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Determination of microhardness of bulk-fill resins at different depths

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ABSTRACT

The aim of this study was to determine Vickers microhardness (HV) in bulk fill resins at different depths. Test specimens were prepared with different bulk fill resins: Filtek Bulk-Fill (3M ESPE) [FBF], Surefill SDR flow (Dentsply) [SDR], Fill-UP (COLTENE) [FU] and Surefill (Dentsply) [SF]. Semi-cylindrical test specimens were prepared in a mold 6 mm in diameter and 4 mm thick (n=5). A 1000 mW/cm2 light curing unit was applied (Coltolux LED - Coltene) for 20 seconds. HV was determined with three indentations (Vickers Future Tech FM300, 300 g, 8 s) at four depths: 1, 2, 3 and 4 mm from the top surface to the interior. Data were recorded immediately (t0) and 24 hours later (t24). Results were analyzed with two-way ANOVA ($p<0.05$), and multiple comparisons were performed using Tukey's test. Mean and SD of HV at t0 for each mm were: [FBF] t0: 49.23(4.65) / 48.32(3.36) / 44.38(2.06) / 40.59(2.58); [FBF] t24: 61.37(3.47) / 62.63(3.03) / 57.27(5.22) / 56.37(5.88); [SDR] t0: 27.81(3.13) / 28.07(2.4) / 27.24(2.94) / 25.71(3.0); [SDR] t24: 35.11(2.16) / 35.17(1.96) / 35.53(1.81) / 33.18(2.08); [FU] t0: 41.43(1.41) / 39.87(0.88) / 38.11(1.81) / 39.09(1.92); [FU] t24: 49.27(1.54) / 48.77(1.77) / 48.65(1.88) / 46.76(4.93); [SF] t0: 71.35(7.09) / 67.39(9.76) / 68.95(6.21) / 64.1(8.35); [SF] t24: 76.06(6.61) / 75.31(9.37) / 75.2(11.57) / 69.81(12.14).

ANOVA showed significant effect of material, depth and recording time ($p<0.05$), and Tukey's test showed that recording sites (depths) differed significantly, giving four homogeneous groups.

Under the conditions of this study, it can be concluded that microhardness of bulk-fill resins can be affected by depth and post-curing time.

Keywords: *hardness - composite resins - hardness test.*

Determinación de microdureza de resinas bulk-fill en diferentes profundidades

RESUMEN

El objetivo del presente estudio fue determinar la microdureza Vickers (HV) en resinas bulk-fill a diferentes profundidades. Se confeccionaron probetas semicilíndricas de 6 mm de diámetro y 4 mm de profundidad con diferentes composites de aplicación en bloque (Bulk-fill): Filtek Bulk-Fill (3M ESPE) [FBF], Surefill SDR flow (Dentsply) [SDR], Fill-UP (COLTENE) [FU] y Surefill (Dentsply) [SF]. Se polimerizaron con Coltolux LED (Coltene) con 1000 mW/cm^2 durante 20s. La HV se determinó realizando 3 indentaciones con 300 g durante 8 s a 1,2 3 y 4 mm desde la superficie de la probeta hacia el interior inmediatamente después de curada y a las 24 h. Se utilizó un microdurómetro Vickers Future Tech FM300. Los resultados se analizaron estadísticamente mediante ANOVA de dos vías y Prueba de Tukey.

La media y DS de HV fueron: [FBF] t0: 49,23(4,65) / 48,32(3,36) / 44,38(2,06) / 40,59(2,58); [FBF] t24: 61,37(3,47) / 62,63(3,03) / 57,27(5,22) / 56,37(5,88); [SDR] t0: 27,81(3,13) / 28,07(2,4) / 27,24(2,94) / 25,71(3,0); [SDR] t24: 35,11(2,16) / 35,17(1,96) / 35,53(1,81) / 33,18(2,08); [FU] t0: 41,43(1,41) / 39,87(0,88) / 38,11(1,81) / 39,09(1,92); [FU] t24: 49,27(1,54) / 48,77(1,77) / 48,65(1,88) / 46,76(4,93); [SF] t0: 71,35(7,09) / 67,39(9,76) / 68,95(6,21) / 64,1(8,35); [SF] t24: 76,06(6,61) / 75,31(9,37) / 75,2(11,57) / 69,81(12,14).

