

Investigation of the effect of different application techniques on microhardness in bulk-fill composite resins

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Abstract

Aim: The aim of this study was to investigate the effect of different application techniques on microhardness in one universal resin material and three different bulk-fill composite resin materials.

Materials and Methods: In this study, one universal composite resin (Universal Restorative 200 [U], 3M-ESPE, USA) and three different bulk-fill composite resins (Filtek-One Bulk-Fill Restorative [F]; 3M-ESPE, USA; Tetric Evo-Ceram Bulk-Fill [E]; and Ivoclar-Vivadent, Liechtenstein; Tetric N-Ceram Bulk-Fill [N], Ivoclar-Vivadent, Liechtenstein) materials were used. A total of 60 disk-shaped samples, including 20 samples from each bulk-fill composite resin group, were prepared. The polymerization of the bulk-fill composite resins in one group was completed using a 2 mm (40 sec) + 2 mm (40 sec) incremental technique, and in the other group, it was completed using a 4 mm bulk technique (40 sec). The U composite resin (control group) polymerization was completed using a 2 mm + 2 mm (40 sec) incremental technique (n = 10). The Vickers microhardness (VHN) values of the samples were calculated. The data were analyzed with Kruskal-Wallis and Mann-Whitney U tests ($p < 0.05$).

Results: Higher VHN values were found in the U composite resin compared to those in the bulk-fill composite resins ($p < 0.05$). In the bulk technique, a lower VHN value was observed in the N bulk-fill composite resin than in the control group ($p < 0.05$), while there was no difference between the N and E composite resin groups ($p > 0.05$). A lower N bulk-fill composite resin was observed in the groups in which the incremental technique was applied than in the control group ($p < 0.05$). When the techniques applied to the bulk-fill composite resins were evaluated within themselves, higher VHN values were observed in the E composite resin in the group in which the incremental technique was applied but not in the group in which the bulk was applied.

Conclusion: It was observed that the content differences of composite resins were effective at different VHN values. The U composite resin (control) group showed higher VHN values than all the bulk-fill composite groups.

Keywords: bulk-fill composite resin, bulk technique, incremental technique, microhardness

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Introduction

Composite materials are generally used as tooth-colored materials in dentistry because of their good aesthetic properties, advanced mechanical properties, biocompatibility, and adhesion to the tooth structure (1). The content of composite materials is generally classified according to the filler shape and size, the resin matrix, and the area of use (2). Composite resins differ according to the area of the treated tooth (anterior or posterior), the viscosity of the material (flowable or packable), the application method (incremental or bulk), and the bonding capacity to the tooth structure (self-adhesive composites) (1). Composite resins are typically applied using an incremental technique, and each layer is polymerized separately (3). The incremental technique has disadvantages, including combining layers, the risk of contamination between layers, bonding failures, and a lengthy application time (4).

Bulk-fill composites have been developed in recent years to allow composites to be applied to cavities of greater thickness (5, 6). Bulk-fill composites have been effective in shortening the clinical study period and decreasing polymerization shrinkage due to their placement in a single layer. They also have the important advantages of the prevention of interlayer gap and contamination, good abrasion resistance against chewing forces, surface properties, and color matching (7). Recent advances in composites, especially regarding new monomers, transparency, initiator systems, and filler technology, have led to the production of bulk-fill composites to overcome these disadvantages (8). Differences in the filler properties of composites, such as monomer resin formulations, type, density, particle size, and distribution, can affect the polymerization depth and mechanical properties of composite resins (8, 9). For the mechanical and physical properties (e.g., abrasion resistance and biocompatibility) of resin-based composites to be

sufficient, they must be properly polymerized. Although manufacturers recommend single-layer bulk-fill composite materials to be 4 mm, many clinicians have questioned the acceptability of post-polymerization mechanical properties in clinical use (10).

The purpose of the study was to determine the effect of applying one universal composite and three different bulk-fill composite resin materials with different application techniques (incremental and bulk) on VHN values.

