

*Original Article / Araştırma Makalesi*


**COMPARISON OF THE MICROHARDNESS OF BULK FILL COMPOSITES**

**Bulk Fill Kompozitlerin Mikrosertliklerinin Karşılaştırılması**

Reyhan ŞİŞMAN<sup>1</sup> 

Burak DAYI<sup>2</sup> 

Hacer TURGUT<sup>3</sup> 

Enis ŞİMŞEK<sup>4</sup> 

<sup>1</sup>Private Dentist, Gümüşhane

<sup>2,3</sup>Inonu University, Faculty of Dentistry, Malatya

<sup>4</sup>Inonu University, Vocational School of Health Services, Malatya

*Geliş Tarihi / Received:* 31.03.2022

*Kabul Tarihi / Accepted:* 20.04.2022

**ABSTRACT**

The aim of this study is to compare the microhardness of five different bulk fill composites (SDR, TEC, XTF, SF, FBF). A total of 25 cylindrical molds were prepared. Composite resin was placed in each mold in sequence, at one time, by condensing it thoroughly. Then, the polymerization of the composites was achieved with the LED light device which was applied for 20 seconds. Microhardness measurements of the samples were made with the classical Vicker's test. The data of our study were evaluated by using the Kruskal Wallis H test with the SPSS 20.0 package program. When the measured surface microhardness values were compared between the groups; microhardness values SDR and FBF groups were found to be significantly lower than TEC, XTF and KSF groups ( $p<0.05$ ). When the sub-surface microhardness values were compared between the groups; the microhardness value of the SDR group was found to be significantly higher than the FBF group, and significantly lower than the TEC, XTF and KSF groups ( $p<0.05$ ). When both the lower and upper surface microhardness values were compared between the groups, XTF was found to be the bulk fill composite group with the best microhardness.

**Keywords:** Bulk fill resin composite, Composite resins, Microhardness.

**ÖZ**

Bu çalışmanın amacı beş farklı bulk fill kompozitin mikrosertliklerinin karşılaştırılmasıdır (SDR, TEC, XTF, SF, FBF). Toplam 25 adet silindirik kalıp hazırlandı. Sırası ile her bir kalıba tek seferde kompozit rezin iyice kondanse edilerek yerleştirildi. Daha sonra 20 sn uygulanan LED ışık cihazı ile kompozitlerin polimerizasyonu sağlandı. Örneklerin mikrosertlik ölçümleri klasik Vicker's testi ile yapıldı. Çalışmamızın verileri SPSS 20.0 paket programı ile Kruskal Wallis H testi kullanılarak değerlendirildi. Ölçülen üst yüzey mikrosertlik değerleri gruplar arasında karşılaştırıldığında; SDR ve FBF grubunun mikrosertlik değeri, TEC, XTF ve KSF gruplarına göre anlamlı derecede daha düşük bulundu ( $p<0.05$ ). Gruplar arasında alt yüzey mikrosertlik değerleri karşılaştırıldığında; SDR grubunun mikrosertlik değeri FBF grubuna göre anlamlı derecede daha yüksek, TEC, XTF ve KSF gruplarına göre ise anlamlı derecede daha düşük bulundu ( $p<0.05$ ). Hem alt hem de üst yüzey mikrosertlik değerleri gruplar arasında karşılaştırıldığında, XTF en iyi mikrosertliğe sahip bulk fill kompozit grubu olarak bulundu.

**Anahtar kelimeler:** Bulk fill kompozit rezin, Kompozit rezinler, Mikrosertlik.

---

## INTRODUCTION

In these days, composite resins have extensive use in many fields due to their developing physical, mechanical and aesthetical specifications. The application of composite resins differs based on the differences in their composition and the implementers (Ferracane, 1985). Thus, the traditional composite resins are placed with a maximum of 2 mm thickness in cavities and each layer is polymerized by light for at least 20-40 seconds (Ferracane, 1985; Rueggeberg & Margeson, 1990). In addition to increased patient treatment duration, this situation holds the risk of air entrainment between the composite resin layers or contamination with humidity (Cohen, Leonard, Charlton, Roberts & Ragain, 2004).

