Journal of Advanced Medical and Dental Sciences Research

@Society of Scientific Research and Studies

Journal home page: www.jamdsr.com doi: 10.21276/jamdsr

(e) ISSN Online: 2321-9599; (p) ISSN Print: 2348-6805 SJIF (Impact factor) 2017= 6.261; Index Copernicus value 2016 = 76.77

Original Article

Microhardness of Flowable Bulk-Fill Composite Materials

Mayyadah Almozainy

Lecturer at Restorative Dental Science Department, King Saud University, Riyad, Saudi Arabia

ABSTRACT:

Purpose: To evaluate the curing efficiency of various flowable bulk fill resin materials using Vickers hardness measurements and compare them to conventional resin-based composite materialaccording to the manufacturer recommendation time. **Materials and method:** Four composite materials were used: Tetric N-Ceram, Tetric N-flow Bulk fill, Filtek bulk fill flowable composite and Surefil SDR bulk fill flowable. Six discs of each material were prepared and light polymerized according to the manufacturer recommendation with Bluephase G2 curing unit (Ivoclar Vivadent).Vickers hardness was used to determine microhardness (NOVA 130series, Vickers and Knoop hardness testing instrument) under a 200-gram load and a dwell time of 10 seconds. One-way analysis of variance was used to compare the mean values of the ratio of the bottom and top measurements of tested materials, followed by Tukey's multiple comparison tests. A p-value of <0.05 was used to report the statistical significance. **Results:** The mean value of the ratio of the bottom and top measurement shows highly statistically significant difference among the four materials ($F=6427.77$, $p<0.0001$) (SDR> Filtek bulk fill flowable> Tetric N-flow Bulk fill> Tetric N-ceram). **Conclusion:** Manufacturers' recommendations in regards to curing protocol could result in lower hardness value ratio. Accordingly, the curing time protocol should be longer than that indicated by the manufacturer to achieve 70%- 80% hardness ratio. Subsequently, manufacturers' recommendation regarding the curing time should be reevaluated and updated based on the results of current literature. Although some of the tested materials exhibit high hardness value, it is still recommended to cap the flowable bulk fill materials with 2mm layer of conventional composite to prevent subsequent water sorption of the composite material.

Key words: Composite resins; Bulk-fill; Microhardness.

Received: 20 April 2018 Accepted: 28 April 2018

Corresponding Author: Dr. Mayyadah Almozainy, Lecturer at Restorative Dental Science Department, King Saud University, Riyad, Saudi Arabia

This article may be cited as: Almozainy M. Microhardness of Flowable Bulk-Fill Composite Materials. J Adv Med Dent Scie Res 2018;6(5):36-41.

INTRODUCTION

Since their introduction in the 1960s, resin-based composites showed significant improvements in regards to aesthetics, durability as well as physical and mechanical properties.[1] As a result of these improvements, resin-based composites have become the material of choice over amalgam in posterior restorations. However,they are considered time-consumingbecause of the layering technique used to overcome the polymerization shrinkage and depth of cure issues.[2, 3]Thus, bulk fill resin composites have been introduced to the market with an improved chemical composition that can be curedup to 4mm as a single increment based on the manufacturers' recommendations.[4]

One of theconcerns with bulk fill resin materials is the difficulty of adaptation to internal cavity walls, due to the high viscosity of the paste. To solve this issue, a new class of bulkfill resin materials have been introduced, which is the low-viscosity flowable bulk fill resin materials.[5]The rheology of these materials has changed allowing a better adaption to the cavity walls and improved self-leveling effect in comparison to conventional resin-based materials.[6] This improvement in depth of cure could be explained by many factors such as high translucency, increased photoinitiator content or an additional photoinitiator type.[7]They are recommended to be placed in one layer of 4mm thickness to reduce polymerization stress in low load bearing areas as dentine layer substitute, and it is mandatory to cover them with 2mm layer of conventional resin-based composite, [8, 9] because of their low modulus of elasticity and hardness.[10]

Surface hardness is considered an essential property of any resin-based material, especially when they are used as posterior restorations.[11] Since materials with more surface hardness are more resistant to wear, thus this mechanical property is usually used to characterize the wear resistance of the materials.[12]Surface hardness results could also give good information regarding materials' polishability, abrasive effect on antagonist's teeth,[13, 14] as well as degree of conversion.[15-17]Several contributing factors could influence the hardness of composite resin materials, such as organic matrix composition, type of the filler particles and degree of conversion.[18, 19]

