IDR International Dental Research

Original Article

Investigation of nanohardness values of dual-cure, bulk-fill composite resins at different time intervals

Eda Özdemir[1], Behiye Bolgül[1]

[1] Private Dentaliva Oral and Dental Health Clinic, Department of Pedodontics, Istanbul, Turkey
[2] Hatay Mustafa Kemal University, Faculty of Dentistry, Department of Pedodontics, Hatay, Turkey

Correspondence:

Dr. Eda ÖZDEMİR
Private Dentaliva Oral and Dental Health Clinic, Department of Pedodontics, Istanbul Turkey.
E-mail:edaozdemir@hotmail.com

Received: 15 January 2021
Accepted: 3 May 2021

Access Online

DOI: 10.5577/intdentres.2021.vol11.suppl1.4

Abstract

Aim: The aim of this study was to evaluate the surface hardness of dual-cure, bulk-fill composite resins at different depths and time intervals using nanoindentation.
Methodology: In the study, a comparison was conducted of the nanohardness values of Fill-Up, HyperFIL, and Cention N dual-cure, bulk-fill composite resins in the experimental group and Reveal HD, a light-cured, bulk-fill composite, in the control group. The dual-cure, bulk-fill composites used in the experimental group were divided into two subgroups. The first group was polymerized with an LED light source, and the second group was chemically polymerized in line with the manufacturer's instructions. The dual-cure composite resins were prepared in two different depths, 5 mm and 10 mm. In the control group, specimens with a depth of 5 mm were prepared, and their polymerization was provided by an LED light source. The nanohardness values of the lower and upper surfaces of the samples were measured using the Hysitron TI 950 Triboindenter 24 hrs and 7 days after polymerization.
Results: Following statistical evaluations, no statistically significant difference was found between the nanohardness values measured 24 hrs and 7 days after light or chemical polymerization of the dual-cured, bulk-fill composite resins. It was observed that light polymerization of HyperFIL and Fill-Up dual-cure, bulk-fill composite resins had no effect on nanohardness values. When Cention N dual-cure, bulk-fill composite resin was polymerized with a light source, higher values of nanohardness were obtained.
Conclusion: It was determined that the investigated dual-cure, bulk-fill composites reached their final hardness after 24 hrs. In all groups, it was observed that the highest nanohardness values belonged to Cention N for both polymerization methods.

Keywords: dual-cure, bulk-fill, composite resin, nanohardness, pediatric dentistry

How to cite this article: Özdemir E, Bolgül B. Investigation of nanohardness values of dual cure bulk-fill composite resins at different time intervals. Int Dent Res 2021;11(Suppl.1):19-25. https://doi.org/10.5577/intdentres.2021.vol11.suppl1.4

Introduction

In recent years, new dual-cure, bulk-fill composites designed for direct restorations have been developed and are claimed to provide unlimited depth of polymerization when placed in a single layer. These dual-cure, bulk-fill composites contain components that can be polymerized both chemically and using light. The light-cure component provides rapid and initial curing of the top layers of the composite and stabilizes restorations; then, the deeper layers of the composite that receive insufficient light are polymerized using a slower chemical-cure reaction (1). Restorative materials are exposed to many physical and chemical effects in the mouth (2). Their hardness plays an important role in maintaining the durability of materials against occlusal forces that occur, especially during mastication (3). The high surface hardness values of composite resins increase the resistance of the materials to abrasion and scratches and prevent deformation when force is applied, which has a positive effect on the clinical success and life of restorations (4).

Most studies are based on hardness tests measured at a microscopic level (5). Due to advancing technology, a new approach, nanoindentation, was available to be used in this study to obtain the hardness values of the materials at the nano level.

While dual-cure, bulk-fill composites allow for shorter treatment times in children, disabled patients, and patients with compliance problems, the application procedures require less technical precision, making it easier for physicians to work. However, there are very few studies in the literature investigating the mechanical and physical properties of dual-cure, bulk-fill composite resins. Therefore, in our study, the aim was to evaluate the nanohardness values of the upper and lower surfaces of three dual-cure, bulk-fill composite resins of different brands at two different time intervals, 24 hrs and 7 days after polymerization.