It is known that bulk fill composite resins that were brought into use recently and enables polymerization of 4 mm thick composite resins at once, decrease the polymerization shrinkage (Park, Chang, Ferracane & Lee, 2008) and can contain systems that can induce new polymerizations (Wieczkowski, Joynt, Klockowski & Davis, 1988). In addition, another bulk fill composite resin was produced, which is placed in the cavity by using a sonic dental drill, unlike traditional and other bulk fill composite resins (SonicFill, Kerr, Orange, CA, USA). Sonic-Fill (SF) composite resins can be placed up to 5 mm thickness in one step (Yap, 2000). Sonic-Fill composites are a combination of both universal and flowable composites and are activated by sonic vibration, transforming from high viscosity to low viscosity composites, providing ease of application. However, there is not a sufficient database about these new resin systems.

Surface hardness is one of the most significant mechanical characteristics of composite resins (Ferracane, 1985) and it can be affected from small changes in polymer crosslinks in high transformation fields (Rueggeberg & Margeson, 1990). There is a correlation between the hardness value and the degree of transformation as shown in the literature (Cohen et al., 2004). It is known that for the polymerization of traditional composite resins to be completed fully, they have to be placed in layers of maximum 2 mms in the cavity (Park et al., 2008). Incompletion of the polymerization fully results in low hardness values, residual monomer excess, difficulties in bonding, leakages, and fractures (Wieczkowski et al., 1988; Yap, 2000). Certain studies conducted with bulk fill composite resins that can be placed in the cavity in 4mm and bigger thicknesses did not have any reservations for their clinical use (Flury, Hayoz, Peutzfeldt, Husler & Lussi, 2012; Moorthy et al., 2012; Roggendorf, Kramer, Appelt, Naumann & Frankenberger, 2011), while others argued that polymerization of composite resins in 4 mm depth reduces the mechanical characteristics of composite resins (Ilie &

Hickel, 2011). In a study by Ilie et al., it was determined that hardness and elastic modules of bulk fill composites were lower than traditional composites (Ilie, Bucuta & Draenert, 2013). In another study by El-Safty et al., the nano-hardness of bulk fill composites and flowable composites was found to be lower than traditional composites (El-Safty, Akhtar, Silikas & Watts, 2012).

The objective of the study is to investigate the surface hardness of five different bulk fill composite resins that are in use. Null hypothesis is that there is no difference between the surface hardness of the composites used in the study.

## MATERIAL AND METHOD

Five different bulk composite resins; SDR (Smart Dentin Replacement), Dentsply, Caulk, Milford DE, USA), Tetric EvoCeram Bulk Fill (TEC, Ivoclar, Vivadent, Schaan, Liechtenstein), X-trafil (XTF, Voco GmbH, Cuxhaven, Germany), Sonic Fill (SF, Kerr, Orange, CA, USA), Filtek Bulk Fill (FBF, 3M Espe, USA) were used in the study (Table 1).

**Table 1.** The Bulk Fill Composite Resins Used Contents, Filler Rates, Types, Manufacturers in the Study.

Bulk Fill Composite Resine	Composition	Filler Ratio (Weight, Volume)	Type	Manufacturer
SDR	SDR™ patented modified UDMA, TEGDMA.BisGMA Barium and stronsiumalumino-floro-silikat glass	68% 45%	Flowable	Dentsply, Caulk, Milford, DE, USA
TEC	Bis-GMA, TEGDMA (%17-18) barium glass, ytterbiyumtriflorid	79-81% 60-61%	Nanohybrid	Ivoclar, Vivadent, Schaan, Liechtenstein
XTF	Bis-GMA, UDMA, TEGDMA	86% 50.1%	Hybrid	Voco, GmbH, Cuxhaven, GERMANY
SF	Bis-GMA, TEGDMA, EBPADMA glass, glass dioxide	83.5% 66%	Nanohybrid	Kerr, Orange, CA, USA
FBF	Bis-GMA,UDMA, Bis-EMA ytterbiyum triflorid,zircon silica	64.5% 42.5%	Flowable	3M Espe, St.Paul, USA