Hardness tests are the most frequently used method to evaluate the curing depth and the polymer cross-linking of dental composites. VariousHardness tests are available; the commonly used one is the Vickers microhardness test (VHN) that is usually used for brittle materials and small film thickness materials.[15, 18, 20-23]

The recommended curing time defers between various materials and different manufacturing companies; dentistsareadvised to follow manufacturers' recommendations in regards to time and intensity of light curing unit. However, some studies have shown that manufacturers' recommendations are insufficient for adequate hardness, especially in bottom service.[24, 25]

Accordingly, the purpose of the present study is to evaluate the curing efficiency of various flowable bulk fill resin materials using Vickers hardness measurements and compare them to conventional resin-based composite material following the manufacturers' recommendation.

MATERIALS AND METHOD

Three commercially available flowable bulk fill resin materials served asthe test groups, and a nanohybrid resin-based composite servedas the control group. Product specifications are presented in table (1).

Six discs of each material were prepared using a custom-madecylindrical mold with 5mm diameter and 4mm height for the bulk fill materials and 2mm height for the nanohybrid composite. The mold was placed over a thick glass slide, and the composite resins were packed as one increment, a myler strip and a glass slide were placed over the specimens to ensure a smooth surface, so finishing or polishing following polymerizationis not needed. Each sample was light polymerized according to the manufacturer recommendation with Bluephase G2 curing unit (Ivoclar Vivadent, Schaan, Liechtenstein) in a high-intensity mode with an irradiance of 1200 mW/cm2. The distance between the light source and the material was constant throughout the experiment (1mm), which represented the thickness of the glass slide. The samples were dry stored in a light-proof bottle for 24 hours in an incubator at 37^0 C to complete the polymerization process.

Vickers hardness was used to determine microhardness of each material at the top and the bottom surfaces of each specimen using (NOVA 130series, Vickers and Knoop hardness testing instrument) under a 200-gram load and a dwell time of 10 seconds. Three indentations with the random distance of 1mm were takenfromthe top, and bottom surfaces of each disc and the mean value were calculated (n=18 top and n=18 bottom). The microhardness was determined by measuring the diameters of indentation, which was produced by the pyramidal square-base diamond indenter. The mean bottom/top ratio was calculated by dividing VHN of the bottom surface by VHN of the top surface.

Table 1: Products specification of investigated materials

Material	Resin	Filler	Photoinitiator	Filler % by weight / (volume)	Curing time	manufacturer
Surefil SDR bulk fill flowable	Urethane di-methacrylate resin	(UDMA) Barium and strontium aluminofluoro silicate glasses	Camphoroquinone	68% wt / 45% vol	20 Sec.	Dentsply
Filtek bulk fill flowable composite	BisGMA, BisEMA, Procry-lat, UDMA	Zirconia or silica, ytterbium trifluoride	Camphoroquinone	64.5 wt% / 42.5%vol	20 Sec.	3M ESPE
Tetric N-Flow Bulk fill	monomethacrylate s and dimethacrylates	barium glass, ytterbium trifluoride, and copolymers	Ivocerin	68.2 wt% / 46.4%vol	10 Sec.	Ivoclar Vivadent
Tertric N-Ceram	BisGMA, UDMA, TEGDMA, EthoxylatedBis-EMA	Barium aluminium silicate glass, ytterbium trifluoride, mixed oxide, Prepolymer	Camphoroquinone	80-81 wt.% / 55–57 vol.%	10 Sec.	Ivoclar Vivadent

Data Analysis:
Data were analyzed using SPSS Pc+ 21.0 version (IBM Inc., Chicago, USA) statistical software. Descriptive statistics (mean and standard deviation) were used to describe the values of top and bottom measurements and its ratio across the four materials. Student's paired t-test was used to compare the mean values of top and bottom readings in each of the four materials (SDR, Filtek bulk fill flowable, Tetric N-Flow Bulk fill and Tetric N-ceram). One-way analysis of variance was used to compare the mean values of the ratio of the bottom and top measurements of tested materials, followed by Tukey's multiple comparison tests. A p-value of <0.05 was used to report the statistical significance of results.