Materials and Methods

Ethics committee approval was received for this study from Hatay Mustafa Kemal University (Decision number: 2019/18).

In this study, the nanohardness values of Fill-Up (Coltene/Whaledent AG, Altstätten, Switzerland), HyperFIL (Parkell, Inc., Edgewood, NY, USA), Cention N (Ivoclar/Vivadent, Schaan, Liechtenstein) dual-cure, bulk-fill composite resins in the experimental group and Reveal HD (Bisco, IL, USA), a light-cure bulk-fill composite, in the control group were compared. The contents of the composite resins used in the study are shown in Table 1. The manufacturers of these three dual-cure, bulk-fill composite resins claim that they provide unlimited polymerization depth due to the dual-curing properties of the materials and that restorations can be finished directly with these materials without the need for any capping layer. Reveal HD can be applied to the cavity in 5-6 mm layers.

Table 1. The contents of the composite resins used in the study and their manufacturers

Material	Type	Content / Filler Type	Filler Ratio	Particle Size	Manufacturer
Fill-Up	Dual-cure bulk-fill composite	TMPTMA, UDMA, Bis-GMA, TEGDMA Glass, Amorphous Silica, Zinc oxide	weight: %65 volume: %49	2 µm	Coltene/ Whaledent AG
HyperFIL	Dual-cure bulk-fill composite	Bis-EMA, UDMA and other dimethacrylate monomers Barium glass and silica	weight: %70-75	15 nm 3.5 µm	Parkell, Inc.
Cention N	Dual-cure bulk-fill composite	UDMA and other dimethacrylates Calcium fluorosilicate glass, barium glass, calciumyum barium aluminum fluorosilicate glass, iterbi-yum trifluoride	weight: %78,4 volume: %57,6	0.1-7 µm	Ivoclar Vivadent; Schaan
Reveal HD	Light-cure bulk-fill composite	UDMA, Bis-GMA, ytterbium trifluoride,	Not obtained from company	Not obtained from company	Bisco

A total of 65 composite samples from the experimental and control groups were prepared in our study (Fig.1). Each dual-cure, bulk-fill composite used in the experimental group was divided into two subgroups. The first group was polymerized only on the upper surfaces with a 3M ESPE Elipar DeepCure-L (St. Paul, MN, USA) LED light source, and the second group was chemically (self) polymerized according to the manufacturer's instructions. Each of these subgroups consisted of a total of 10 specimens prepared using cylindrical plexiglass molds at two different depths, 5 and 10 mm (n = 5). In the control group, Reveal HD was polymerized with an LED light source using molds of a 5 mm depth (n=5). The polymerized samples were polished using polishing discs (Optidisc, Kerr) containing aluminum oxide particles. Composite samples, after polishing processes, were kept in distilled water at 37 ºC for 24 hrs.

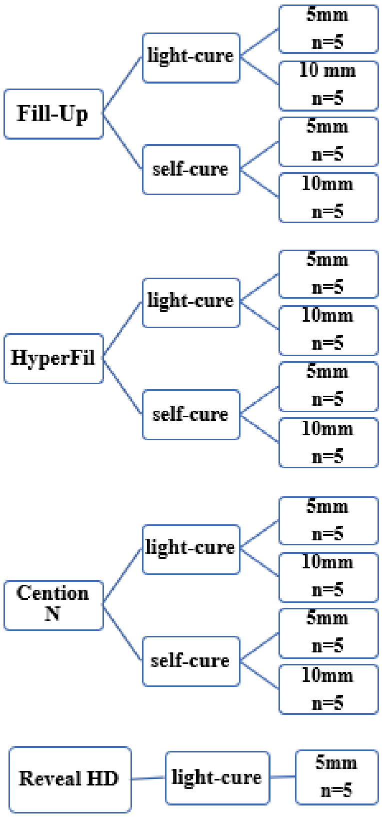

Figure 1. A total of composite samples for the experimental and control groups

The first measurements were made 24 hrs after polymerization of the samples using a Hysitron TI 950 Triboindenter (Hysitron, USA) device to apply 1000 µN force to five different points on each sample, from the upper and lower surfaces. Second measurements were made 7 days later. The average of the five measurements made of each sample surface was calculated and the upper and lower surface hardness values of the samples were determined.