SDR(Smart Dentin Replacement), TEC (Tetric EvoCeram), XTF (X-trafil), SF ( Sonic Fill), FBF (Filtek Bulk Fill)

### Preparation of the samples

25 cylindrical molds (4mm x 5mm), 5 for each group, were prepared (n=5). The upper surfaces of the molds were marked. Composite resin was placed and condensed in each mold respectively in one step. Upper and bottom surfaces were pressed using strip band and glass respectively to obtain the desired level of condensation. Later on, the glass on top was taken away and they were polymerized over the strip band for 20 seconds using LED light equipment (Elipar S10, 3M ESPE, St. Paul, MN, USA). When the half-lives of composite

monomers are considered, polymerization can continue for a few days after the polymerization process (Bouschlicher, Rueggeberg, and Wilson, 2004). Thus, the samples were kept in incubator in distilled water for 24 hours and then the hardness measurement tests were conducted.

### Surface Hardness Measurements

The surface hardness measurements of the samples were conducted by Vicker's method in Erciyes University Faculty of Engineering Research Laboratory using Streuers Duramin-5 micro hardness equipment (Streuers Corp. Japan) and Duramin 5 Measurements software Version 3.2.6.1 (Product 3.2.6.0). Vicker's hardness value was obtained by measuring the trace diagonals created by the application of 300 gr of weight on the sample for 10 seconds using the computer software. Average hardness values were obtained by taking six measures from top and bottom surfaces of each sample and calculating the averages of these measurements. The data of the study was analyzed using Kruskal Wallis H and Mann Whitney-U tests with the SPSS 20.0 software package program.

