Original Article

Assessment of bulk-fill of resins microhardness longitudinal

Fernanda Santos Araújo[1] , Wilton Mitsunari Takeshita[2] , Regiane Cristina do Amaral[2] , Adriano Augusto Melo de Mendonça[2*]

[1] Master's student of the Post-graduation Program in Dentistry (PRODONTO), Federal University of Sergipe (UFS).

[2] Adjunct professor of the Department of Dentistry of the Federal University of Sergipe (UFS).

Corresponding author:
Adriano Augusto Melo de Mendonça
Adjunct professor of the
Department of Dentistry
Federal University of Sergipe (UFS)
E-mail: adri_amm@yahoo.com.br

Aim: This study aimed to assess the polymerization effectiveness of bulk-fill composite resins in longitudinal microhardness. **Methods:** Blocks of bulk-fill composite resin with thicknesses of 6 mm were analyzed with Vickers microhardness. The resin blocks were divided into two groups (n=6): resin AURA and OPUS. The microhardness test was performed before (base and top) and after (longitudinal microhardness) sectioning the blocks at distances of 2 mm, 4 mm, and 6 mm from the top of the block. The mean microhardness values were tabulated and subjected to ANOVA followed by Tukey's test (p<0.05). **Results:** The OPUS bulk-fill resin samples presented microhardness means of 55.9 kgf/mm^2, 53.7 kgf/mm^2, and 49.3 kgf/mm^2, the AURA bulk-fill resin samples presented microhardness means of 57,02 kgf/mm2, 55,86 kgf/mm^2 e 51,77 kgf/mm^2 for the distances of 2 mm, 4 mm, and 6 mm, respectively. Tukey's statistical test showed a significant difference in microhardness values at different distances of 2 mm, 4 mm, and 6 mm (p<0.001) for each resin. Although there was a statistically significant difference within and between the groups assessed, all samples showed polymerization effectiveness when comparing the top and base of the block. **Conclusion:** Polymerization was effective in different thicknesses (2 mm, 4 mm, and 6 mm) in both resins studied. The microhardness ratio was adequate when comparing the base and top.

Keywords: Composite resins. Hardness. Polymerization.

Introduction

Over the last decade, resin composite materials have increased significantly in the dental field. Improvements in esthetic properties and strategies of adherence to the dental structure increased the number of direct and semi-direct procedures with resin materials[1]. However, polymerization shrinkage is still the most significant disadvantage to be overcome regarding resin materials[2]. Some resin composites can have a volumetric polymerization shrinkage variation from 2% to 3%, producing cracks between the dental structure and the restorative material[3]. Such cracks may form secondary caries or pathological changes on the dental pulp.

To overcome the polymerization shrinkage phenomenon, a new category of resins has been developed over the last years. Bulk-fill resins may be applied in increments with a thickness equal to or greater than 4 mm and polymerized in a single step[4]. According to the manufacturer's data, combining a potent photoinitiator system and high material translucency allows polymerization to reach deeper layers adequately. Some studies performed with bulk-fill resins showed satisfactory polymerization results compared to composite resins used in restorative procedures in increments[5-7].

The microhardness test has been an effective tool for assessing the polymerization effectiveness of composite resins[8,9]. This test allows measuring the degree of monomer conversion into polymers inside resin materials. The degree of conversion depends mainly on factors such as monomer chemical structure, photoinitiator concentration, polymerization conditions, and curing mode[9]. In more significant increments, a light-emitting source's energy drastically decreases as it enters the material. Thus, resins inserted in increments may present more standardized results[10].

Regarding the degree of conversion and the behavior of bulk-fill resins, this result needs to show a consensus in the scientific literature[11]. This becomes even scarcer when investigating increments of different thicknesses. The literature reports that different brands of bulk-fill resins present limits for diversified increments, which does not standardize the material[11]. In the study by Silva (2019)[12], when testing three different brands of Bulk-fill resin, it was found that in one of the brands, there was a statistically significant difference between the microhardness between the layers evaluated. Therefore, microhardness must be compared, mainly at the top and base, to verify its homogeneity in the restored material.