Materials and Methods

Ethics committee approval was received for this study from Necmettin Erbakan University (Decision no: 2020/02-01).

In this study, one universal (Universal Restorative 200, 3M-ESPE, USA) and three different bulk-fill (Filtek One Bulk-Fill Restorative, 3M-ESPE, USA; Tetric EvoCeram Bulk-Fill, Ivoclar Vivadent, Liechtenstein; Tetric N-Ceram Bulk-Fill, Ivoclar Vivadent, Liechtenstein) composite resins were used (Table 1). A total of 70 samples in the form of disks were prepared, including 20 specimens from each bulk-fill composite and 10 specimens from the Universal Restorative 200 composite as the control group. For bulk-fill composite resins, Teflon molds of 4 mm depth and 10 mm diameter (bulk technique) and 4 mm depth and 10 mm diameter (2 mm + 2 mm for the incremental technique) were used for the preparation. While composite resin specimens were prepared, Mylar strips were used to prevent them from sticking to the glass. After the composite was placed using a Mylar strip, the glass was removed and polymerization was completed with an LED light device (Woodpecker LED.E [P], Guilin Woodpecker Medical Instrument Co., Guilin, Guangxi, China).

Table 1. Composition of the composite resins

	Manufacturer	Type	Composition	wt%- vol%	Lot No
Universal Restorative 200	3M Espe, St. Paul, USA	Universal/ microhybrid	BisGMA, UDMA, Bis-EMA, zirkonium/silica, camphorquinone, 0,01-3,5 µm	82% wt 60% vol	N997960
Filtek One Bulk-Fill	3M-ESPE, St. Paul, USA	Bulk-Fill	UDMA, DDDMA, Zirconia/silica (4-20 nm) cluster filler, ytterbium fluoride (100 nm) AUDMA, AFM, and 1, 12-dodecane-DMA, camphorquinone	76.5% wt 58,4% vol	NC6052
Tetric EvoCeram Bulk-Fill	Ivoclar Vivadent, Liechtenstein	Bulk-Fill	Bis-GMA, Bis-EMA, UDMA, barium glass, ytterbium trifluoride, mixed oxide, silica nanohybrid; (17% pre-polymers), Lucirin, Ivocerin, camphorquinone	78-8% wt 62.5% vol	Z0032W

Tetric N-Ceram Bulk-Fill	Ivoclar Vivadent, Liechtenstein	Bulk-Fill	Bis-GMA, Bis-EMA, UDMA bariumglass,ytterbium trifluoride, mixed oxide, and prepolymer; Lucirin, Ivocerin, camphorquinone	75% wt 53% vol	W93899
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The polymerization of the samples was done in the middle mode at 1,200 mW/cm² light (power) intensity. Samples of bulk-fill composite resins were prepared by polymerizing each layer for 40 seconds using the 2 mm + 2 mm layering technique, and by polymerizing the 4 mm as a single layer for 40 seconds using the bulk technique. The samples for the control group were prepared by polymerizing each layer for 20 seconds with the universal composite resin 2 mm + 2 mm incremental technique. After the groups were formed, the specimens were kept in distilled water at 37°C for 24 hours.

Microhardness measurements

The VHN values were calculated using a Vickers hardness tester (LHV 1-D, Bursam NDT, Bursa, Turkey) to measure the microhardness values of the samples. Three measurements were taken from each sample for 10 seconds under a 300 g load, and the average of these measurements was recorded.

Statistical Analysis

The statistical package program SPSS software version 22.0 was used in the analysis of the dataset (IBM Corp., Armonk, NY, USA). The normality of the distribution was first checked with the Kolmogorov-Smirnov test; the data were not normally distributed and represented the median (min-max). Kruskal-Wallis

and Mann-Whitney U tests were used for the evaluation of the data (p = 0.05).

Results

The microhardness values of the Universal Restorative 200 composite resin group selected as the control group in the study were found to be higher than all other bulk-fill composite resin groups (p <0.05; p = 0.000) (Table 2; Table 3).

