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BASIC RESEARCH:

Microhardness According to Surface, Distance and Time of Photopolymerization of a Bulk-Fill Resin: *In Vitro* Study

Microdureza según superficie, distancia y tiempo de fotopolimerización de una resina bulk-Fill: estudio *in vitro*

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ABSTRACT: This study compared Vickers microhardness (VHN) according to the surface, distance and time of photopolymerization (PP) of Tetric® N-Ceram Bulk Fill (TNCBF). An *in vitro* study was performed of 80 cylindrical samples (4 mm×4 mm) randomly distributed into 8 groups according to distance (0 and 3 mm) and time (10, 20, 30, 40 s) of LED PP 1470 mW/cm^2. The depth of cure was adequate when the ratio of tripled VHN (Kg/mm^2) between lower/upper surfaces was>0.8. Statistical analysis was performed with the three-way ANOVA test with post hoc Tukey and Tamhane ($p<0.05$). The effect on VHN was highly significant when individually analyzed by the surface, time and distance of PP and interaction time × distance of PP ($p<0.001$). The average depth exceeded 80% of VHN regardless of the distance and time of PP. The VHN was higher at a distance of 0 mm compared to 3 mm at 10 and 20 s of PP regardless of the surface ($p<0.001$). The VHN was similar at all times with a PP distance of 0 mm ($p\geq0.377$) and increased at 20 s with a PP distance of 3 mm ($p\leq0.001$). The surface, distance and time of PP affected the hardness of TNCBF, which can have an impact on its clinical performance. The depth of cure using VHN results were favorable with 20 s of PP at a distance of 0 mm for a thickness of 4 mm of TNCBF.

KEYWORDS: Composite resins; Dental healing lights; Dental materials; Hardness; Polymerization, Time factors.

RESUMEN: Comparar la microdureza de Vickers (VHN) según la superficie, distancia y el tiempo de fotopolimerización (FP) de Tetric® N-Ceram Bulk Fill (TNCBF). Estudio *in vitro* realizado en 80 muestras cilíndricas (4 mm×4 mm) distribuidas al azar en 8 grupos según distancia (0 y 3 mm) y tiempo (10, 20, 30, 40 s) de FP LED 1470 mW/cm^2. La profundidad de curado fue adecuada cuando el ratio de

VHN triplicadas (Kg/mm²) entre superficies inferior/superior fue>0,8. El análisis estadístico se realizó con la prueba ANOVA de tres vías con post hoc de Tukey y Tamhane (p<0,05). El efecto en la VHN fue altamente significativo al análisis individual de la superficie, tiempo y distancia de FP y por interacción tiempo × distancia de FP (p<0,001). El promedio de profundidad de curado superó el 80% de VHN independientemente de la distancia y tiempo de FP. La VHN fue mayor a una distancia de 0 mm en comparación a 3 mm con 10 y 20 s de FP independientemente de la superficie (p<0,001). La VHN fue similar en todos los tiempos con 0 mm de distancia de FP (p ≥ 0,377) y aumentó a partir de 20 s con 3 mm de distancia de FP (p≤0,001). La superficie, distancia y tiempo de FP afectaron la dureza de TNCBF lo que puede repercutir en su rendimiento clínico. La profundidad de curado mediante VHN fueron favorables con la fotopolimerización con 20 s a una distancia de 0 mm para un espesor de 4 mm de TNCBF.

PALABRAS CLAVE: Resinas compuestas; Luces de curación dental; Materiales dentales; Dureza; Polimerización; Factores de tiempo.

INTRODUCTION

Composite resins are indispensable for direct restorative treatments in dentistry (1-3), but they require good components to achieve long-term success (4). In 2009, a new generation of composite bulk-fill resins (BFR) by Dentsply (5) was marketed with a composition similar to conventional composite resins but with the incorporation of modified monomers and more efficient photoinitiators for correct photopolymerization (PP) (6). BFR use a 4 mm-5 mm incremental technique in wide and deep preparations (7), reducing the clinical and contamination time of the procedure (3, 8). Regular viscosity BFR have become a good restorative due to their durability of up to 10 years (9) and for presenting better or similar results compared to conventional composite resins in terms of PP stress, cusp deflection, marginal quality, microfiltration, fracture resistance, biocompatibility and wear (9, 10).