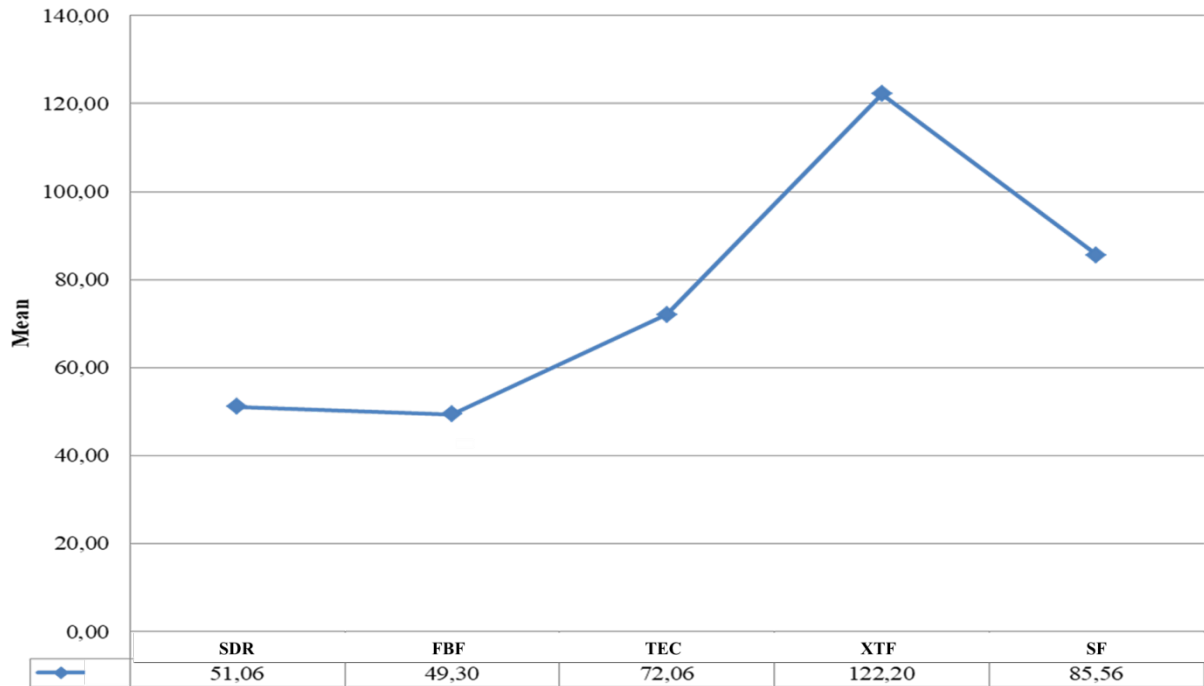
## RESULTS

Top and bottom surfaces micro hardness values for bulk fill composite resins used in the study are displayed in Table 2 (Figure 1, Figure 2).

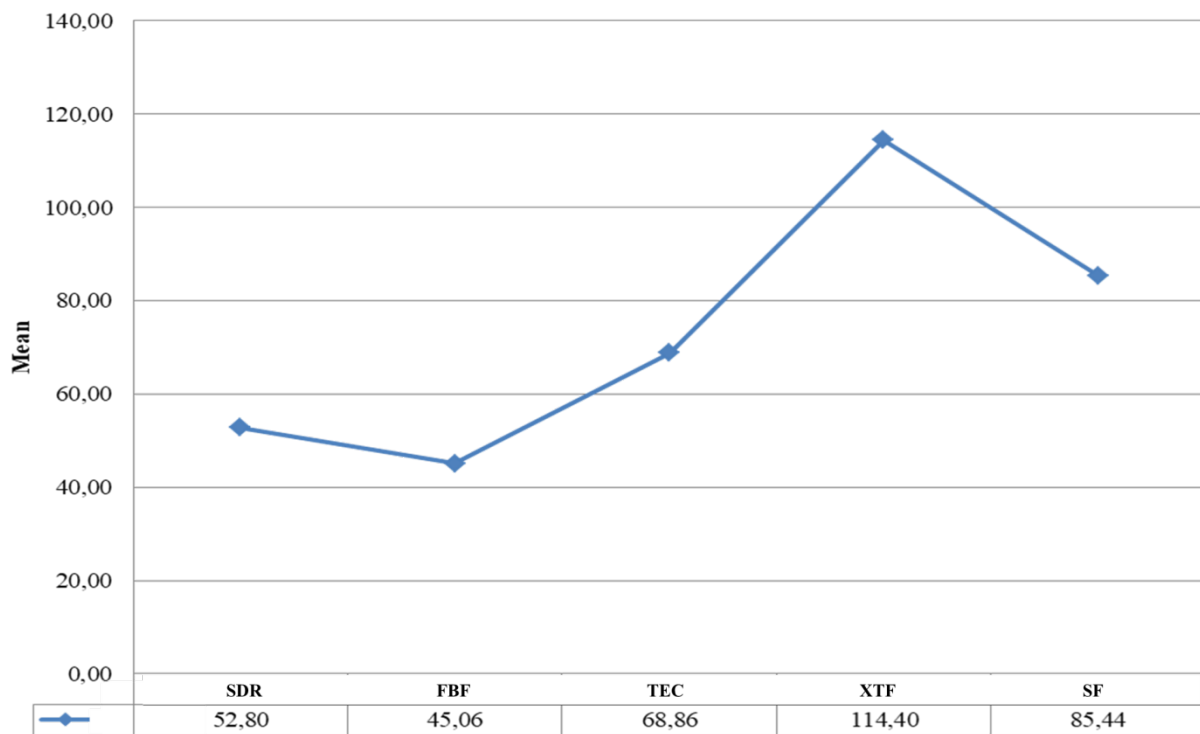
**Table 2.** Average of Upper/Lower Microhardness Values

Bulk-fill Composite Resine		Mean ± SD	
SDR	upper	51.06±3.46	p>0.05
	lower	52.80±4.44	
FBF	upper	49.30±1.34	p<0.05
	lower	45.06±2.67	
TEC	upper	72.06±5.22	p>0.05
	lower	68.86±5.84	
XTF	upper	122.20±4.76	p>0.05
	lower	114.40±7.60	
SF	upper	85.56±12.2	p>0.05
	lower	85.44±7.75	