RESULTS:
The mean comparison values of top and bottom measurements, which were observed for each of the four composite resin materials, show highly statistically significant difference between the mean measurement of the top and bottom side of all the four materials. Out of the two measurements, the mean value of topside of the material is statistically significantly higher than the mean value of bottom side of the material across all the four materials. Out of the four materials, the mean difference for the Tetric N-Ceram material is significantly higher (22.91) followed by Tetric N-Flow Bulk fill (15.93), Filtek bulk fill flowable (5.12) and SDR (2.32). (Table 2)
The comparison of the mean value of the ratio of the bottom and top measurement values (bottom: top) shows highly statistically significant difference in the mean values of the ratio among the four materials (F=6427.77, p<0.0001). By using the multiple comparisons among the four materials, it was observedthat the mean value of ratio with SDR material is significantly higher followed by; Filtek bulk fill flowable, Tetric N-flow Bulk fill and Tetric N-ceram. This indicates that the mean difference between the top and the bottom measurement value of SDR is smaller, whereas it is larger in Tetric N-ceram, Tetric N-Flow Bulk fill and also in Filtek bulk fill flowable materials. (Table 3)

Table 2: Comparison of mean values of four type of Flowable Bulk-fill resin materials of the top and bottom measurements

Type of material	Top Mean (Sd.,)	Bottom Mean (Sd.,)	Mean Difference	t-value	p-value	95% CI for the difference of mean
SDR	30.59(0.23)	28.27(0.19)	2.32	34.96	<0.0001	2.19,2.46
Filtek bulk fill flowable	30.49(0.32)	25.37(0.22)	5.12	45.89	<0.0001	4.88.5.36
Tetric N-Flow Bulk fill	42.27(0.22)	26.33(0.24)	15.93	187.06	<0.0001	15.75,16.11
Tetric N-ceram	50.39(0.20)	27.48(0.24)	22.91	328.31	<0.0001	22.76,23.05

Table 3: Comparison of mean values of the ratio of bottom: top measurements among the four types of composite resin materials

Type of material	Mean (sd.,) of bottom: top*	F-value	p-value
SDR	0.924(0.008)	6427.77	<0.0001
Filtek bulk fill flowable	0.832(0.014)		
Tetric N-Flow Bulk fill	0.623(0.007)		
Tetric N-ceram	0.545(0.004)		

*By multiple comparison tests: Each material ratio is different with each other

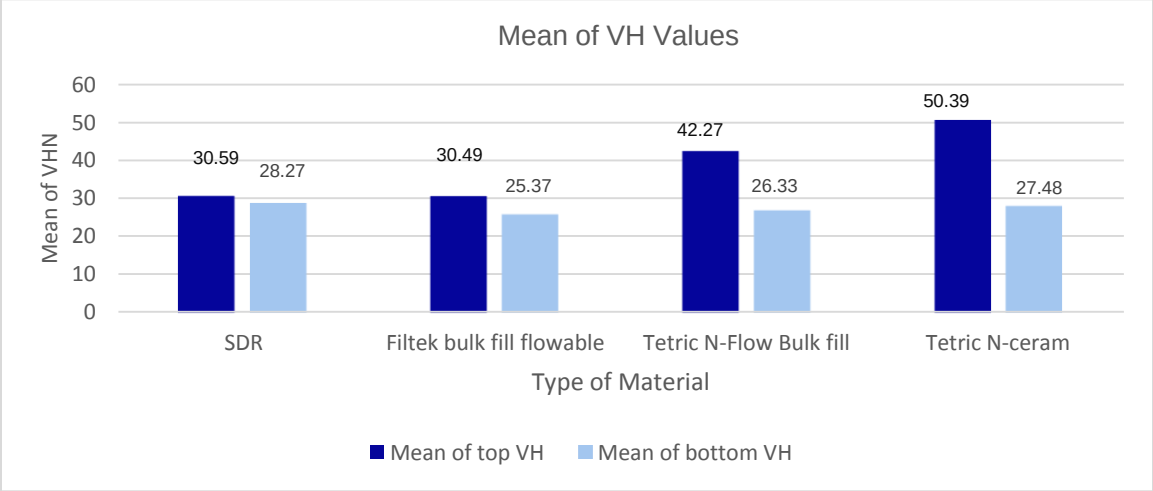

Figure 1 Mean of hardness value of top and bottom surfaces.