La evaluación con análisis de varianza mostró el efecto significativo de las variables material, profundidad y momento del registro ($p<0,05$) y la prueba de Tukey mostró que los sitios de registro (profundidad) fueron estadísticamente significativos, dando cuatro grupos homogéneos.

Bajo las condiciones de este estudio podemos concluir que la microdureza de las resinas de inserción en bloque se ve afectada por el nivel de profundidad y el tiempo pos curado.

Palabras clave: *dureza - resinas compuestas - pruebas de dureza.*

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INTRODUCTION

Light-curing composites are the most frequently used materials in dental practice, with a wide range of applications. In recent years, a new kind of composite resins has been developed, known as bulk-fill composites because they can be placed in a single increment, thereby simplifying and shortening the restoration procedure. They are presented commercially according to consistency as high- or low-viscosity, and according to polymerization activation as self-curing, light-curing or dual-curing. These materials polymerize adequately when applied in layers 4 or 5 mm thick, according to brand. Some manufacturers explain that the greater curing depth of these materials is due to the addition of a more sensitive photoinitiator system and greater translucence of the material[1]. At the same time, they generate less shrinkage stress, which may vary according to composition, whether by modification of monomers or the filler content, or by addition of stress mitigators or polymerization modulators[2,3].

Increasing the thickness of the layer of material would imply an increase in polymerization shrinkage. This needs to be considered in the development of these materials in order to compensate for it by modifying the formulations, e.g., by increasing the ceramic filler load or the molecular weight of the monomers[4]. These modifications imply an increase in the modulus of elasticity of the material, which minimizes the possibility of dissipating tensions generated during polymerization[5].

Flowable bulk-fill composites have a greater content of organic matrix, which may lead to greater polymerization shrinkage and low mechanical properties, which conditions its application in occlusal areas. Bulk-fill composite manufacturers therefore indicate that they must be covered with a layer of conventional composite[3,6]. Some authors confirm a reduction in shrinkage stress in bulk-fill composites with low percentage of filler despite the increase in thickness of the layer of material[2,4]. These materials with low percentage of ceramic filler, such as SDR Flow, minimize shrinkage stress because they contain a chemical component that acts as polymerization modulator, with the aim of slowing polymerization speed to reduce shrinkage stress in spite of being polymerized with curing units in continuous, high-intensity mode[2].

The aim of this new kind of restorative composites, which is to shorten operation times by increasing the thickness of each layer, may hinder the penetration of curing light, reducing the degree of conversion of monomers to polymers[7]. The degree of conversion of a composite depends not only on its composition, but also on factors related to photoactivation, including the curing unit used, the type of photoactivation selected and the quantity of energy applied[1]. Another factor to consider with relation to degree of conversion is the possibility of composite resins undergoing elution in the oral cavity, with special interest in the release of monomers, due to their potential cytotoxicity[8]. It has been shown that monomer release is inversely proportional to the degree of conversion of monomers into polymers, which is related to exposure time to light, among other factors. Nevertheless, arbitrarily increasing polymerization time with the aim of preventing lack of curing may damage not only the pulp, but also adjacent tissues due to increase in temperature[9-11].

Previous studies have shown that degree of conversion can be measured directly or indirectly. Czasch et al.[12] and Leprince et al.[13] recommend evaluating the degree of conversion directly, while other authors recommend measuring microhardness as an indirect method for determining degree of conversion[14-16], since there are publications that have reported a good correlation between degree of conversion and microhardness[17-19]. Another method for evaluating degree of curing according to thickness of the material is by evaluating hardness at the surface exposed to the light (top) and the opposite surface (bottom), considering polymerization to be adequate when the ratio between them is 80% or higher.