BFR avoid compromising the degree of conversion or mechanical properties, having more translucency and a lower filler concentration favors light penetration and the efficient use of PP systems (11, 12). They also incorporate photoinitiators, such as camphorquinone, phenylpropanidone, Lucerin TPO and Ivocerin, which increase absorption by influencing the hardness of the compound (2, 10). The inclusion of strain relievers in this material reduces the stress of contraction in PP (13). The reduced filler content of BFR to achieve high light transmission may weaken their mechanical properties compared to composite resins (14). In different studies, operator technique followed the manufacturer's instructions, placing a 4 mm increment and with a PP time of 20 s, having a good result with respect to the lower/upper mcirohardness (MHN) ratio>80% (13, 15).

The degree of conversion of monomers to polymers in BFR is an efficiency indicator indirectly assessed with MHN (16, 17). MHN is a physical quality assessed on surfaces and represents the best indicator of the grade of PP of composite resins (3, 11, 13, 15). A higher MHN of BFR provides resistance, while the opposite can lead to bacterial plaque buildup (2, 9, 13). The MHN of BFR with thicknesses>2 mm has been described as similar to conventional composite resins and presents a sufficient depth of cure (> 80%) (11, 18, 19). BFR brands have been evaluated for their physical properties. Tetric® N-Ceram Bulk Fill (TNCBF) is a moldable, high-viscosity (filling: 75-77%), nanohybrid composite with a transparency of 15%-17%

with application increments ≤ 4 mm that requires a PP wavelength of 400 nm-500 nm (blue light) and time of 10 s (≥1000 mW/cm^2) or 20 s (≥ 500 mW/cm^2) (12, 13, 20, 21). TNCBF incorporates a patented Ivocerin photoinitiator and strain relievers (15, 22-24). This composition increases the degree of conversion, avoiding physicomechanical properties, such as MHN (3, 16, 18, 19, 21, 22, 25-31).

In clinical practice, it is recommended to control the light quality of the PP device. However, some situations can increase restoration times and distances to the light source (10, 23, 30-33). Given the frequent use of BFR, it is necessary to evaluate whether the resin compromises performance, especially in the furthest part of the restoration. Therefore, the primary aim of the present *in vitro* study was to compare the MHN of TNCBF according to the interaction of surface, distance and time of PP factors, and the secondary objective was to evaluate the depth of cure of composite resins using the MHN index according to the distance and time of PP. The null hypothesis was that MHN is not affected by the interaction of the factors on the surface, distance and time of PP, and the secondary hypothesis was that the depth of cure is not affected by distance and time of PP.

MATERIALS AND METHODS

STUDY DESIGN

This experimental *in vitro* study was approved by the Stomatology Career of the Universidad Científica del Sur (N° 0025-DACE-DAFCS-U. CIENTÍFICA-2023) and developed in accordance with the International Organization for Standardization (ISO) 4049:2019 (Dentistry: Polymer-based restorative materials) and the CRIS (Checklist for Reporting *In-vitro* Studies) guideline.

STUDY GROUPS

The sample consisted of 80 standardized cylindrical discs with a diameter of 4 mm and height of 4 mm of TNCBF with IVA tone (Ivoclar, Vivadent, Schaan, Lichtenstein) (Table 1). The neasurements of all the specimens had a margin of error ≤ 1 mm corroborated with a vernier caliper and presented compact material without bubbles. The discs were evaluated and randomly distributed into 8 groups (n=10 per group) of PP protocols according to surface (upper and lower), distance (0 mm and 3 mm) and time (10 s, 20 s, 30 s, 40 s).

SAMPLE SIZE

Sample size was calculated according to Program G*Power 3.1.9.732 based on a pilot study of 24 discs included in the study (n=3 per group) with the formula for estimating the interaction of means with a large effect size (f=0.405), a confidence level of 95%, a power of 80%, numerator of 3 ((# surface groups-1) × (# distance groups-1) × (# time groups-1)) and number of groups of 8, giving a total of 71, which was increased to 80 to include 10 discs per group.

PREPARATION OF THE RESIN DISCS

The discs were made using a Teflon matrix with internal dimensions of 4 mm×4 mm, after which a thin layer of petroleum jelly was applied. The procedure included placing the matrix on top of a glass stage (10 cm×10 cm) separated with a celluloid matrix, with placement in a single increment of TNCBF by means of a Teflon spatula (Hu-Friedy, USA), and then covering with a second celluloid tape and glass plate with light pressure to compact the material and remove the residues until obtaining a homogeneous surface. The upper plate was removed to apply a thin layer of glycerin gel (KY Samutprakarn, Thailand) spread on the surface in order to inhibit the oxygen layer (2).