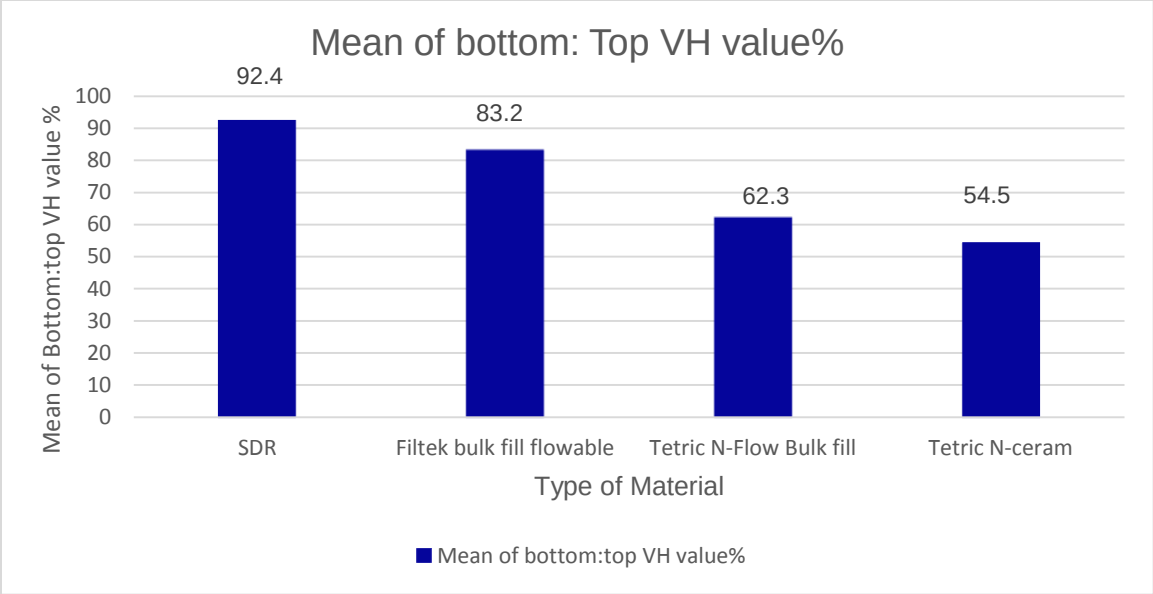

Figure 2 Mean of hardness value ratio.

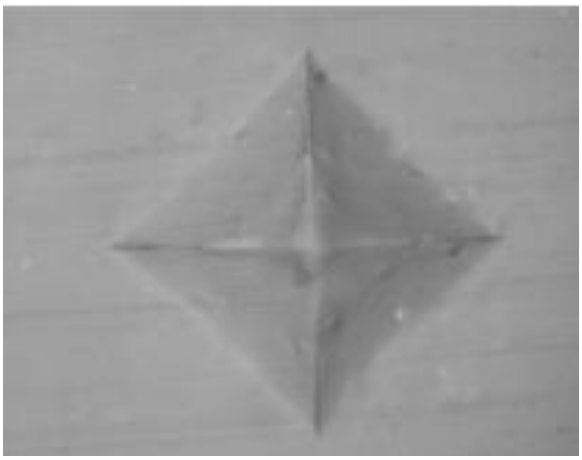

Figure 3 VH indentation on the top surface of SDR

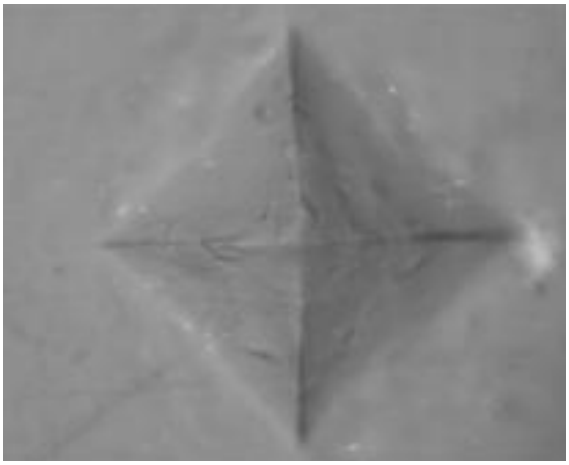

Figure 4 VH indentation on the bottom surface of SDR

DISCUSSION

Hardness is often expressed in percentage; the surface hardness is always compared to 100%, which represents the maximum surface hardness. Experience has shown that the simple hardness measures (top and bottom) correspond well to the more thorough hardness profile measurements.[26]Enamel and Dentine Vickers hardness values have been stated as 348 VHN and 80 VHN respectively.[27]Restorative materials has to have hardness value at least similar to dentine hardness throughout the depth of the restoration to ensure an optimal clinical performance.[20] Previous studies measuring Vickers hardness ratio have reported that if the tested resin-based composite materials reached 80% using the bottom to top surface Vickers hardness ratio (%), they are considered adequately cured.[28, 29]

