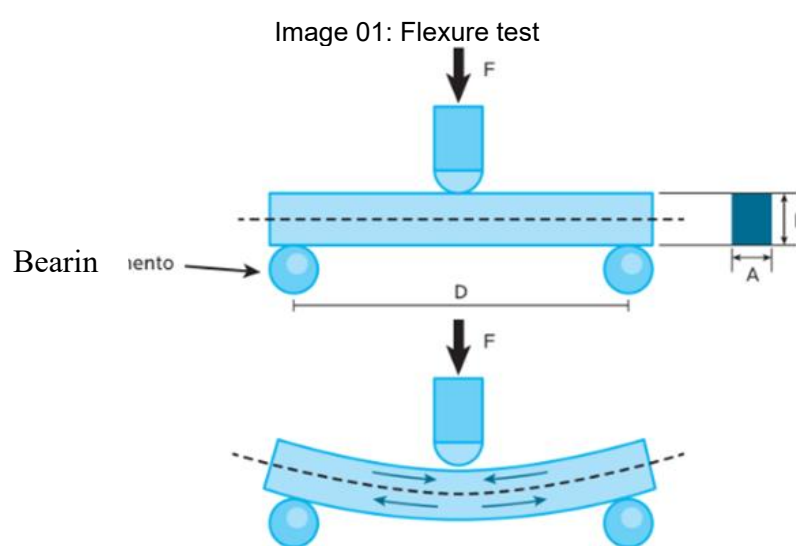
BENDING TEST

Flexural strength is an intrinsic property of materials, defined as the maximum stress supported by a specimen immediately before failure during the application of a flexural test. This parameter, often referred to as transverse breaking strength, flexural strength, or modulus of rupture, is widely used to characterize the mechanical performance of materials under flexing-inducing loads. The transverse flexure test, performed on specimens with standardized geometry, usually with a circular or rectangular cross-section, allows for a precise evaluation of the material's ability to resist flexural stresses before reaching the breaking point. (Plotinus *et al.*, 2007; Novais *et al.*, 2009; Thomas *et al.*, 2015; Janani *et al.*, 2022.)

The flexure test evaluates the strength of a bar resting on the ends under static load to quantify the energy that the material can absorb without fracture. If the flexural strength of the material is not sufficient to withstand chewing stresses, there is a higher risk of fracture. Most materials fail under tensile stress before failing under compressive stress; therefore, the maximum value of tensile stress that can be supported before the specimen fails represents its flexural strength. This test serves as a predictor of the mechanical behavior of a clinically used resin composite (Braem *et al.*, 1994; Anusavice., 2013; Calheiros *et al.*, 2013; Dathan *et al.*, 2023).

As established in the international standard ISO 4049, the procedure for evaluating the flexural strength of dental composites requires the preparation of rectangular specimens in the form of bars, with dimensions of $10 \times 2 \times 2 \text{ mm}^3$, to perform the three-point flexure test. During load application, specimen deformation under bending results in tensile stresses on the lower convex surface, which are potentially responsible for the initiation of the failure mechanism. This analysis allows a better understanding of the mechanical properties of dental composites under conditions of clinical stress (Darvell., 2002).

In the three-point flexure test, the rectangular specimens are positioned on two lower supports of a universal testing machine. The application of force occurs at a single upper point, situated at the midpoint of the sample, which results in an even load distribution. This experimental configuration allows the concentration of the load in the center of the specimen, where the maximum deflection and maximum stress are established, thus facilitating the evaluation of the mechanical properties of the material in relation to its flexural strength. This methodology is crucial for the characterization of the performance of materials in clinical applications (Chain, 2013). ISO 4049 standards for resins and ISO 6872 for ceramics.



Fonte: Chain, *et al.* 2023

MICROHARDNESS TESTING

Among the various types of *in vitro tests* commonly used to simulate the stresses that act on the tooth-restoration complex during the masticatory act, the microhardness test is one of the most performed, as it is able to predict whether the restorative material will resist the wear and tear resulting from the oral environment (Benetti *et al.*, 2011; De Mendonça *et al.*, 2021). This aspect is of paramount importance in dentistry, since surface

behavior is related to long-term clinical efficiency (Moraes *et al.*, 2008; Bragança *et al.*, 2020).