Statistical analysis

The data in our study were analyzed using the SPSS software version 21 (IBM Corp., Armonk, NY, USA) with 95% confidence. In the expression of the data, mean standard deviation and minimum and maximum values were used. A Mann–Whitney U test was used to evaluate independent groups. A Wilcoxon signed-ranks test was used for repeated measurements (hardness 1st day, 7th day). After analyzing measurements from more than two groups using a Kruskal-Wallis test, a Mann–Whitney U test with a Bonferroni correction (p = .05/comparison number) was used for paired comparisons. The significance value for all tests was determined to be .05.

Results

In the study, the difference between the hardness measurements made on the 1st and 7th days of all material groups was not found to be statistically significant. (p>0,05).

The comparison of the upper and lower surface hardness values of the composite samples according to the polymerization types is shown in table 2. No statistically significant difference was found between the hardness values of the Fill-Up composite resin self-cure and light cure material groups. (p>0,05). The surface hardness values of the light cure group were found to be statistically significantly higher than the self-cure group in the measurements made from the lower surfaces on day 7 in the HyperFIL group (p<0,05). In the Cention N group, both the upper and lower surface hardness values of the light cure group were found to be significantly higher than the self-cure group (p<0,05).

The comparison of the lower and upper surface hardness values of the material groups according to the application thickness is shown in table 3. The difference between the values obtained in the hardness measurements of Fill-Up and HyperFIL at 5- and 10-mm thickness was not statistically significant (p> 0.05). The hardness values of the lower surfaces of the 10 mm thick groups of Cention N are significantly lower than the hardness values of the lower surfaces of the 5 mm thick groups (p<0,05).

The comparison of the upper and lower surface nanohardness values of the chemically polymerized groups of the composite resins according to the brands, is shown in table 4. A statistically significant difference was found in the measurements on the 1st and 7th day on the upper surfaces of the materials applied with 5 mm thickness and on the 7th day for 10 mm thickness (p<0,05).

On the lower surface, only the difference between the nanohardness values on day 7 was found to be significant (p<0,05). In paired comparisons, it was found that the surface hardness values of the material groups in which Cention N was used were significantly higher than the other groups.

Table 2. Mean hardness values (GPa) and p values of the upper and lower surfaces of the material groups according to the polymerization patterns.

		Upper Surface			Lower Surface		
		Self Cure	Light Cure	p	Self Cure	Light Cure	p
		Mean ± SD	Mean ± SD		Mean ± SD	Mean ± SD	
Fill-Up 5 mm	Hardness 1st day	0,32 ±0,08	0,39±0,07	0,175	0,25 ±0,05	0,30 ±0,04	0,117
	Hardness 7th day	0,40 ±0,12	0,39±0,12	0,754	0,33 ±0,11	0,29 ±0,08	0,602
Fill-Up 10 mm	Hardness 1st day	0,35 ±0,14	0,35±0,07	0,35	0,25 ±0,09	0,24 ±0,07	0,841
	Hardness 7th day	0,35 ±0,04	0,34±0,03	1	0,24 ±0,03	0,27 ±0,06	0,31
HyperFIL 5 mm	Hardness 1st day	0,33 ±0,15	0,51±0,09	0,05	0,29 ±0,12	0,41 ±0,07	0,142
	Hardness 7th day	0,39 ±0,09	0,50±0,09	0,175	0,27 ±0,06	0,42 ±0,08	0,016*
HyperFIL 10 mm	Hardness 1st day	0,36 ±0,07	0,48±0,08	0,117	0,27 ±0,05	0,35 ±0,10	0,175
	Hardness 7th day	0,41 ±0,05	0,48±0,09	0,173	0,30 ±0,03	0,38 ±0,05	0,008*
Cention N 5 mm	Hardness 1st day	0,58 ±0,14	0,94±0,08	0,009*	0,49 ±0,17	0,89 ±0,11	0,008*
	Hardness 7th day	0,57 ±0,10	0,95±0,10	0,009*	0,46 ±0,05	0,91 ±0,15	0,008*
Cention N 10 mm	Hardness 1st day	0,49 ±0,16	0,84±0,06	0,009*	0,36 ±0,11	0,71 ±0,05	0,008*
	Hardness 7th day	0,63 ±0,09	0,81±0,08	0,016*	0,40 ±0,04	0,69 ±0,07	0,008*

The p value of the groups that make a difference is marked with *

Table 3. Mean hardness (GPa) and p values on the 1st and 7th days according to the application thickness of the upper and lower surfaces of the material groups.