PHOTOPOLYMERIZATION

The LED PP was performed in a laboratory under dark conditions using an Elipar™ Deep Cure-L lamp (1500 mW/cm^2, 430 nm-480 nm, 3M ESPE, USA) with a 9 mm diameter tip. The radiance emission was verified before the procedure with a radiometer (±10% range, Woodpecker, China) every 5 samples (34). The lamp was programmed at the selected time (10 s, 20 s, 30 s, and 40 s) and the position was set with the help of a metal support so that the tip remained centered on the disc and in direct contact with the celluloid tape (distance 0 mm) or separated with a spacer device (distance 3 mm). The discs were removed from the mold and both surfaces were polished with fine-grained silicon carbide (3M, USA) abrasive papers for 10 s and identified with an indelible marker on the top surface. Finally, the discs were washed with distilled water (Alko, Peru) for 10 s and stored in amber containers with distilled water at 37°C in an incubator (HTL, China) for 24 h.

SURFACE MICROHARDNESS

The hardness test was blinded and performed by the same operator using the Vickers microhardness method (VHN). The upper and lower surfaces were analyzed with triplicate diamond-tipped indentations randomly spaced by at least 1 mm. The values for each surface were averaged to obtain a single value per disc. The VHN used a load of 50 g×15 s with a Vickers digital microdurometer (HVS-1000, China) expressed in Kg/mm^2. The depth of cure in percentage was adequate when the ratio of tripled VHN between the lower surfaces: upper surfaces was>80% (17). The flow chart of the experiment is presented in Figure 1 and the photos of the study procedure are presented in Figure 2.

STATISTICAL ANALYSIS

Descriptive statistics included means and standard deviation. A normal distribution was corroborated by the Shapiro-Wilk test ($P>0.05$). The homogeneity of variances was analyzed with Levene's test. The inferential comparison used was the three-way ANOVA analysis test of variance (surface*time*distance). The effect size (ω^2) was evaluated according to Cohen: very small (0–<0.02), small (0.02–<0.13), medium (0.13–< 0.26), and large (≥0.26) (35). Post hoc comparisons with homogeneous variances were made with Tukey (PP surfaces and distances) and with Tamhane for non-homogeneous variances (PP times). The data were analyzed with the IBM-SPSS v.27 statistical software at a significance level of 0.05.

Table 1. Description of the materials of the experiment.

Material	Composition	Manufacturer	Batch
Tetric® N-Ceram Bulk Fill	Monomers (Bis-GMA, Bis-EMA, UDMA), organic matrix (aluminofluoride borosilicate, glass, ytterbium triofluo-ride, mixed oxide, prepolymer) and photoinitiator (ivocerin and camphorquinone), with padding (76% weight/54% volume) and mean particle size (0.04 µm–3 µm).	Ivoclar, Vivadent, Schaan, Lichtens-tein	Z02V1F