Hardness is observed to be an important surface property and is defined as the resistance of the material to permanent indentation or penetration under the application of a stress. This method can be performed in a simple and effective way, and can be indicated indirectly, to evaluate the degree of conversion of low-weight molecules (monomers) into high-weight molecules (polymers) of resinous materials (Lee; Lai; Hsu, 2002; Schoeffel *et al.*, 2020). This degree of conversion causes the light from the curing unit to attenuate as it passes through the material, where the depth of curing can be analyzed (Shimokawa *et al.*, 2017; De Mendonça *et al.*, 2021).

Microhardness data for a specific material provides information about its wear, polishing, and abrasive effect on antagonist teeth (Marovic *et al.*, 2013). Microhardness is strictly related to the compositional characteristics of the materials evaluated, and is influenced by aging, water absorption, and reactions that occur on the surface of the material (Prabhakar; Swamp; Basappa, 2010; Colombo *et al.*, 2019).

The Knoop and Vickers microhardness tests measure the microhardness of dental materials, they are used to evaluate the quality of the polymerization process of composite resins (Schneider *et al.*, 2016). These tests are applied to produce microscopic indentations, their application consists of penetrating an indenter in the shape of an elongated pyramid on a flat surface (Knoop) and in the shape of a pyramid with a square base (Vickers) (Anusavice; Shen; Rawls, 2013). These can provide a good determination of the resistance to plastic deformation generated by the application of a stress (Deniz Arisu *et al.*, 2018; Colombo *et al.*, 2019).

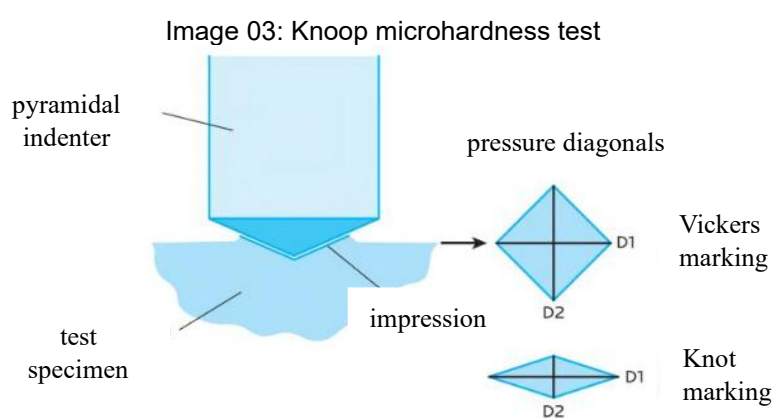
Materials with low surface hardness are more susceptible to roughness and this can compromise the fatigue strength of the material and cause premature failure of the restoration (Rodrigues *et al.*, 2010).

The specimens are indicated to be made based on the International Organization Standardization (ISO), it is an organization that aims to promote standardization in order to contribute to scientific research. Based on ISO 4049/2019 - *Dentistry-Polymer-Based Restorative Materials*, it is recommended that the specimens be made and tested at $23 \pm 2^\circ$ (except if the manufacturer's guidance is otherwise) with relative humidity control in order to remain greater than 30% and less than 70%. If the material is refrigerated for storage, it should be waited for it to be at the temperature mentioned above, before the test.

Light, both natural and artificial, is capable of triggering the activation of resinous materials, in an attempt to avoid this, the test should be performed in a dark environment

with any artificial light filtered by a yellow filter (ISO 4049/2019 *Dentistry-Polymer-Based restorative Materials*).

When fully cured specimens are required for testing, it is important to observe and ensure that they are homogeneous after removal from the mold. There should be no cracks, voids, discontinuities, or air inclusions present when viewed without magnification. The photopolymerization will be done based on the manufacturer's recommendations, it is important to make sure that the external light source is in a satisfactory operating condition. It is recommended by ISO that the curing depth of composite resins should not be less than 1 mm if they are labeled as opaque by the manufacturer, or not less than 1.5 mm for other restorative materials (ISO 4049/2019 *Dentistry-Polymer-Based restorative Materials*).



Fonte: Chain, *et al.* 2023

MATERIAL AND METHODS

This is a pilot study for the evaluation of a microhybrid composite resin, using physical-mechanical tests of flexural strength and Knoop microhardness.