The aim of this study was to determine Vickers microhardness (HV) in bulk-fill resins at different depths.

MATERIALS AND METHODS

Four bulk-fill composites were used for this study: 1) Filtek Bulk-Fill (3M ESPE), 2) Surefill SDR flow (Dentsply), 3) Fill-UP (COLTENE), and 4) Surefil (Dentsply) (Table 1).

Semi-cylindrical test specimens were prepared in a mold 6 mm in diameter and 4 mm deep (n=5). The flat surface was dismountable to allow microhardness to be determined in the depth of the specimen (Fig. 1). Specimens were cured with a Coltolux LED unit (Coltene) at intensity 1000 mW/cm^2 for 20 s.

Table 1. Information on the materials used

MATERIAL	MANUFACTURER	DESCRIPTION	BATCH
Filtek Bulk-Fill **[FBF]**	3M	Light-cured composite with filler loading 42% by volume	Batch N711074
Surefill SDR flow **[SDR]**	Dentsply	Flowable light-cured composite with filler loading 44% by volume	Batch 1508283
Fill-UP **[FU]**	Coltene	Dual-cured flowable composite with filler loading 49% by volume	Batch H28295
Surefil **[SF]**	Dentsply	Light-cured packable composite with filler loading 62% by volume	Batch 131024

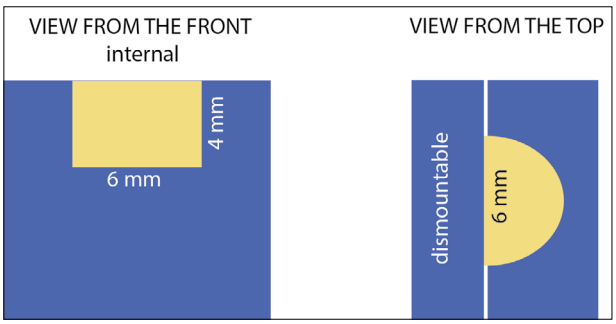

Fig. 1: Diagram of test specimens.

An extra-fine indelible marker was used to draw a vertical mark on each specimen to divide it in half and separate the indentations made immediately after light curing (t0) on one side from those made at 24 hours (t24) on the other side (Fig. 2).

Hardness was measured with a Vickers Future Tech FM300 microhardness tester by indenting with 300 g for 8 seconds at depths of 1, 2, 3 and 4 mm. Fig. 3 shows an example of the indentations made.

Measurements were recorded and analyzed statistically by ANOVA for repeated measures ad

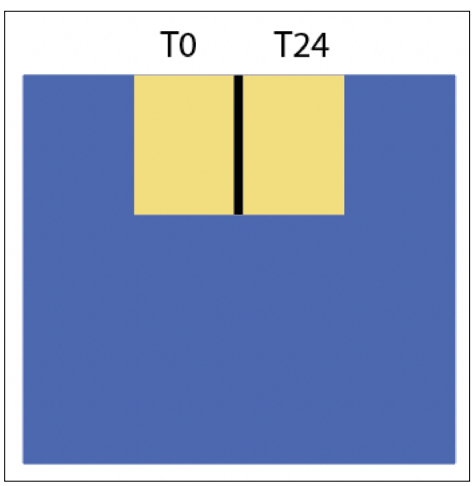

Fig. 2: Diagram showing division of the test specimen for indentations at T0 and T24.