Hardness values of the bulk-fill composite resin groups prepared by applying the incremental technique were lower than the control group. When the hardness values of bulk-fill composite resin groups prepared by incremental technique were compared with the control group, it was observed that Tetric N-Ceram bulk-fill composite resin group had the lowest hardness value (p <0.05; p = 0.000) (Table 2).

Hardness values of the bulk-fill composite resin groups prepared by applying the bulk technique were lower than the control group. While the lowest values were observed in the Tetric N-Ceram bulk-fill composite resin group (p <0.05; p = 0.000), no difference was observed between the Tetric N-Ceram bulk fill and Tetric Evo Ceram bulk-fill composite resin groups (p > 0.05; p = 0.631) (Table 3).

When the techniques compared within the bulk-fill composite resins was found only in the Tetric Evo Ceram composite resin group differences (p <0.05; p = 0.001) (Table 4).

Table 2. Comparison of the incremental technique with the control group

	Incremental technique
Universal	88,63(84,87-92,50) a
3M Bulk	65,95(64,13-66,83) b
Tetric Evo Ceram Bulk	58,76(57,43-60,07) c
Tetric N-Ceram Bulk	57,05(54,70-58,17) d

Means followed by distinct lower letters represent statistically significant differences in each column (p < 0.05). There is no difference between the same letters.

Table 3. Comparison of the bulk technique with the control group

	Bulk technique
<i>Universal</i>	88,63(84,87-92,50) c
<i>3M Bulk</i>	66,36(64,77-69,23) a
<i>Tetric Evo Ceram Bulk</i>	56,66(54,70-58,13) b
<i>Tetric N-Ceram Bulk</i>	56,46(53,03-58) b

Means followed by distinct lower letters represent statistically significant differences in each column ($p < 0.05$). There is no difference between the same letters.

Table 4. Comparison of bulk and incremental techniques within bulk composites

	Bulk	Incremental	p
<i>3M Bulk</i>	66,36(64,77-69,23) a	65,95(64,13-66,83) a	P=0.165
<i>Tetric Evo Ceram Bulk</i>	56,66(54,70-58,13) a	58,76(57,43-60,07) b	P=0.001
<i>Tetric N-Ceram Bulk</i>	56,46(53,03-58) a	57,05(54,70-58,17) a	P=0.247

Means followed by distinct lower letters represent statistically significant differences in each row ($p < 0.05$). There is no difference between the same letters.

Discussion

Today, the trend to use bulk-fill composites is increasing due to their simplified procedures (11). Due to its good physical and mechanical properties, bulk-fill composite materials are used in large and deep cavities (12).

Surface hardness is one of the most substantial properties used to compare dental materials and is defined as resistance to deformation or fracture. Mechanical features of restorations should always be considered, especially when confronted with large areas of masticatory force (13). Surface microhardness is one of the basic requirements, especially in areas bearing posterior stress (11). The microhardness test method is an effective for determining the mechanical strength and rigidity of the material. One of the most frequently used devices for this purpose is the Vickers hardness device. This device has two different diagonal-shaped diamond tips that apply a pre-set force to the surface of the material, leaving a mark on the material surface. The diamond tips are shorter than the tips of other devices used in hardness measurement. Vickers hardness tester allows less affected by the surface properties of the material

during hardness measurement and allows better measurement (14). Therefore, hardness measurements were made with the Vickers hardness tester in the study. There is still no consensus on acceptable VHN values (15). It is believed that the ideal hardness values of composite resins can exceed 50 (VHN) (16). In this study, composite resins polymerization was completed with a LED light device, showed average hardness values above 50 VHN. In our study, differences were found in VHN values among composite resin materials. The VHN average values of the Universal Restorative 200 composite resin group selected as the control group were found to be higher than the bulk-fill composite resin groups. Tornavoi et al. (17) reported that the hardness values of composite resin (Z250-microhybrid) with silica and zirconia content were better among different composite resins. In the materials used in our study, we thought that the Filtek (3M-ESPE) group has higher VHN values compared to the other materials examined, having silica and zirconia content. Studies have reported that increased filler levels cause an increase in hardness values for resin composites (14). The high VHN values of the Universal Restorative 200 composite resin group may also be a role in the high filler ratio.