		Upper Surface			Lower Surface		
		5 mm	10 mm	p	5 mm	10 mm	p
		Mean ± SD	Mean ± SD		Mean ± SD	Mean ± SD	
Fill-Up Self Cure	Hardness 1st day	0,32 ±0,08	0,35 ±0,14	0,834	0,25±0,05	0,25 ±0,09	0,465
	Hardness 7th day	0,40 ±0,12	0,35 ±0,04	0,175	0,33±0,11	0,24 ±0,03	0,175
Fill-Up Light Cure	Hardness 1st day	0,39 ±0,07	0,35 ±0,07	0,602	0,30±0,04	0,24 ±0,07	0,117
	Hardness 7th day	0,39 ±0,12	0,34 ±0,03	0,624	0,29±0,08	0,27 ±0,06	0,754
HyperFIL Self Cure	Hardness 1st day	0,33 ±0,15	0,36 ±0,07	0,327	0,29±0,12	0,27 ±0,05	0,462
	Hardness 7th day	0,39 ±0,09	0,41 ±0,05	0,753	0,27±0,06	0,30 ±0,03	0,251
HyperFIL Light Cure	Hardness 1st day	0,51 ±0,09	0,48 ±0,08	0,602	0,41±0,07	0,35 ±0,10	0,834
	Hardness 7th day	0,50 ±0,09	0,48 ±0,09	0,465	0,42±0,08	0,38 ±0,05	0,465
CentionN Self Cure	Hardness 1st day	0,58 ±0,14	0,49 ±0,16	0,249	0,49±0,17	0,36 ±0,11	0,117
	Hardness 7th day	0,57 ±0,10	0,63 ±0,09	0,465	0,46±0,05	0,40 ±0,04	0,075
CentionN Light Cure	Hardness 1st day	0,94 ±0,08	0,84 ±0,06	0,12	0,89±0,11	0,71 ±0,05	0,028*
	Hardness 7th day	0,95 ±0,10	0,81 ±0,08	0,06	0,91±0,15	0,69 ±0,07	0,028*

Table 4. Nanohardness evaluation of self-cure material groups according to brands.

			Fill-Up	HyperFIL	CentionN	p
			Mean ±SD	Mean ± SD	Mean ±SD	
Self Cure Upper Surface	5 mm	Hardness 1th day	0,32±0,08	0,33±0,15	0,58±0,14	0,032*
		Hardness 7th day	0,40±0,12	0,39±0,09	0,57±0,1	0,037*
Self Cure Lower Surface	5 mm	Hardness 1th day	0,25 ± 0,05	0,29±0,12	0,49±0,17	0,113
		Hardness 7th day	0,33 ± 0,11	0,27±0,06	0,46±0,05	0,014*
	10 mm	Hardness 1th day	0,25±0,09	0,27±0,05	0,36±0,11	0,202
		Hardness 7th day	0,24±0,03	0,3±0,03	0,40±0,04	0,003*

The comparison of the nanohardness values of the upper and lower surfaces of the bulk-fill composite groups consisting of Fill-Up, HyperFIL, Cention N, and Reveal HD polymerized with light, according to the brands, is shown in table 5. A statistically significant difference was found between the hardness values of the upper and lower surfaces on day 1 and day 7 of 4 light-polymerized bulk-fill composites ($p<0,05$). As a result of the paired comparisons made to determine the group causing the difference, the surface hardness values of the material groups formed by Cention N were found to be significantly higher than the other groups.

Table 5. Nanohardness evaluation of light cure material groups according to brands.