In this study, although Tetric N-Ceram and Tetric N-Ceram bulk fill showed the highest mean hardness values at their top surface, they failed to reach Vickers hardness ratio of 80% (VH of 54.5% and 62.3% respectively) when the manufacturers' recommendation was followed in regards to the curing protocol. These findings are in agreement with Aldossary et al., results, where the tested samples were cured for 10 seconds, failed to reach the accepted hardness ratio.[24]

Another study has also reported that a prolonged polymerization time of 30 secondsin comparison to the manufacturers' recommendation of 10–20 seconds may improve bulkfill materials performance especially hardness value, without a significant simultaneous increase of polymerization volume shrinkage.[30]Moreover,Cohen et al., reported thatif the curing time indicated by the manufacturer was increased by 5-to 20-fold longer, 70%-

80% bottom-surface hardness will be achieved.[25]Osternack et al., also suggested using a longer curing time to increase the energy densityat the bottom surface and increased the degree of conversion.[22]

In the current study, the highest Vickers hardness ratio was observed with Surefil SDR bulk fill flowable followed by Filtek Bulk fill flowable composite samples (92.4% and 83.2% respectively), these findings were in accordance with results from a previous study where they reported that Surefil SDR bulk fill resin has better depth of cure (3.89 mm (±0.103)) and degree of conversion(78.51 (±47.8))compared to (3.54 mm (±0.129)) depth of cure and (39.8 (±5.2)) degree of conversion for Filtek Bulk fill flowable.[31]The superior performance of the SDR could be due to the fact that it contains a patented modified UDMA, which is claimed to reduce polymerization shrinkage, shrinkage stress and improve the degree of conversion.[32]Another contributing factor could be the translucent matrix being highly conducible to light transmission.[33]Moreover, the manufactures stated that "the unique combination of high glass filler loading with SDR resin provides high depth of cure and proper rheology for self-leveling characteristic for optimum adaptation and marginal integrity. Thiswas confirmed by other studies showing significant lower polymerization stress values for Surefil SDR flow".[9]Similarly, with Filtek Bulk fill flowable composites the high Vickers hardness ratio could be a result of containing a proprietary monomer analogous to Bis-GMA and patented as Procrylat resin. It was found that these modified monomers had altered polymerization kinetics and delayed the monomer conversion.[5]

Although SDR and Filtek Bulk fill flowable composites reported high Vickers hardness ratios at the recommended 4mm thickness, it is still recommended that Flowable bulk fill resin composite materials should be capped with 2mm thick layer of conventional non flowableresin-based composites.[32] The importance of this step is not only related to the surface hardness but also to prevent subsequent water sorption of the composite material.[34]Previous research indicated that the composites intended for bulk fill, including SDR, are more susceptible to water deterioration in comparison with conventional composites, causing creeping deformation of the composites.[35]

Despite the fact that the tested flowable bulk fill resin-based composite materials do not differ much in filler loading, they showed different Vickers hardness ratios. This could be related to the fact that surface hardness, as well as some physical and mechanical properties, are significantly affected by many factors such as mass fractions [17, 36, 37], particle shape and density, monomer type, ratio, degree of polymers crosslinking, and photoinitiators.[36, 38, 39]

In conclusion, the results of the current study indicated that manufacturers' recommendations in regards to curing protocol of resin-based composite could result in lower hardness value ratio. Accordingly, the curing time protocol should be longer than that indicated by the manufacturer to achieve 70%- 80% bottom-surface hardness in relation to the top. Subsequently, manufacture recommendation regarding the curing time should be reevaluated and updated based on the results of current literature.

Although some of the tested materials exhibit high hardness value, it is still recommended to cap the flowable bulkfillmaterials with 2mm layer of conventional composite.