In the literature that many factors related to the content of the material influence on surface hardness of the resin composite restorative material (18). However, Tetric N-Ceram bulk-fill and Tetric Evo Ceram bulk-fill composite resin groups showed similar VHN values applied with the bulk technique. Similar contents of the same company may be effective in this study. However, in the incremental technique, Tetric Evo Ceram bulk-fill composite has higher VHN values than Tetric N-Ceram bulk-fill composite resin group. Tetric N-Ceram bulk fill groups containing TPO and Ivocerin with camphorquinone as photoinitiator were found to be significantly lower than composites in incremental and bulk technique. But in the bulk technique, there was no difference between it and Tetric Evo Ceram. Some studies have highlighted that Tetric N-Ceram and Tetric Evo-Ceram containing Ivocerin and TPO have lower surface hardness (19, 20). Studies in the literature have reported that the shape, proportion, and type of filler particles significantly affect the light transmittance and polymerization of the material (21). The lower filler ratio of Tetric N-Ceram bulk-fill composite compared to other composites may adversely affect the light transmittance with its larger filler particle size (22). In addition, due to the differences in the refractive index of the materials between the inorganic filler and the resin matrix, it has been reported that light scattering increases and decreases light transmittance in material with a large filler-matrix interface area (23). This may be effective in layering technique for Tetric N-Ceram. Manufacturers also noted that the main advance of bulk-fill composite materials, namely increased curing depth, possibly due to higher translucency and lower polymerization shrinkage stress, was related to changes in filler content and/or organic matrix (11).

In the current literature; can be affected the polymerization degree of bulk-fill composite resins such as composition of the material (photoinitiators, fillers, and organic matrix), (24) properties of the light-curing unit (light intensity, thermal emission, wavelength, distance, diameter of the light device tip) and photopolymerization (curing mode and exposure time), (25) temperature, (26) increasing thickness of the material (27). In addition, it has been reported that the size and distribution of filler particles have a significant influence on physical-mechanical properties, including surface hardness (11).

The incremental technique has long been accepted as the gold standard for inserting composite resin into the cavity to reduce shrinkage stress. (28). Sarret (29) and Campodonico et al.(30) noticed that reducing the layering and using the bulk technique may result in successful applications. Winkler et al. (31) reported that the layering technique does not have an advantage over the bulk technique when approximal stresses are taken into consideration. It is important for the longevity that the composite resin restorations are polymerized sufficiently (27) to give the polymerization reaction a sufficient hardness level (28). In this study, teflon molds that absorb much less light and allow the light emitted from the light device to pass deeper were used during the preparation of the samples, and the

microhardness values were measured after the specimens were kept in distilled water for 24 hours. A previous study (32) observed a tendency for microhardness to decrease as the thickness of the composite resin increases. Regarding the amount of hardness change, bulk-fill composite resins exhibited less stiffness variations with respect to thickness, while normal conventional composite resins showed a significant decrease in 4 mm samples. According to the results of the study, it was reported that bulk-fill composite resins up to 4 mm thick were at clinically acceptable levels. Incompatible with these findings, VHN values in bulk-fill composites, which are the current results of our study, are at clinically acceptable levels when placed at 4 mm thickness by both stratification and bulk technique. Restorative materials can be best evaluated with clinical studies (33, 34). Saliva, temperature changes, and pH levels in the oral environment can also affect the long-term microhardness values of composite resin materials. More studies are required to evaluate the mechanical and physical properties of conventional composite resins and bulk-fill composite resin materials.

Conclusions

Within the limitations of the current study, we can conclude that:

- It was observed that the difference in the content of composite resins affected different microhardness values.
- The highest VHN values were recorded in the Universal Restorative 200 composite resin (control) group compared to all bulk-fill composites.
- When composite resins applied with the bulk technique and incremental technique were evaluated, no difference was observed in other bulk-fill materials (Tetric N-Ceram Bulk Fill, Filtek One Bulk Fill) except Tetric Evo Ceram Bulk-Fill.