			Fill-Up	HyperFIL	CentionN	RevealHD	p
			Mean ±SD	Mean ± SD	Mean ±SD	Mean ± SD	
Light Cure Upper Surface	5 mm	Hardness 1th day	0,39±0,07	0,51±0,09	0,94±0,08	0,45±0,1	0,004*
		Hardness 7th day	0,39±0,12	0,5±0,09	0,95±0,1	0,47±0,08	0,007*
Light Cure Lower Surface	5 mm	Hardness 1th day	0,3±0,04	0,41±0,07	0,89±0,11	0,27±0,14	0,003*
		Hardness 7th day	0,29±0,08	0,42±0,08	0,91±0,15	0,29±0,11	0,004*
	10 mm	Hardness 1th day	0,24±0,07	0,35±0,1	0,71±0,05		0,006*
		Hardness 7th day	0,27±0,06	0,38±0,05	0,69±0,07		0,002*

Discussion

Dual cure bulk-fill composite resins; It has important advantages such as mass placement, savings in clinical time, successful polymerization in deep cavities due to chemical polymerization, and lower shrinkage stress (6).

Studies have shown that most of the polymerization of light-cured composite resins occurs within the first few minutes or one hour after irradiation, and then the polymerization reaction continues with a slow increase for up to 24 hours or longer (7). For this reason, it is stated that in order to obtain accurate results in hardness measurements, it is required to wait at least 24 hours (8).

Mohamad et al. reported that the hardness values obtained in a study comparing the nanohardness values of composite resins immediately after the light application and on the 7th day were higher on the 7th day (9). In the studies of Amalavathy et al. comparing the nanohardness values of Cention N and Equia Forte Fil on the 1st and 28th days, Cention N showed significantly higher nanohardness values on the 28th day compared to the 1st day (10). In contrast to these studies, in our study, the difference between hardness measurements at 24 hours and 1 week was not found to be statistically significant in all groups. However, although the difference was not statistically significant in our study, we can say that the hardness values were higher in the self-cure groups on the 7th day.

Despite the presence of dual curing mode in dual cure bulk-fill composites, several studies have shown that most dual cure materials do not achieve similar hardness values when light-activated or self-polymerization is achieved (11). In their study, Rueggeberg, and Caughman concluded that for the polymerization of dual-cured resins, light polymerization is also necessary, rather than just waiting for self-curing (12). In our study, while the hardness values of the light-polymerized group of Cention N were higher than the chemically polymerized group, there was no difference between the hardness values of Fill-Up, HyperFIL's self-cure, and light cure groups on the 1st day.

For both groups of Fill-Up and HyperFIL dual cure composites chemically and light polymerized, no statistically significant difference was found between the lower surface hardness values of the 5- and 10-mm thickness. Hardness values obtained for Fill-Up and HyperFIL were not affected by layer thickness. In Cention N dual cure bulk-fill composite, in the light polymerized group, the bottom surface hardness values of the 5 mm thick samples are higher than the 10 mm. A more uniform polymerization was obtained in the chemical polymerization of Cention N, and it was determined that the hardness values in the case of light activation of the polymerization were higher than the values obtained in chemical polymerization.

The surface hardness values of composite resins are largely dependent on the shape, size, weight, and volume of the filler particles they contain (13). In their study, Flury et al. stated that there is a positive correlation between the surface hardness and the filler amounts of composite resins (14). Among the four bulk-fill composite resins we evaluated in our study, Cention N showed the highest surface hardness values in all groups. According to the results we obtained from our study, the order of surface hardness values of bulk-fill composite resins is Cention N> HyperFIL> Fill-Up in the self-cure group. In the light cure group, Cention N> HyperFIL> Reveal HD> Fill-Up. According to the results of this study, Cention N dual cure bulk-fill composite resin has high hardness values and contains alkaline fillers, and it is the highest inorganic composite among the bulk-fill composites we evaluated in the study. We think this is since it has a high filler content ratio.

Conclusions

All values obtained shown that dual-cure bulk-fill composite resins reach their maximum hardness after 24 hours when polymerized both by light and chemically, and after that, there is no change in hardness values. It was concluded that light curing of HyperFIL and Fill-Up dual-cure bulk-fill composite resins had no effect on the hardness values, except that the materials polymerized rapidly and facilitated the completion of the restoration and polishing processes. Although the manufacturers of Cention N dual-cure bulk-fill composite resin claim that light curing is optional, higher hardness values were obtained when polymerized with a light source.