ACKNOWLEDGEMENT

The author would like to express the deepest appreciation to Professor NahidAshri, Department of Periodontics and Community Dentistry, College of Dentistry, King Saud University for her support in executing this study and her advice in writing the manuscript.

REFERENCES

1. Bowen, R., Dental filling material comprising vinyl silane treated fuse silica and a binder consisting of the reaction product of Bis phenol and glycidyl acrylate. 1962: United States.
2. Versluis, A., et al., Does an incremental filling technique reduce polymerization shrinkage stresses? J Dent Res. 1996 Mar;75(3):871-8. doi: 10.1177/00220345960750030301., 1996.
3. Roulet, J.F., Benefits and disadvantages of tooth-coloured alternatives to amalgam. J Dent. 1997 Nov;25(6):459-73., 1997.
4. Ilie, N., A. Kessler, and J. Durner, 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. Epub 2013 May 21., 2013.
5. Par, M., et al., Raman spectroscopic assessment of degree of conversion of bulk-fill resin composites--changes at 24 hours post cure. Oper Dent. 2015 May-Jun;40(3):E92-101. doi: 10.2341/14-091-L. Epub 2014 Oct 2., 2015.
6. Ilie, N., S. Bucuta, and M. Draenert, 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. Epub 2013 Apr 9., 2013.
7. Moorthy, A., et al., Cuspal deflection and microleakage in premolar teeth restored with bulk-fill flowable resin-based composite base materials. J Dent. 2012 Jun;40(6):500-5. doi: 10.1016/j.jdent.2012.02.015. Epub 2012 Mar 3., 2012.
8. Burgess, J. and D. Cakir, Comparative properties of low-shrinkage composite resins. Compend Contin Educ Dent. 2010 May;31 Spec No 2:10-5., 2010.
9. Ilie, N. and R. Hickel, Investigations on a methacrylate-based flowable composite based on the SDR technology. Dent Mater. 2011 Apr;27(4):348-55. doi: 10.1016/j.dental.2010.11.014. Epub 2010 Dec 30., 2011.
10. Jun, S.K., et al., Investigation of the correlation between the different mechanical properties of resin composites. Dent Mater J. 2013;32(1):48-57., 2013.
11. Phillips', A.K., science of dental materials. 12th ed. 2013: Elsevier Inc.