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References

1. Alrobeigy NA. Mechanical properties of contemporary resin composites determined by nanoindentation. *Tanta Dent J* 2017;14(3):129-138. ([Crossref](#))
2. Autio-Gold, JT, Barrett AA. Effect of fluoride varnishes on color stability of esthetic restorative materials. *Oper Dent*. 2004;29(6):636-641.
3. Rees JS, Jagger DC, Williams DR, Brown G, Duguid W. A reappraisal of the incremental packing technique for light cured composite resins. *J Oral Rehabil* 2004;31(1):81-84. ([Crossref](#))
4. Alrahlah A, Silikas N, Watts D. Post-cure depth of cure of bulk fill dental resin-composites. *Dental Mater* 2014;30(2):149-154. ([Crossref](#))
5. Donadio-Moura J, Gouw-Soares S, de Freitas PM, Navarro RS, Powell LG, Eduardo CDP. Tensile bond strength of a flowable composite resin to ER: YAG-laser-treated dentin. *Lasers Surg Med* 2005;36(5):351-5. ([Crossref](#))
6. Karaman E, Yazici AR, Ozgunaltay G, Dayangac B. Clinical evaluation of a nanohybrid and a flowable resin composite in non-carious cervical lesions: 24-month results. *J Adhes Dent* 2012;14(5):485-492.
7. El-Damanhoury H, Platt J. Polymerization shrinkage stress kinetics and related properties of bulk-fill resin composites. *Oper Dent* 2014;39(4):374-82. ([Crossref](#))
8. Cakir NN, Demirbuga S. Bulk fill composite resins in restorative dentistry. *J Health Sci Med / JHSM* 2019;28(3):199-193. ([Crossref](#))
9. Van Ende A, De Munck J, Lise DP, Van Meerbeek B. Bulk-fill composites: A review of the current literature. *J Adhes Dent* 2017;19(2):95-109.
10. Czasch P, Ilie N. In vitro comparison of mechanical properties and degree of cure of bulk fill composites. *Clin Oral Investig* 2013;17(1):227-35. ([Crossref](#))
11. Abed Y, Sabry H, Alrobeigy N. Degree of conversion and surface hardness of bulk-fill composite versus incremental-fill composite. *Tanta Dent J* 2015;12(2):71-80. ([Crossref](#))
12. Cabadağ ÖG, Gönülol N, Almasifar L, Misilli T. Effect of food simulating liquids on color stability of bulk-fill composites. *7tepe klinik* 2020;16(3):198-204. ([Crossref](#))
13. Bek Kürklü ZG, Kalkan Devenci C. Evaluation of microhardness and Bis-GMA release of a new composite restorative material. *Acta Odontol Turc* 2020;37(1):18-23. ([Crossref](#))
14. Karataş O, Iscan Yapar M, Ozakar Ilday N, Bayındır YZ. Comparison Depth of Cure of Bulk-Fill and Flowable Composite Resins. *Türkiye Klin J Dent Sci* 2015;21(3):216-220. ([Crossref](#))
15. Galvão MR, Caldas SG, Bagnato VS, de Souza Rastelli AN, de Andrade MF. Evaluation of degree of conversion and hardness of dental composites photo-activated with different light guide tips. *Eur J Dent* 2013;7(1):86-93.
16. Sharkey S, Ray N, Burke F, Ziada H, Hannigan A. Surface hardness of light-activated resin composites cured by two different visible-light sources: an in vitro study. *Quintessence Int* 2001;32(5):401-405.
17. Tornavoi TD, Sato S, Silva LJ, Agnelli JAM, Reis dos Cândido EA. Analysis of surface hardness of artificially aged resin composites. *Mater Res* 2012;15(1):9-14 ([Crossref](#))
18. Okada K, Tosaki S, Hirota K, Hume WR. Surface hardness change of restorative filling materials stored in saliva. *Dent Mater* 2001;17(1):34-39. ([Crossref](#))