Thus, the present study aimed to investigate the degree of conversion, through the microhardness test, of three different thicknesses (2 mm, 4 mm, and 6 mm) in two composite resins and analyze the top and base microhardness.

Materials and Methods

For this study, two bulk fill resins were used from the commercial brands AURA and OPUS, which, according to the manufacturers, can produce resin increments of up to 5 mm in thickness.

Specimens were produced (n=36) in a cylinder shape, 10 mm x 6 mm in diameter and thickness. Measuring the depth of the metallic mold to build the specimens required a millimeter periodontal probe (Millennium). The specimens were produced by inserting a single composite resin increment with an insertion spatula (Duflex).

A polyester matrix and a glass slide were placed on the resins. Then, a mild digital pressure was applied to eliminate potential material excess and ensure surface smoothness for the samples to be analyzed. Later, the glass slide was removed, maintaining the polyester matrix, followed by material curing with a light-curing device (SDI, Victoria, Australia) with an intensity of 470 mW/cm^2 placed on the upper surface of the sample for 40 seconds, according to the manufacturer's recommendations. During the study, the intensity of luminous energy emitted by the light source was monitored with the help of a radiometer (SDS Kerr, Middleton, USA).

After polymerizing the specimens, the excess was removed with a #15 scalpel blade, and the blocks were subjected to Vickers microhardness at the top and base. After verifying surface microhardness, the specimens were sectioned with sandpaper discs attached to a handpiece and a bench motor (Beltec) to verify the longitudinal microhardness of the blocks.

The sectioned surfaces were polished with 1200-granulation sandpaper for 60 seconds. Next, each block was stored in a white and opaque recipient to prevent light from entering and identifying.

Microhardness was determined with the Vickers microhardness test. First, the samples were subjected to superficial silver metallization with the device (model 108, Kurt J. Lesker Company, Pennsylvania, USA) to make indentation reading easier. In each sample, the base and top were analyzed, whereas the base was the surface away from the light source, and the top was the layer closest to it. Four indentations were performed at the top and four at the base. For longitudinal microhardness (2 mm, 4 mm, and 6 mm), four indentations were performed at each distance (resulting in 12 longitudinal indentations in the block). A load of 1 kg/F was applied for 10 seconds in a microhardness tester (FM 800).

The sample was placed on the device platform to stand perpendicular to the axis of the indentation device. A 40x objective and the sample area were selected to perform indentation. After indentation, the marks of both diagonals left on the material surface after load ablation were measured. After reading the values, the machine calculated the arithmetic mean and the area of the inclined indentation surface, recording the Vickers hardness value, which is the quotient obtained when dividing the load (in kgf) by the indentation area.

The data were statistically analyzed with the Shapiro-Wilk normality test. The Levene test was used to assess homoscedasticity, which later allowed the analysis of variance (2−way ANOVA, resin, and thickness) complemented by Tukey's HSD test (SPSS for Windows 11.5, SPSS, Chicago, IL) at an significance level of $p < 0.05$.

Results

The OPUS bulk-fill resin samples presented microhardness means of 55.9 kgf/mm^2, 53.7 kgf/mm^2, and 49.3 kgf/mm^2 for the distances of 2 mm, 4 mm, and 6 mm, respectively. Tukey's test was applied to compare the samples and identified a significant difference among the three values (p<0.001). The same pattern was observed for the microhardness means of the AURA resin samples. The microhardness values for 2 mm, 4 mm, and 6 mm away from the light source were 57.02 kgf/mm^2, 55.86 kgf/mm^2, and 51.77 kgf/mm^2, respectively. Tukey's statistical test showed a significant difference in microhardness values at different distances of 2 mm, 4 mm, and 6 mm (p<0.001) when compared.

When comparing the microhardness means between both bulk-fill resins investigated, the microhardness values for the AURA bulk-fill resin were lower than those of the OPUS bulk-fill resin (Table 3).

Table 1. Resins used and composition of organic and inorganic portions.