SAMPLE

For the study, Fill Magic - Vigodent composite resin was used (Table 01).

Table 01 - Composition of the study material

MATERIAL	BRAND	MAKER	MONOMERS	INORGANIC CARGO (% by weight)	OTHER COMPONENTS
Fill Magic	Vigodent	Vigodent	Bis-GMA, Up to EMA, UDMA, TEGMA	<ul style="list-style-type: none"> • 5.0 µm charge • 75% 	<ul style="list-style-type: none"> • Photoinitiator • Pigments

Source: VIGODENT INDÚSTRIA E COMÉRCIO LTDA.

A total of 9 specimens were made, divided into 3 groups according to the mode of photopolymerization (Table 02).

Table 02 - Division of groups

GROUP	MATERIAL	N SAMPLE	PHOTOPOLYMERIZATION MODE
Group 1	Fill Magic	3	<ul style="list-style-type: none"> • STANDARD • 20 seconds • Tip leaning against the specimen
Group 2	Fill Magic	3	<ul style="list-style-type: none"> • HIGH • 6 seconds • Tip leaning against the specimen
Group 3	Fill Magic	3	<ul style="list-style-type: none"> • HIGH • 6 seconds • Tip at a distance of 10mm from the specimen

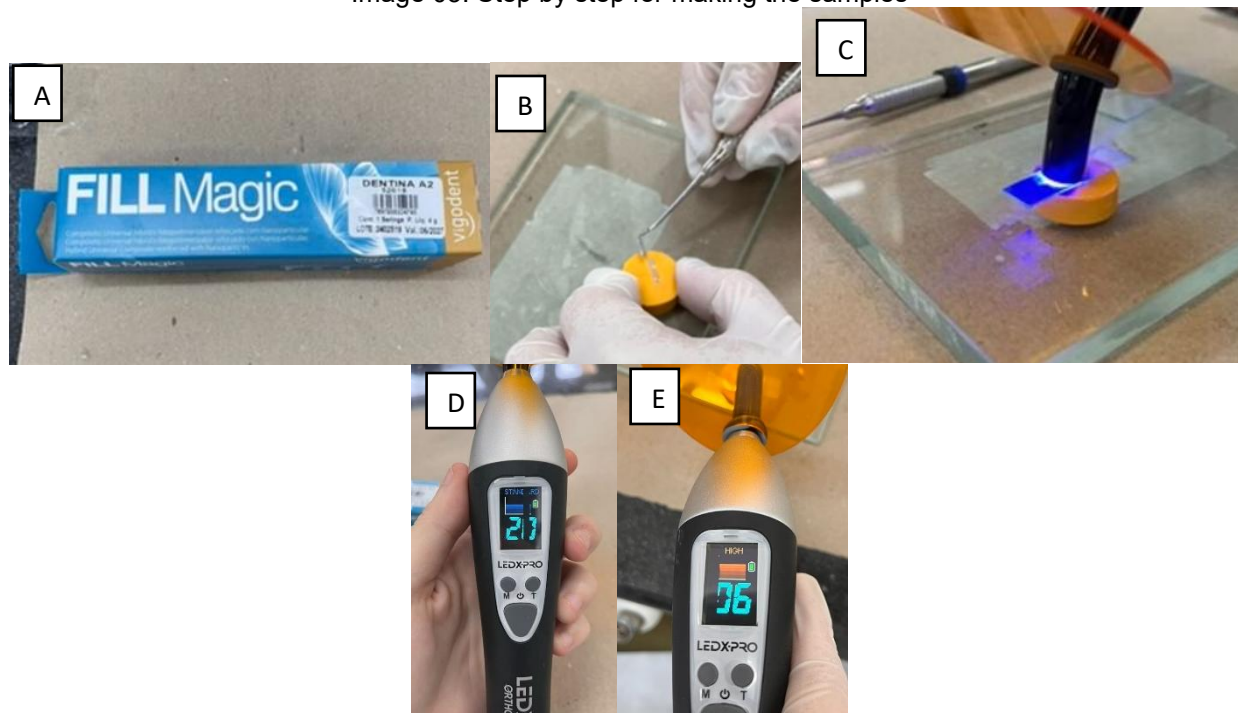
Source: Authors, 2024.