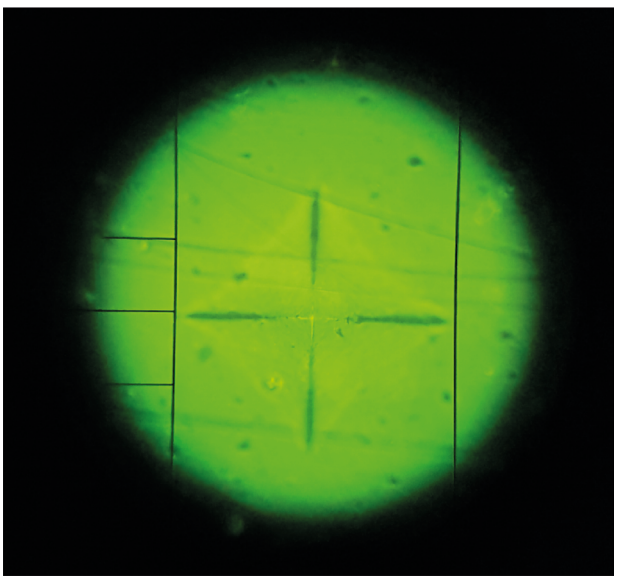

Fig. 3: Photomicrograph of an indentation made with the hardness tester.

Tukey's test. Two-way ANOVA was used to analyze the time variable.

RESULTS

Table 2 shows means and standard deviations of the values recorded.

Table 3 shows the value of the ratio of the hardness measured at 4 and 1 mm depths, according to the formula *hardness at 4 mm / hardness at 1 mm.*

Analysis of variance showed a significant effect of depth and depth-time interaction ($p<0.05$) when microhardness was measured immediately after polymerization (T0) (Table 4). Tukey's test described the presence of 3 subsets: 1) Surefill SDR flow, 2) Fill-UP and Filtek Bulk-Fill, and 3) Surefil.

At 24 hours (T24), a statistically significant difference was found for depth and not for depth/material interaction (Table 5). Tukey's test showed four subsets, with all materials differing significantly from each other.

Table 2: Mean and standard deviation found for each material immediately after light-curing (0) and 24 hours later (24)

Depth	FBF0	FBF24	SDR0	SDR24	FU0	FU24	SF0	SF24
1 mm	48.9 (3.9)	60.3 (4.7)	28.0 (3.6)	34.8 (2.9)	41.9 (1.6)	49.2 (1.1)	73.3 (6.6)	74.7 (9.1)
2 mm	48.3 (3.4)	60.9 (4.2)	27.6 (2.3)	35.2 (1.9)	40.0 (0.7)	48.0 (2.2)	63.1 (12.8)	73.4 (9.8)
3 mm	44.0 (2.5)	57.5 (5.9)	27.0 (3.4)	35.6 (2.1)	38.2 (2.2)	49.8 (1.5)	67.5 (6.6)	74.2 (13.4)
4 mm	41.5 (2.5)	55.1 (8.7)	25.5 (3.4)	33.3 (2.4)	39.2 (1.9)	45.9 (5.0)	62.6 (9.2)	66.8 (11.9)

Table 3: Ratio between hardness values at 4 and 1 mm depths for each material at 24 hours

	4 mm / 1 mm ratio
FBF24	0.92 (92%)
SDR24	0.95 (95%)
FU24	0.95 (95%)
SF24	0.92 (92%)

Table 4: Analysis of variance of data recorded immediately after polymerization

Effect	Value	F	DF of the hypothesis	DF of the error	Sig.
Depth	0.801	17.425	3	13	<0.001
Depth * material	1.164	3.169	9	45	0.005

Table 5: Analysis of variance of data recorded 24 hours after polymerization

Effect	Value	F	DF of the hypothesis	DF of the error	Sig.
Depth	0.489	4.145	3	13	0.029
Depth * material	0.368	0.700	9	45	0.706

Table 6: Post-hoc analysis. Tukey's test (microhardness at 4 mm)

Hardness (4 mm)				
Material	N	Subset		
		1	2	3
Surefill SDR flow -2-	10	29.6500		
Fill-UP -3-	10		42.86	
Filtek Bulk-Fill -1-	10		49.05	
Surefil -4-	8			66.00

Taking as a reference the values detected at 4 mm depth, analysis of variance showed the significant effect of the variables time and material (p<001), with no significant difference in the interaction between these two variables (p=0.706). Tukey's test described the presence of 3 subsets: 1) Surefill SDR flow, 2) Filtek Bulk-Fill and Fill-UP, and 3) Surefil (Table 6).