Material	Organic matrix	Inorganic load	Load (weight %)	Manufacturer
AURA Bulk fill	UDMA Bis-GMA	UHD load, silica of 0.02-0.04 μm, barium glass of 0.4 μm	81.00%	SDI
OPUS Bulk fill	UDMA Bis-EMA TEGDMA	Silanized barium glass, red iron oxide, white titanium oxide	79.00%	FGM

Table 2. Group distributions, mean (standard deviation), lower and upper values, and base/top ratio of each group analyzed.

Brand	N	Mean (standard deviation) kgf/mm^2	Confidence interval		Ratio (base/top)
			Lower limit	Upper limit	
OPUS 2 mm	6	55.91(0.19)A	55.71	56.12	
OPUS 4 mm	6	53.72(0.53)B	53.17	54.29	0.88
OPUS 6 mm	6	49.37(0.86)C	48.47	50.29	
AURA 2 mm	6	57.02(0.29)D	56.72	57.33	
AURA 4 mm	6	55.86(0.46)A	55.37	56.35	0.90
AURA 6 mm	6	51.77(0.55)E	51.19	52.36	

*Different letters determine statistical differences between the groups investigated.

Table 3. Comparison of resin types and increment thicknesses for the top and base of the samples.

Brand	N	Mean kgf/mm^2	Standard deviation	Confidence interval		Tukey's HSD
				Lower limit	Upper limit	
top						
OPUS 2 mm	6	60.69	4.88	55.56	65.81	AB
OPUS 4 mm	6	81.29	4.89	76.15	86.41	D

Continuation

OPUS 6 mm	6	72.19	3.45	68.56	75.81	CD
AURA 2 mm	6	52.75	9.2	43.09	62.41	A
AURA 4 mm	6	67.66	2.98	64.53	70.79	BC
AURA 6 mm	6	66.29	5.13	60.89	71.68	BC
base						
OPUS 2 mm	6	60.16	4.50	55.44	64.88	AB
OPUS 4 mm	6	80.65	4.75	75.67	85.64	D
OPUS 6 mm	6	58.91	3.54	55.20	62.62	ABC
AURA 2 mm	6	52.98	9.58	42.92	63.03	AC
AURA 4 mm	6	63.88	3.86	59.84	67.93	B
AURA 6 mm	6	49.65	4.66	44.76	54.54	C

*Different letters represent groups with statistically significant differences ($p<0.05$). ANOVA followed by Tukey's test.

According to Tukey's statistical test, when comparing the microhardness values for increments at the same distance from the light source, all means presented statistically different values (Table 3).

Discussion

Several methodologies have been used to assess the polymerization effectiveness of resin materials[10,11]. Degree of conversion and microhardness are physical tests established in the literature[10,13]. Despite the great variety of tests, the Vickers microhardness test has been effective, extensively promoted, and shown results that indicate the behavior of resin materials.

The microhardness of different composite resins was assessed because it is among the most important physical characteristics of restorative materials and is directly linked to the strength and ability of abrasion or wear of material[14]. The Vickers microhardness test is performed over the length of the specimen and used to determine polymerization depth because changes in microhardness may reflect the degree of material polymerization[10].

Conventional composite resins are indicated for use in increments of 2 mm[15]. Bulk-fill resins may be used in more significant increments[16]. The maximum thickness indicated by the manufacturers of AURA and OPUS bulk-fill resins is 5 mm. To analyze polymerization effectiveness, the methodology used had the sectioning technique of composite resin samples in the direction of their long axis. Thus, it was possible to assess microhardness in three thicknesses of each specimen (2 mm, 4 mm, and 6 mm).

According to Orlawski and colleagues (2015)[17], to achieve satisfactory polymerization effectiveness, bulk-fill resins must present some fundamental characteristics, such as material translucency, to allow the light to penetrate more efficiently in deeper areas, thus increasing polymerization effectiveness. Moreover, the chemical composition of

the material and the distance between the light source and the composite resin surface also interfere with polymerization depth[18].

Another factor that explains the differences in resin hardness is that polymerization depends on other factors of composite resins, such as the type and size of the load and its chemical composition. Load size interferes with light dispersion[19] and, according to Guiraldo and colleagues (2009)[20], the loss of polymerization light energy is related to the light dispersion by load particles. Thus, it is assumed that the larger the load size, the lower the light dispersion and the higher the degree of conversion and microhardness.