19. Flury S, Hayoz S, Peutzfeldt A, Hüsler J, Lussi A. Depth of cure of resin composites: is the ISO 4049 method suitable for bulk fill materials?. *Dent Mater* 2012;28(5):521-528. ([Crossref](#))
20. Jang JH, Park SH, Hwang IN. Polymerization shrinkage and depth of cure of bulk-fill resin composites and highly filled flowable resin. *Oper Dent* 2015;40(2):172-180. ([Crossref](#))
21. Arikawa H, Kanie T, Fujii K, Takahashi H, Ban S. Effect of inhomogeneity of light from light curing units on the surface hardness of composite resin. *Dent Mater J* 2008;27(1):21-28. ([Crossref](#))
22. Ozyurt E, Kurt A, Yıldırım H. Evaluation of the polymerization depth of bulk fill resin composites polymerized by different procedures: An in-vitro study. *Clin Exp Heal Sci* 2019;9(4):304-309. ([Crossref](#))
23. Tarle Z, Attin T, Marovic D, Andermatt L, Ristic M, Tauböck TT. Influence of irradiation time on subsurface degree of conversion and microhardness of high-viscosity bulk-fill resin composites. *Clin Oral Investig* 2015;19(4):831-840. ([Crossref](#))
24. Amirouche-Korichi A, Mouzali M, Watts DC. Effects of monomer ratios and highly radiopaque fillers on degree of conversion and shrinkage-strain of dental resin composites. *Dent Mater* 2009;25(11):1411-1418. ([Crossref](#))
25. Torno V, Soares P, Martin JM, Mazur RF, Souza EM, Vieira S. Effects of irradiance, wavelength, and thermal emission of different light curing units on the Knoop and Vickers hardness of a composite resin. *J Biomed Mater Res B Appl Biomater* 2008;85(1):166-171. ([Crossref](#))
26. Par M, Gamulin O, Marovic D, Klaric E, Tarle Z. Effect of temperature on post-cure polymerization of bulk-fill composites. *J Dent* 2014;42(10):1255-1260. ([Crossref](#))
27. Flury S, Peutzfeldt A, Lussi A. Influence of increment thickness on microhardness and dentin bond strength of bulk fill resin composites. *Dent Mater* 2014;30(10):1104-1112. ([Crossref](#))
28. Temirek MMA. Effect of packing technique and curing time on the marginal sealing and surface hardness of two bulk-fill resin composites. *Egypt Dent J* 2019;65(3):2587-2596. ([Crossref](#))
29. Sarrett DC. Clinical challenges and the relevance of materials testing for posterior composite restorations. *Dent Mater* 2005;21(1):9-20. ([Crossref](#))
30. Campodonico CE, Tantbirojn D, Olin PS, Versluis A. Cuspal deflection and depth of cure in resin-based composite restorations filled by using bulk, incremental and transtooth-illumination techniques. *J Am Dent Assoc* 2011;142(10):1176-1182. ([Crossref](#))
31. Winkler MM, Katona TR, Paydar NH. Finite element stress analysis of three filling techniques for class V light-cured composite restorations. *J Dent Res* 1996;75(7):1477-1483. ([Crossref](#))
32. Kim EH, Jung KH, Son SA, Hur B, Kwon YH, Park JK. Effect of resin thickness on the microhardness and optical properties of bulk-fill resin composites. *Restor Dent Endod* 2015;40(2):128-135. ([Crossref](#))
33. Nikaido T, Kunzelmann KH, Chen H, et al. Evaluation of thermal cycling and mechanical loading on bond strength of a self-etching primer system to dentin. *Dent Mater* 2002;18(3):269-275. ([Crossref](#))
34. Koyuturk AE, Kusgoz A, Ulker M, Yeşilyurt C. Effects of mechanical and thermal aging on microleakage of different fissure sealants. *Dent Mater J* 2008;27(6):795-801. ([Crossref](#))