DISCUSSION

The flowable bulk-fill resins used in this study had lower microhardness values than those of regular consistency, in agreement with previous studies[3,11,16,20], possibly due to their low ceramic filler content. In addition, SDR Flow resin is light-curing, while Fill Up resin is dual-curing, which suggests that it may be harder than SDR Flow as a result of the sum of the two forms of activation. It is also important to consider the post-cure factor, since microhardness values measured immediately after curing the composites differed significantly from those measured 24 hours later[11,21,22].

Composite resin microhardness is also affected by the thickness of the layer[20]. It was concluded in that study that resin hardness in the area farthest from the curing unit (bottom) differed significantly from hardness at the top in specimens 4 or 5 mm thick. Lower microhardness values at 4 mm thickness agree with results of other studies[23].

Regarding the evaluation of microhardness in depth, some studies have determined top and bottom hardness of specimens of different thicknesses of light-cured composite resin to define its curing depth. Kim et al.[20] evaluated Vickers microhardness only at top and bottom of different specimens 2, 3 and 4 mm thick, using a load of 200 grams with a 10-second dwell time, finding that hardness decreases with increasing depth, though the decrease is less in bulk-fill composites. They conclude that there is statistically significant difference in microhardness according to type and thickness of the material, and the interaction between them, in agreement with the results found in the current study, even though a different measuring method was used. Another variable considered in the literature is the uniformity of polymerization throughout the thickness of the material, e.g., the study by Fronza et al.[16] showing that degree of conversion is not uniform in specimens thicker than 4mm. In that study, only SDR and FBF showed uniform

polymerization throughout the restoration. It is therefore necessary to evaluate microhardness not only at the surface, but also at different depths. Our study took measurements at different depths in each specimen to minimize the factors that could influence results. This methodology was also used by Comba et al.[24], who evaluated Vickers microhardness not only by means of the bottom/top ratio, but also at each millimeter in depth in specimens 6 mm thick. Considering surface microhardness values as reference points, the regression analysis showed that SDR had a significant difference at 2 mm depth, and X-tra Base and Filtek Bulk Fill showed a significant difference at 3 mm depth, with values lower than those recommended by the manufacturer. They also found that SDR had the lowest microhardness values, attributable to its low percentage of ceramic filler. According to the authors, other materials such as Filtek Bulk Fill, showed a low percentage in filler content by volume, but higher microhardness values, which may also be attributed to other factors unrelated to filler content, but strictly associated to the composition of the matrix.

Although the results showed statistically significant differences at different depths, analysis of the general behavior shows that the level of polymerization was acceptable at the depths suggested by the manufacturers, considering that the ratio between hardness measured at depths of 4 and 1 mm was greater than 80% for all materials. A bottom/top hardness ratio higher than 80% is usually used as a minimum clinically acceptable threshold for degree of conversion. Although our study did not directly evaluate top and bottom microhardness, but measured it instead at each millimeter of depth, the hardness ratio between mm 1 and mm 4 was 80% or more for the bulk-fill composites used. This means that the study materials can be adequately placed and cured in thicknesses of 4 mm, with statistically significant differences at the depths evaluated. These results agree with Kim et al.[20] and Rizzante et al.[3], who concluded that the bottom/top ratio was higher than 80% down to depths of 4.0 and 4.5 mm in all Bulk-Fill composites.

It would be advisable to conduct further studies to evaluate the degree of cytotoxicity of this type of bulk-fill resins in order to securer a more complete evaluation of their characteristics.

CONCLUSIONS

Under the conditions in this study, it can be concluded that the microhardness of bulk-fill resins is affected by the material evaluated, depth, and post-curing time.

DECLARATION OF CONFLICTING INTERESTS

The authors declare no potential conflicts of interest regarding the research, authorship, and/or publication of this article

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