The main point that restricts our study is the lack of literature investigating the hardness values of dual-cure bulk-fill composites using the nanoindentation method. More research is needed to evaluate the clinical performance of dual-cure bulk-fill composite resins.

Acknowledgments: This study was presented as a full-text oral presentation at the 1st International Dental Research and Health Sciences Congress held between 20-22 May 2021.

Ethical Approval: Ethics committee approval was received for this study from Hatay Mustafa Kemal University in accordance the World Medical Association Declaration of Helsinki, with the approval number: 2019/18.

Peer-review: Externally peer-reviewed.

Author Contributions: Conception – E.Ö.; Design – E.Ö., B.B.; Supervision – B.B.; Materials – E.Ö.; Data Collection and/or Processing – E.Ö.; Analysis and/or Interpretation – E.Ö., B.B..; Literature Review – E.Ö.; Writer – E.Ö.; Critical Review – B.B.

Conflict of Interest: No conflict of interest was declared by the authors.

Financial Disclosure: This experimental work was supported to %100 by the Scientific Research Projects Coordination of Mustafa Kemal University.Project number: 19.U.017

References

1. Wang R, Liu H, Wang Y. Different depth-related polymerization kinetics of dual-cure, bulk-fill composites. Dental Materials 2019. (Crossref)
2. Yan YL, Kim YK, Kim KH, Kwon TY. Changes in degree of conversion and microhardness of dental resin cements. Operative Dentistry 2010, 35, 203-210. (Crossref)
3. Abed Y, Sabry H, Alrobeigy N. Degree of conversion and surface hardness of bulk-fill composite versus incremental-fill composite. Tanta Dental Journal 2015, 12, 71-80. (Crossref)
4. Anusavice KJ, Shen C, Rawls HR. Phillips' science of dental materials; Elsevier Health Sciences, 2012. pp. 279.
5. Oliver WC, Pharr GM. Measurement of hardness and elastic modulus by instrumented indentation: Advances in understanding and refinements to methodology. Journal of materials research 2004, 19, 3-20. (Crossref)
6. Bolhuis PB, de Gee AJ, Kleverlaan CJ, El Zohairy AA, Feilzer AJ. Contraction stress and bond strength to dentinfor compatible and incompatible combinations of bonding systems and chemical and light-cured core build-up resin composites. Dental Materials 2006, 22, 223-233. (Crossref)
7. Alrahlah A, Silikas N, Watts D. Post-cure depth of cure of bulk fill dental resin-composites. Dental materials 2014, 30, 149-154. (Crossref)
8. Yan Y, Kim Y, Kim K, Kwon T. Changes in degree of conversion and microhardness of dental resin cements. Operative Dentistry 2010, 35, 203-210. (Crossref)
9. Mohamad D, Young R, Mann A, Watts D. Post-polymerization of dental resin composite evaluated with nanoindentation and micro-Raman spectroscopy. Archives of Orofacial Sciences 2007, 2, 26-31.

10. Amalavathy RK, Sahoo HS, Shivanna S, Lingaraj J, Aravinthan S. Staining effect of various beverages on and surface nano-hardness of a resin coated and a non-coated fluoride releasing tooth-coloured restorative material: An in-vitro study. Heliyon 2020, 6, e04345. (Crossref)
11. Kwon TY, Bagheri R, Kim YK, Kim KH, Burrow MF. Cure mechanisms in materials for use in esthetic dentistry. Journal of investigative and clinical dentistry 2012, 3, 3-16. (Crossref)
12. Rueggeberg F, Caughman WF. The influence of light exposure on polymerization of dual-cure resin cements. Operative Dentistry 1993, 18, 48-55.
13. Jain P, Pershing A. Depth of cure and microleakage with high-intensity and ramped resin-based composite curing lights. The Journal of the American Dental Association 2003, 134, 1215-1223. (Crossref)
14. Flury S, Peutzfeldt A, Lussi A. Influence of increment thickness on microhardness and dentin bond strength of bulk fill resin composites. Dental Materials 2014, 30, 1104-1112 (Crossref)