Therefore, light intensity directly affects polymerization depth, and the microhardness value obtained on the surface closest to the light source may be higher than[21]. Light intensity was not assessed in this study; thus, during the entire experiment, the intensity of luminous energy emitted by the light source was standardized, aided by a radiometer to measure intensity. The time of light exposure was the same for all samples. Moreover, the superficial values of the top of each resin sample differed from the material.

The results show a statistically significant difference between the layers of the samples within the same group and between the resin groups used in the experiment. There was a decrease in microhardness values toward the deepest layers of the composite resins, obeying a pattern in both resin groups studied.

A lower hardness in the deepest layers comes from inefficient polymerization, which may reduce composite resin component conversion and, consequently, more excellent residual monomer remnants[11]. The presence of residual monomers at the base of the composite resin layer may diffuse through the hybrid layer and reach dentinal tubules toward the pulp tissue. This may lead to resin cytotoxicity, cause pulp sensibility, and reduce the material's mechanical properties[15].

According to Esteves (2015)[13], the statistical difference between the mean microhardness values of the layers and the decrease in verified microhardness by the increase in composite resin depth may occur due to the decrease in light-curing intensity over the composite thickness. This may explain the result in the present study regarding the differences in microhardness values among the inner layers of bulk-fill composite resins assessed.

When comparing the microhardness means between both bulk-fill resins investigated, the microhardness values for the AURA bulk-fill resin (2 mm, 4 mm, and 6 mm) were lower than those of OPUS bulk-fill resin (2 mm, 4 mm, and 6 mm). However, although presenting differences for hardness, all samples obtained polymerization effectiveness in different thicknesses, considering that the ratio (base/top) values were higher than 0.8 kgf/mm^2.

The most common form of measuring composite resins' conversion or polymerization effectiveness rate is verifying the value obtained through the ratio between base/top microhardness[10,21]. This reflects the relative extension of conversion of the most profound surfaces relative to the top surface. The value used as a criterion has been 0.8 kgf/mm^2, indicating an adequate polymerization rate of the material[10,22].

Translucency is one of the factors that can explain the difference in hardness among resins. Studies such as those by Arimoto and colleagues (2010)[23] reinforce the role of material translucency in light transmission. Translucency is the relative amount of light transmittance or diffuse reflection of a material surface through a turbid medium, which is affected by the background color[24]. More translucent material allows better light transmission, resulting in better conversion and, consequently, greater hardness[24].

Bulk-fill resins are more translucent than conventional ones because their composition includes different photoinitiators[25]. Conventional resins have camphorquinone, which has a more yellowish color and makes resins more opaque, complicating the passage of light. Regarding bulk-fill resins, the photoinitiators are phosphine oxides (BAPO and TPOb)[26]. They present lower yellow tonality than materials formulated with camphorquinone and provide higher material translucency, allowing light to penetrate up to the base (6 mm) effectively. This explains why conventional resins should be inserted in increments and bulk-fill resins can be inserted in a single increment[24,26].

According to data provided by the manufacturers, the OPUS and AURA bulk-fill resins have 79% and 81% load weight, similar values. However, the material microhardness assessment with Tukey's test showed a statistically significant difference among the superficial values of this mechanical property. Hence, other components in the composition of materials may contribute to the mechanical properties of bulk-fill resins. Some studies suggest that monomers such as TEGDMA and UDMA are responsible for decreasing material viscosity[12]. In this case, the presence of these components may contribute to the difference in microhardness values presented by the materials investigated. This confirms that the increased amount of load particles increases hardness[20].

The data of the present study corroborate Reis and colleagues (2017)[27], who reported that low-viscosity bulk-fill resins such as the ones analyzed (OPUS and AURA) showed adequate light-curing efficiency up to 4 mm when analyzing the base and top of the material.

A limitation of the present study is that it did not test different conversion degrees using a higher-intensity polymerization light or increasing exposure time to the polymerization light. Then, a suggestion for further new studies to analyze microhardness variations and the achievement of the degree of conversion using a polymerization light with higher intensity or increasing the time of exposure to the polymerization light, aiming to analyze whether a higher degree of conversion and higher microhardness values can be obtained in bulk-fill composites.