MAKING OF SAMPLES

Composite resin samples were prepared in a 2x2x10 mm filler silicone matrix with Fill Magic composite resin (Vigodent). The resin increments were dispensed into the matrix by means of a number 1 spatula, and the matrix was placed on a glass plate, while in the upper portion, polyester tape was placed over the matrix and covered with a glass sheet, ensuring superficial smoothness for the side of the composite resin that was light-cured (Image 02).

The samples were light-cured with the LED X Pro-Orthometric light-curing device. The polymerization methods used were *the STANDART* and *HIGH* modes. Photopolymerization was performed in the center of the samples.

Image 03: Step by step for making the samples



Source: Authors, 2024. Legend: 3A: Fill Magic composite resin (Vigodent) used to make the specimens. 3B: Preparation of specimens in addition silicone matrix with dimensions 2x2x10mm. 3C: Sample light-curing protocol with a near-tip tip. 3D: *STANDART* mode for 20 sec. 3E: *HIGH* mode for 6s.

The specimens were fixed in a metal device, coupled to the universal testing machine (INSTRON - 4411), and submitted to the three-point flexural strength test (Image 4). The force was applied to the center of the specimen at a speed of 1mm/min with a load cell of 500 N, until the specimen failed completely. Then, the same specimens submitted to the flexion test were evaluated in the Knoop microhardness test (Image 05).

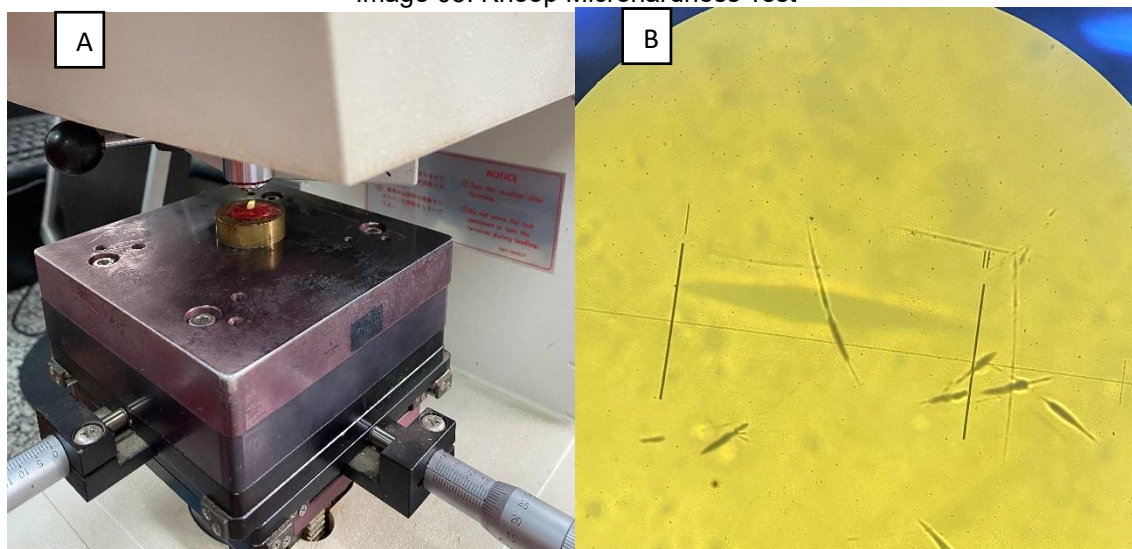
Image 04: Mechanical flexural test on INSTRON machine - 4411



Source: Authors, 2024.

The knoop microhardness test was performed on specimens that underwent the flexural strength test.

Image 05: Knoop Microhardness Test



Source: Authors, 2024. Legend: 5A: Knoop microhardness testing machine. 5B: Microscopic view of Knoop's indentation.

RESULTS AND DISCUSSION

The results of the flexion tests were tabulated in Excel spreadsheets for comparison of the values between the groups (Tables 03, 04, 05 and 06).

Table 03: Flexure test of Group 01 (Composite resin photocured for 20 s, with "STANDART" mode and tip close to the sample).