SDR (Smart Dentin Replacement), FBF (Filtek Bulk Fill), TEC (Tetric EvoCeram), XTF (X-trafil), SF (Sonic Fill)



**Figure1.** Average of Upper Microhardness Values



**Figure2.** Average of Lower Microhardness Values

Thus, when the measured top surface micro hardness values were compared between the groups, micro hardness values for SDR and FBF groups were found to be significantly lower when compared to TEC, XTF and KSF groups ( $p < 0.05$ ). Micro hardness value for XTF group was found significantly higher when compared to TEC and SF groups ( $p < 0.05$ ). When the top and bottom surface micro hardness values were compared within groups, only the

bottom surface micro hardness value for FBF group was found to be statistically significantly lower when compared to the top surface ( $p < 0.05$ ). In other groups, the micro hardness values for bottom surfaces were found to be lower than top surfaces, however there was no statistically significant difference between these values ( $p > 0.05$ ).

## DISCUSSION

Micro hardness of five bulk fill resin composites was tested in this study. It was observed that FBF and SDR had the lowest micro hardness values among the materials tested. The manufacturers of SDR and FBF bulk fill composite resins that were used in this study suggest that after placement of these materials in the cavity in a thickness of 4mm and polymerization, it should be covered with 2mm thick traditional composite material. The fact that hardness values for SDR and FBF were found to be lower than other bulk fill composite resins in this study is in support of this information. According to these results, the null hypothesis was rejected.

It is determined that inorganic filling content used in composite resins is among the factors that affect mechanical and physical features of composite resins (Cabadağ, Misilli & Gönüloğlu, 2021; Kusgoz et al., 2011). Studies showed that there was a direct proportion between the filling content and hardness values of composite resins, and different composite resins having different surface hardness values was due to different matrixes and different fillings (Cekic-Nagas, Egilmez & Ergun, 2010; Scougall-Vilchis, Hotta, Hotta, Idono & Yamamoto, 2009). Filling rates of the bulk fill composite resins used in this study by weight from the highest to the lowest were as follows: XTF, SF, TEC, SDR and FBF. Micro hardness values of the bulk fill composite resins used in this study from the highest to the lowest were as follows: XTF, SF, TEC, SDR and FBF. These findings showed that there was a direct proportion between the micro hardness values of bulk fill composite resins and filling rates in the study.

One of the concerns about placing composites in excessive amounts is the fear of the light not reaching the lower surfaces of the composites and polymerization not being effective in areas far from the light device. One of the methods used for evaluating the degree of polymerization is the surface hardness measurement (Frauscher & Ilie, 2012). Determination of the degree of polymerization by surface hardness measurement is based on the ratio of bottom surface hardness value of the composite resin to the top surface hardness value (Bouschlicher et al., 2004). Theoretically, for polymerization to be accepted as successful,

bottom surface hardness of the composite resin should be at least 80% of the top surface hardness (Alkan, Arısu & Dalkılıç, 2020; Bouschlicher et al., 2004; Ilie & Stark, 2014).

Thus, in addition to top surface micro hardness of the bulk fill composite resins tested in the study, bottom surface micro hardness was measured as well. The findings of the study demonstrated that bottom surface micro hardness values were lower than top surface micro hardness values in all groups. However, based on the data from previous studies, the rates of difference of hardness values between the bottom and top surfaces were in acceptable amounts (bottom surface / top surface  $\geq$  80%) (El-Damanhoury & Platt, 2014; Ilie & Stark, 2014; Jang, Park & Hwang, 2014). This finding also means that, in accordance with the findings of other studies (El-Damanhoury & Platt, 2014; Flury et al., 2012), bulk fill composite resins could reach sufficient polymerization thickness by polymerization in 4mm thicknesses using light. In FBF bulk fill composite group bottom surface hardness values were found significantly lower than top surface hardness levels in the study. This finding showed that polymerization occurred less than other groups in FBF.