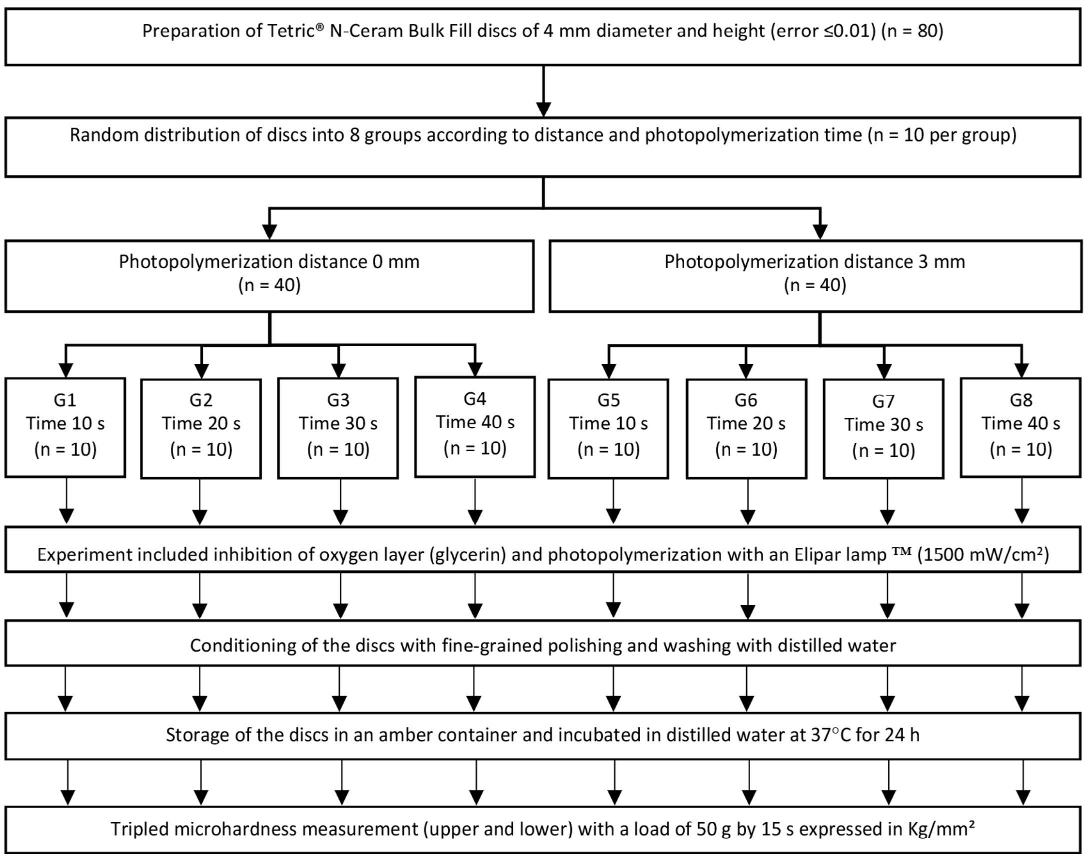

Figure 1. Flow chart of the microhardness experiment.

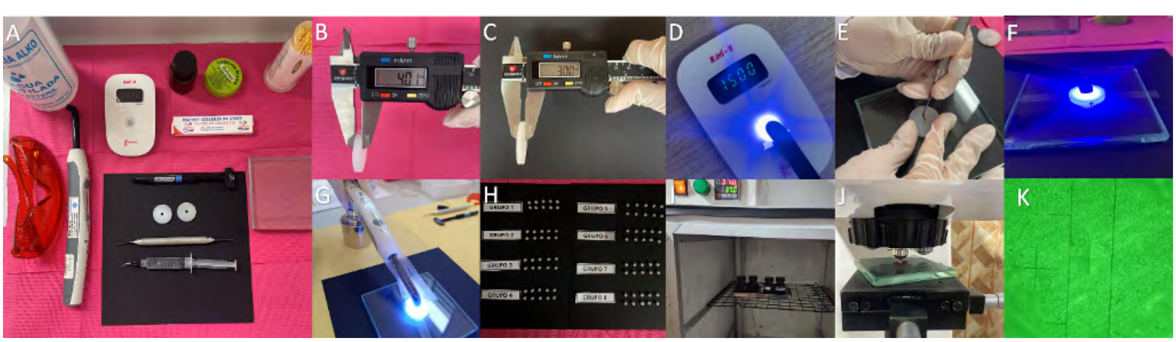

Figure 2. Photos of the study procedure: (A) Materials, (B) Teflon matrix measurement 4 mm, (C) Spacer device measurement 3 mm, (D) LED lamp calibration, (E) Resin placement, (F) Photopolymerization of resin discs at 0 mm, (G) Photopolymerization of resin discs at 3 mm, (H) Groups of resin discs, (I) Incubation at 37 °C, (J) Digital microdurometer and (K) Indentation for microhardness test.

RESULTS

Table 2 presents the interaction of the variables surface, time and distance of PP in the VHN of TNCBF. The surface of PP had a large effect on VHN, while PP time, distance, and time × distance had a small effect on VHN ($p<0.001$).

Table 3 shows the VHN according to surface area, time and distance of PP in TNCBF. On the upper surfaces the VHN ranged from 41.91 Kg/mm² (3 mm/10 s) to 46.87 Kg/mm² (0 mm/20 s) and from 34.57 Kg/mm² (PP: 3 mm×10 s) to 41.09 Kg/mm² (PP: 0 mm×20 s) on the lower surfaces. Regardless of the distance of PP, the VHN at a distance of 0 mm was similar at all PP times ($p\geq0.377$) and was greater after 20 s of PP at a distance of 3 mm ($p<0.001$). The VHN at 10 s and 20 s was greater than 0 mm on the upper surfaces compared to other PP distances and lower surfaces, while at 30 s and 40 s it was higher on the upper compared to the lower surfaces ($p<0.001$).

Table 4 presents the depth of cure calculated by VHN in the distance and time groups of PP. The average depth of cure ranged from 86.48%–89.45% at 0 mm and from 82.52%–90.3% at 3 mm. The % depth was greater at a distance of 0 mm versus 3 mm with 10 s of PP ($p=0.002$) and at a time of 20 s and 30 s versus other times of PP ($p=0.001$).