Conclusion

Polymerization effectiveness was found in different thicknesses (2 mm, 4 mm, and 6 mm) in both resins studied. This was verified with base/top surface microhardness. However, when comparing the microhardness means between both bulk-fill resins investigated, the microhardness values for the AURA bulk-fill resin were lower than those of the OPUS bulk-fill resin.

Author Contributions

Conceptualization: Araújo FS and Mendonça AAM. **Data curation:** Amaral RC. **Formal analysis:** Takeshita WM. **Funding acquisition:** Amara RC. **Methodology:** Mendonça AAM. **Project administration:** Mendonça AAM and Araújo FS. **Writing - original draft:** Araújo FS, Mendonça AAM, Takeshita WM and Amaral RC. **Writing - review & editing:** Araújo FS, Mendonça AAM, Takeshita WM and Amaral RC.

Conflict of interest

No potential conflict of interest relevant to this article was reported.

Acknowledgements

Data availability

Datasets related to this article will be available upon request to the corresponding author.

References

1. Manhart J, Kunzelmann KH, Chen HY, Hickel R. Mechanical properties and wear behavior of light-cured packable composite resins. Dent Mater. 2000 Jan;16(1):33-40. doi: 10.1016/s0109-5641(99)00082-2.

2. Sakaguchi RL, Peters MC, Nelson SR, Douglas WH, Poort HW. Effects of polymerization contraction in composite restorations. J Dent. 1992 Jun;20(3):178-82. doi: 10.1016/0300-5712(92)90133-w.

3. Krejci I, Planinic M, Stavridakis M, Bouillaguet S. Resin composite shrinkage and marginal adaptation with different pulse-delay light curing protocols. Eur J Oral Sci. 2005 Dec;113(6):531-6. doi: 10.1111/j.1600-0722.2005.00259.x.

4. Rothmund L, Reichl FX, Hickel R, Styllou P, Styllou M, Kehe K, et al. Effect of layer thickness on the elution of bulk-fill composite components. Dent Mater. 2017 Jan;33(1):54-62. doi: 10.1016/j.dental.2016.10.006.

5. Ilie N, Keßler A, Durner J. Influence of various irradiation processes on the mechanical properties and polymerisation kinetics of bulk-fill resin based composites. J Dent. 2013 Aug;41(8):695-702. doi: 10.1016/j.jdent.2013.05.008.

6. Roggendorf MJ, Krämer N, Appelt A, Naumann M, Frankenberger R. Marginal quality of flowable 4-mm base vs. conventionally layered resin composite. J Dent. 2011 Oct;39(10):643-7. doi: 10.1016/j.jdent.2011.07.004.

7. Alrahlah A, Silikas N, Watts DC. Post-cure depth of cure of bulk fill dental resin-composites. Dent Mater. 2014 Feb;30(2):149-54. doi: 10.1016/j.dental.2013.10.011.

8. Winchell H. The knoop microhardness tester as a mineralogical tool. Am Mineral 1945;30:583-95.

9. Leprince JG, Palin WM, Hadis MA, Devaux J, Leloup G. Progress in dimethacrylate-based dental composite technology and curing efficiency. Dent Mater. 2013 Feb;29(2):139-56. doi: 10.1016/j.dental.2012.11.005.

10. El-Safty S, Akhtar R, Silikas N, Watts DC. Nanomechanical properties of dental resin-composites. Dent Mater. 2012 Dec;28(12):1292-300. doi: 10.1016/j.dental.2012.09.007.

11. Karacolak G, Turkun LS, Boyacioglu H, Ferracane JL. Influence of increment thickness on radiant energy and microhardness of bulk-fill resin composites. Dent Mater J. 2018 Mar;37(2):206-13. doi: 10.4012/dmj.2017-032.

12. Silva JM. [Knoop microhardness of single-increment bulk-fill resins]. In: XV Seminário PIBIC/UMESP de Pesquisa - V Seminário PIBITI/UMESP. Methodist University of São Paulo; 2019. Available from: http://www.metodista.br/congressos-cientificos/index.php/CM2018/SEMINARIOPIBIC-BIBITI/paper/view/9934. Portuguese.