Sample	Flexural strength (MPa)	Automatic Young's Modulus of Elasticity (GPa)	Maximum Load (N)	Thickness (mm)	Width (mm)
1	213,78	3,68	-181,10	2,20	2,10
2	207,93	3,12	-174,20	2,30	1,90
3	223,32	4,28	-171,10	2,20	1,90
Average	215,01	3,69	-175,47	2,23	1,97
DP	7,77	0,58	5,12	0,06	0,12

Table 04: Table 02: Flexure test of Group 02 (Composite resin photoactivated for 6s, with "HIGH" mode and tip close to the sample).

	Flexural strength (MPa)	Automatic Young's Modulus of Elasticity (GPa)	Maximum Load (N)	Thickness (mm)	Width (mm)
1	185,49	3,11	-143,20	2,10	2,10
2	164,46	3,89	-108,80	2,10	1,80
3	208,50	4,47	-125,10	2,00	1,80
Average	186,15	3,82	-125,70	2,07	1,90
DP	22,03	0,68	17,21	0,06	0,17

Table 05: Flexure test of Group 03 (Composite resin photocured for 6s, with "HIGH" mode and tip at a distance of 10mm from the sample)

	Flexural strength (MPa)	Automatic Young's Modulus of Elasticity (GPa)	Maximum Load (N)	Thickness (mm)	Width (mm)
1	147,54	1,71	-107,10	2,20	1,80
2	139,60	1,75	-107,00	2,20	1,90
3	127,84	2,05	-84,56	2,10	1,80
Average	138,33	1,84	-99,55	2,17	1,83
DP	9,91	0,19	12,98	0,06	0,06

Table 06: Mean and standard deviation of each group

	Group 01	Group 02	Group 03
Average	215,01	186,15	138,33
DP	7,77	22,03	9,91

The results of the Knoop microhardness test were tabulated in an Excel spreadsheet for comparison of the values between the groups (Tables 07, 08, 09).

Table 07: Values of the Knoop Group 01 microhardness test.

GROUP 01	Specimen 01	Specimen 02	Specimen 03	AVERAGE
Indentation 01	49.7	44.5	43.9	46.03
Indentation 02	28.7	28.2	29.1	28.67
Indentation 03	28	27.6	28.0	27.87

Table 08: Values of the Knoop Group 02 microhardness test.

GROUP 02	Specimen 01	Specimen 02	Specimen 03	AVERAGE
Indentation 01	40.5	41.4	40.9	40.93
Indentation 02	27.2	28.1	24.0	26.4
Indentation 03	23.1	27.2	23.1	24.46

Table 09: Values of the Knoop Group 03 microhardness test.

GROUP 03	Specimen 01	Specimen 02	Specimen 03	AVERAGE
Indentation 01	34.4	32.2	34.9	33.8
Indentation 02	25.4	26.1	26.1	25.8
Indentation 03	18.3	19.0	19.4	18.9

According to the result of table 4, group 01 (Composite resin photoactivated for 20s, with "STANDART" mode and tip close to the sample) showed better flexural strength. In addition, the highest values of the Knoop test also refer to group 01, data that can be related to studies that affirm that the physical characteristics of the resin can be influenced by the polymerization protocol carried out (Asmussen *et al.*, 2004; Schneider *et al.*, 2016; Alzahrani *et al.*, 2023).

The directions of the indentations followed the order: top, center of the sample, and bottom surface. When evaluating the results obtained, it is possible to state that the top surface presented higher results for the three groups of polymerization method. These data

can be related to the intensity of incident light, which is in direct contact with the active tip of the light-curing device, which may result in a greater number of photons available for photoinitiator activation, compared to the lower surface of the specimen, which is farther from the tip of the device (Silva *et al.*, 2022; Son *et al.*, 2014). Low conversion of monomers to polymers can result in lower surface microhardness values (Flury *et al.*, 2014).

CONCLUSION

The study demonstrated the importance of physical-mechanical tests, such as flexural strength and Knoop's microhardness, in evaluating the properties of microhybrid composite resins used in dental restorations. The results obtained indicate that the light-curing protocol, especially the exposure time and the proximity to the tip, directly influences the mechanical strength and hardness of the material, with better results observed in the groups with longer light-curing time. In addition, the variation in the conversion of monomers to polymers, especially on different sample surfaces, also contributes to the different mechanical performances, highlighting the importance of optimizing the polymerization process to ensure the longevity and effectiveness of dental restorations.

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