Table 2. Interaction of surface, time and photopolymerization distance variables in the microhardness of Tetric N-Ceram Bulk Fill.

Variables	SC	gl	F	p value	η²	ω²	Effect size
Surface	1247.69	1	262.390	<0.001*	0.510	0.507	Large
Distance	205.21	1	43.156	<0.001*	0.084	0.082	Small
Time	155.20	3	10.880	<0.001*	0.063	0.058	Small
Surface*Distance	0.001	1	0.000	0.988	0.000	-0.002	Null
Surface*Time	5.33	3	0.374	0.772	0.002	-0.004	Null
Distanc*Time	119.54	3	8.379	<0.001*	0.049	0.043	Small
Surfac*Distance*Time	27.76	3	1.946	0.125	0.011	0.006	Very small

*Interaction of variables. SC, sum of squares. Three-way ANOVA test. *p<0.05.

Table 3. Comparison of microhardness in Kg/mm² according to surface, time and distance of photopolymerization of Tetric N-Ceram Bulk Fill.

Photopolymerization time	Top surface, M ± SD		Bottom surface, M ± SD		p value
	0 mm	3 mm	0 mm	3 mm	
10 s	45.56 ± 2.22Aa	41.91 ± 2.81Ab	40.82 ± 3.59Ab	34.57 ± 2.51Ac	<0.001*
20 s	46.87 ± 1.00Aa	43.53 ± 2.73Bb	41.09 ± 0.81Ac	39.24 ± 2.42Bc	<0.001*
30 s	46.19 ± 1.65Aa	44.93 ± 1.09Ba	39.94 ± 1.99Ab	40.11 ± 1.35Bb	<0.001*
40 s	46.58 ± 1.85Aa	45.75 ± 0.69Ba	40.99 ± 3.3Ab	39.88 ± 2.15Bb	<0.001*
P valor	0.377	0.001*	0.762	<0.001*	

M, mean. SD, standard deviation. Distinct capital letters indicate significant differences in microhardness on the same surface and distance using different times of photopolymerization with Tamhane's post-hoc one-way ANOVA test. Distinct lowercase letters indicate significant differences in microhardness at the same time using different surfaces and distances of photopolymerization with Tukey's post hoc one-way ANOVA test. *p<0.05.

Table 4. Comparison of the depth of cure using the microhardness of the lower surface in relation to the upper surface according to photopolymerization time and distance of Tetric N-Ceram Bulk Fill.

	% Depth of cure: Bottom surface / Top, M ± SD		
Photopolymerization time	**0 mm**	**3 mm**	**p value**
10 s	89.45 ± 5.10Aa	82.52 ± 3.47Bb	0.002*
20 s	87.69 ± 2.70Aa	90.30 ± 4.91Aa	0.158
30 s	86.48 ± 3.37Aa	89.32 ± 2.63Aa	0.050
40 s	87.84 ± 4.21Aa	87.25 ± 5.25ABa	0.784
P valor	0.426	0.001*	

M, mean. SD, standard deviation. Distinct capital letters indicate significant differences in microhardness on the same surface using different times of photopolymerization according to Tukey's post-hoc one way ANOVA test. Distinct lowercase letters indicate significant differences in microhardness at the same time using different distances of photopolymerization according to the Student's t-test for independent samples. *p<0.05.

DISCUSSION

BFRs were developed to improve the properties of the material to achieve better performance; however, these results can be affected by factors related to PP (1-5). Surface properties, such as hardness, are an accessible indicator of clinical prognosis related to the esthetics and function of resins (3, 4, 15, 20, 29) and PP efficiency (1, 4, 5, 8-10, 13, 15). The null hypothesis of the study was rejected, finding that surface area, distance, and time of PP factors affected the MHN of the TNCBF. Furthermore, the average depth of cure was adequate, exceeding 80% at all resin distances and times of PP; however, it decreased with shorter PP times and greater PP distances.

The MHN of composite resins is considered adequate when it is greater than 50 (2, 3, 15, 18, 19, 22, 24, 28). In the present study, the type of surface factor had a large and significant effect on VHN. The upper surfaces of the TNCBF with PP at a distance of 0 mm obtained values closer to those recommended, with significant differences with respect to the lower surfaces (45–46 VHN versus 39–40 VHN). In other studies, the results were also close to (2, 15, 18, 23, 24, 29) or greater than 50 (3, 16, 19, 21, 22, 27, 28). This could be due to the attenuation of light (absorption, reflection and scattering) as it passes through the resin (3, 15, 25, 27).