12. Mandikos, M.N., et al., A comparison of the wear resistance and hardness of indirect composite resins. J Prosthet Dent. 2001 Apr;85(4):386-95. doi: 10.1067/mpr.2001.114267., 2001.
13. Marovic, D., et al., Degree of conversion and microhardness of dental composite resin materials. . J Mol Struct, 2012. **1044**: p. 299-304.
14. Moraes, L.G., et al., Infrared spectroscopy: a tool for determination of the degree of conversion in dental composites. J Appl Oral Sci. 2008 Mar-Apr;16(2):145-9., 2008.
15. DeWald, J.P. and J.L. Ferracane, A comparison of four modes of evaluating depth of cure of light-activated composites. J Dent Res. 1987 Mar;66(3):727-30. doi: 10.1177/00220345870660030401., 1987.
16. Santos, G.B., et al., Composite depth of cure obtained with QTH and LED units assessed by microhardness and micro-Raman spectroscopy. Oper Dent. 2007 Jan-Feb;32(1):79-83. doi: 10.2341/06-26., 2007.
17. Knobloch, L.A., et al., Hardness and degree of conversion of posterior packable composites. Operative dentistry, 2004. **29**(6): p. 642-649.
18. Poggio, C., et al., Evaluation of Vickers hardness and depth of cure of six composite resins photo-activated with different polymerization modes. Journal of Conservative Dentistry : JCD, 2012. **15**(3): p. 237-241.
19. Lucey, S., et al., Effect of pre-heating on the viscosity and microhardness of a resin composite. J Oral Rehabil. 2010 Apr;37(4):278-82. doi: 10.1111/j.1365-2842.2009.02045.x. Epub 2009 Dec 29., 2010.
20. Bouschlicher, M.R., F.A. Rueggeberg, and B.M. Wilson, Correlation of bottom-to-top surface microhardness and conversion ratios for a variety of resin composite compositions. Oper Dent. 2004 Nov-Dec;29(6):698-704., 2004.
21. Ferracane, J.L., Developing a more complete understanding of stresses produced in dental composites during polymerization. Dent Mater. 2005 Jan;21(1):36-42. doi: 10.1016/j.dental.2004.10.004., 2005.
22. Osternack, F.H., et al., Impact of refrigeration on the surface hardness of hybrid and microfilled composite resins. Braz Dent J. 2009;20(1):42-7., 2009.
23. Torres, C.R.G., et al., Influence of Concentration and Activation on Hydrogen Peroxide Diffusion through Dental Tissues In Vitro. The Scientific World Journal, 2013. **2013**: p. 5.
24. Aldossary, M., M. Roebuck Elizabeth, and A. Santini, Bulk Fill Resin Composite Materials Cured with Single-Peak versus Dual-Peak LED LCUs, in Acta Medica Marisiensis. 2016. p. 5.
25. Cohen, M.E., et al., Statistical estimation of resin composite polymerization sufficiency using microhardness. Dent Mater, 2004. **20**(2): p. 158-66.
26. Pilo, R. and H.S. Cardash, Post-irradiation polymerization of different anterior and posterior visible light-activated resin composites. Dent Mater. 1992 Sep;8(5):299-304., 1999.
27. Chinelatti, M.A., et al., EVALUATION OF THE SURFACE HARDNESS OF COMPOSITE RESINS BEFORE AND AFTER POLISHING AT DIFFERENT TIMES. Journal of Applied Oral Science, 2006. **14**(3): p. 188-192.
28. Alrahlah, A., N. Silikas, and D.C. Watts, 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. Epub 2013 Nov 20., 2013.
29. Bucuta, S. and N. Ilie, Light transmittance and micro-mechanical properties of bulk fill vs. conventional resin based composites. Clin Oral Investig. 2014 Nov;18(8):1991-2000. doi: 10.1007/s00784-013-1177-y. Epub 2014 Jan 11., 2014.
30. Zorzin, J., et al., Bulk-fill resin composites: polymerization properties and extended light curing. Dent Mater. 2015 Mar;31(3):293-301. doi: 10.1016/j.dental.2014.12.010. Epub 2015 Jan 9., 2015.
31. Yokesh, C.A., et al., Comparative Evaluation of the Depth of Cure and Degree of Conversion of Two Bulk Fill Flowable Composites. J Clin Diagn Res. 2017 Aug;11(8):ZC86-ZC89. doi: 10.7860/JCDR/2017/28004.10444. Epub 2017 Aug 1., 2017.
32. dentsply-international. Surefil SDR. Flow, product Brochure 2011 [cited 2018 8-2].
33. Lassila, L.V., et al., Translucency of flowable bulk-filling composites of various thicknesses. Chin J Dent Res. 2012;15(1):31-5., 2012.
34. Jang, J.H., S.H. Park, and I.N. Hwang, Polymerization shrinkage and depth of cure of bulk-fill resin composites and highly filled flowable resin. Oper Dent. 2015 Mar-Apr;40(2):172-80. doi: 10.2341/13-307-L. Epub 2014 Aug 19., 2015.
35. El-Safty, S., N. Silikas, and D.C. Watts, Creep deformation of restorative resin-composites intended for bulk-fill placement. Dent Mater. 2012 Aug;28(8):928-35. doi: 10.1016/j.dental.2012.04.038. Epub 2012 May 30., 2012.
36. Leprince, J., et al., Investigating filler morphology and mechanical properties of new low-shrinkage resin composite types. J Oral Rehabil. 2010 May 1;37(5):364-76. doi: 10.1111/j.1365-2842.2010.02066.x. Epub 2010 Feb 19., 2010.
37. Chung, K.H. and E.H. Greener, Correlation between degree of conversion, filler concentration and mechanical properties of posterior composite resins. J Oral Rehabil. 1990 Sep;17(5):487-94., 1990.
38. Hahnel, S., et al., The influence of monomeric resin and filler characteristics on the performance of experimental resin-based composites (RBCs) derived from a commercial formulation. Dent Mater. 2012 Apr;28(4):416-23. doi: 10.1016/j.dental.2011.11.016. Epub 2011 Dec 20., 2012.
39. Mobarak, E., et al., Effect of LED light-curing on the relative hardness of tooth-colored restorative materials. Oper Dent. 2009 Jan-Feb;34(1):65-71. doi: 10.2341/08-38., 2009.

Source of support: Nil **Conflict of interest:** None declared