13. Esteves JCG. [Analysis of microhardness and degree of conversion of Bulk Fill composite resins] [master's degree]. Lisboa: Faculty of Dental Medicine, University of Lisbon; 2015. Available from: https://repositorio.ul.pt/bitstream/10451/25437/1/ulfmd03064_tm_Joana_Esteves.pdf. Portuguese.

14. Briso AL, Tuñas IT, de Almeida LC, Rahal V, Ambrosano GM. Effects of five carbamide peroxide bleaching gels on composite resin microhardness. Acta Odontol Latinoam. 2010;23(1):27-31.

15. Cruz JMMS. [Analysis of microhardness in different composite resins] [master's degree]. Lisboa: Faculty of Dental Medicine, University of Lisbon; 2013. Available from: https://repositorio.ul.pt/bitstream/10451/25438/1/ulfmd03052_tm_Joana_Cruz.pdf. Portuguese.

16. Fleming GJ, Awan M, Cooper PR, Sloan AJ. The potential of a resin-composite to be cured to a 4mm depth. Dent Mater. 2008 Apr;24(4):522-9. doi: 10.1016/j.dental.2007.05.016.

17. Orłowski M, Tarczydło B, Chałas R. Evaluation of marginal integrity of four bulk-fill dental composite materials: in vitro study. ScientificWorldJournal. 2015;2015:701262. doi: 10.1155/2015/701262.

18. Meereis CT, Leal FB, Lima GS, de Carvalho RV, Piva E, Ogliari FA. BAPO as an alternative photoinitiator for the radical polymerization of dental resins. Dent Mater. 2014 Sep;30(9):945-53. doi: 10.1016/j.dental.2014.05.020.

19. Singhal S, Gurtu A, Singhal A, Bansal R, Mohan S. Effect of different composite restorations on the cuspal deflection of premolars restored with different insertion techniques- an in vitro study. J Clin Diagn Res. 2017 Aug;11(8):ZC67-70. doi: 10.7860/JCDR/2017/20159.10440.

20. Guiraldo RD, Consani S, Consani RL, Berger SB, Mendes WB, Sinhoreti MA. Light energy transmission through composite influenced by material shades. Bull Tokyo Dent Coll. 2009;50(4):183-90. doi: 10.2209/tdcpublication.50.183.

21. Par M, Lapas-Barisic M, Gamulin O, Panduric V, Spanovic N, Tarle Z. long term degree of conversion of two bulk-fill composites. Acta Stomatol Croat. 2016 Dec;50(4):292-300. doi: 10.15644/asc50/4/2.

22. Ilie N. Impact of light transmittance mode on polymerisation kinetics in bulk-fill resin-based composites. J Dent. 2017 Aug;63:51-59. doi: 10.1016/j.jdent.2017.05.017.

23. Arimoto A, Nakajima M, Hosaka K, Nishimura K, Ikeda M, Foxton RM, et al. Translucency, opalescence and light transmission characteristics of light-cured resin composites. Dent Mater. 2010 Nov;26(11):1090-7. doi: 10.1016/j.dental.2010.07.009.

24. Ilie N, Bucuta S, Draenert M. Bulk-fill resin-based composites: an in vitro assessment of their mechanical performance. Oper Dent. 2013 Nov-Dec;38(6):618-25. doi: 10.2341/12-395-L.

25. Costa DD. Influence of translucency and type of composite resin particles on microhardness [course conclusion work]. Dentistry Course, Health Sciences Center, Federal University of Santa Catarina; 2011. Available from: https://repositorio.ufsc.br/handle/123456789/121058.

26. Caneppele TMF, Bresciani E. [Bulk-fill resin composites – The state-of-the-art]. Rev Assoc Paul Cir Dent.2016;70(3):242-8. Portuguese.

27. Reis AF, Vestphal M, Amaral RCD, Rodrigues JA, Roulet JF, Roscoe MG. Efficiency of polymerization of bulk-fill composite resins: a systematic review. Braz Oral Res. 2017 Aug;31(suppl 1):e59. doi: 10.1590/1807-3107BOR-2017.vol31.0059.