In regard to the distance of PP factor, PP without spacing versus 3 mm of spacing affected the VHN of the upper surfaces of the TNCBF to a lesser extent with up to 20 s of PP. With respect to the distance of PP, at a distance of 0 mm versus 3 mm, PP affected the VHN of the upper surfaces of the TNCBF to a lesser extent at up to 20 s of PP. Although the effect was small, the influence of the lower PP ratio was demonstrated as described in previous studies (2, 25, 30, 32). According to the manufacturer's specifications, a PP distance of 0 mm is considered ideal, and our results adhere to the recommendations and findings of other studies (2, 20, 25, 30, 32).

When the effect of time of PP factor was analyzed, the PP time of 20 s showed a higher VHN efficiency of the TNCBF compared to longer times, as reported in previous studies (21, 23, 24, 33). On the other hand, the results of PP at 0 mm were similar at 10 s and 20 s, while at a distance of 3 mm VHN was greater at 20 s compared to 10 s. The monowave LED unit used at a higher PP distance in the present study could affect the efficiency of Ivocerin-associated camphorquinone photoinitiators; however, this decrease in VHN had a small effect and did not affect the depth of cure (> 80%). Ivocerin is more active in PP due to its greater absorption (400-450 nm), being stimulated by different wavelengths (15, 18, 27, 29). Several studies have confirmed the manufacturer's recommendations and thus, provided that the light source is adequate, mechanical properties, such as shrinkage stress and marginal integrity, are not affected (2, 3, 14, 15, 22-24).

MHN provides useful information about the depth of cure when comparing the top and bottom surfaces of composite resins considered optimal above 80% (2, 3, 7-9, 11, 15, 16, 19, 29). In the present study, the ratio of the surfaces of the TNCBF was greater than 86% at 0 mm and 82% at 3 mm of PP. Similar to multiple previous studies (12, 20, 22, 27), the results were optimal, demonstrating that the requirements stipulated in the ISO 4049 standard were met (1-3, 26-28), although in other studies this result was not achieved (15, 16, 18, 24). The chemical structure of TNCBF includes different monomers of high molecular weights, with which the high viscosity of bis-GMA is diluted by the low viscosity of bis-EMA and UDMA, producing a synergistic effect on the degree of conversion and depth of cure (27, 28). This allows for a decrease in PP shrinkage, a cure depth of 4 mm, and high MHN (2, 27, 28).

The results of this study are relevant to clinical practice by demonstrating that the time and distance of PP can affect the hardness of composite resins. Placing the lamp tip at a distance from surfaces can affect the depth of cure, especially on the lower surfaces of the TNCBF. It is also important to consider other factors related to the characteristics of the PP lamp, such as the type of wavelength, optimal load or the time of use, which may generate shrinkage and filtration of the restoration. In this study, a monoway light-curing unit was used with a wavelength (430 nm-480 nm) above the recommended range to photoactivate Ivocerin from TNCBF (400 nm), although Ivocerin this photoinitiator, might still be sensitive to wavelengths from 400 nm to 430 nm (36). On the other hand, the use of light-curing units could generate clinical challenges to control the thermal emission that affects the contraction of BFRs (37).

Nonetheless, the results of this study should be interpreted with caution as *in vitro* designs do not precisely replicate the occlusal function that would apply forces and wear on the surfaces of restored teeth, or the clinical factors of the operator's techniques for the placing of composite resins (12, 19). Another limitation is that the application of thermocycling was not carried out, which allows for simulating the aging of resins in an intraoral environment, affecting the MHN of the TNCBF (23). However, the experiment considered light and temperature control in the sample incubator to simulate the intraoral cavity and to complete post-polymerization of the TNCBF (12). Other mechanical properties, such as roughness and optics, that could increase the evaluation of properties, were also not explored. Therefore, it is recommended that future studies take these factors into account.

CONCLUSIONS

Within the limitations of an *in vitro* study, it was concluded that the surface, distance, and time of PP affect the hardness of TNCBF, which may have an impact on its clinical performance. Furthermore, surface distance, greater distance, and shorter PP time significantly affected VHN. The depths of cure were favorable in all PP distance and time groups (>80%), but 20 s of PP at a distance of 0 mm was sufficient for the VHN result of a 4 mm thickness of TNCBF, as recommended by the manufacturer.

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