## CONCLUSIONS

The study demonstrated one-to-one relationship between the filling content of bulk fill composites and their micro hardness values. Furthermore, polymerization depths for all groups were found to have acceptable limits in 4 mm thickness.

## REFERENCES

- Alkan, F., Arısu, H. D. & Dalkılıç, E. E. (2020). Bulk-fill kompozitlerde kalınlığın artışının mikrosertlik üzerine etkisi. *Selcuk Dental Journal*, 7(2), 141-147.
- Bouschlicher, M. R., Rueggeberg, F. A. & Wilson, B. M. (2004). Correlation of bottom-to-top surface microhardness and conversion ratios for a variety of resin composite compositions. *Oper Dent*, 29(6), 698-704.
- Cabadağ, Ö. G., Misilli, T. & Gönüloğlu, N. (2021). Bulk-fill kompozit rezinlere güncel bakış. *Selcuk Dental Journal*, 8(1), 220-228.
- Cekic-Nagas, I., Egilmez, F. & Ergun, G. (2010). The effect of irradiation distance on microhardness of resin composites cured with different light curing units. *Eur J Dent*, 4(4), 440-446.
- Cohen, M. E., Leonard, D. L., Charlton, D. G., Roberts, H. W. & Ragain, J. C. (2004). Statistical estimation of resin composite polymerization sufficiency using microhardness. *Dent Mater*, 20(2), 158-166.
- El-Damanhoury, H. & Platt, J. (2014). Polymerization shrinkage stress kinetics and related properties of bulk-fill resin composites. *Oper Dent*, 39(4), 374-382.
- El-Safty, S., Akhtar, R., Silikas, N. & Watts, D. C. (2012). Nanomechanical properties of dental resin-composites. *Dent Mater*, 28(12), 1292-1300.

- 
- Ferracane, J. L. (1985). Correlation between hardness and degree of conversion during the setting reaction of unfilled dental restorative resins. *Dent Mater*, 1(1), 11-14.
- Flury, S., Hayoz, S., Peutzfeldt, A., Husler, J. & Lussi, A. (2012). Depth of cure of resin composites: is the ISO 4049 method suitable for bulk fill materials? *Dent Mater*, 28(5), 521-528.
- Frauscher, K. E. & Ilie, N. (2012). Depth of cure and mechanical properties of nano-hybrid resin-based composites with novel and conventional matrix formulation. *Clin Oral Investig*, 16(5), 1425-1434.
- Ilie, N., Bucuta, S. & Draenert, M. (2013). Bulk-fill resin-based composites: an in vitro assessment of their mechanical performance. *Oper Dent*, 38(6), 618-625.
- Ilie, N. & Hickel, R. (2011). Investigations on a methacrylate-based flowable composite based on the SDR technology. *Dent Mater*, 27(4), 348-355.
- Ilie, N. & Stark, K. (2014). Curing behaviour of high-viscosity bulk-fill composites. *J Dent*, 42(8), 977-985.
- Jang, J. H., Park, S. H. & Hwang, I. N. (2014). Polymerization shrinkage and depth of cure of bulk-fill resin composites and highly filled flowable resin. *Oper Dent*.
- Kusgoz, A., Ulker, M., Yesilyurt, C., Yoldas, O. H., Ozil, M. & Tanriver, M. (2011). Silorane-based composite: depth of cure, surface hardness, degree of conversion, and cervical microleakage in Class II cavities. *J Esthet Restor Dent*, 23(5), 324-335.
- Moorthy, A., Hogg, C. H., Dowling, A. H., Grufferty, B. F., Benetti, A. R. & Fleming, G. J. (2012). Cuspal deflection and microleakage in premolar teeth restored with bulk-fill flowable resin-based composite base materials. *J Dent*, 40(6), 500-505.
- Park, J., Chang, J., Ferracane, J. & Lee, I. B. (2008). How should composite be layered to reduce shrinkage stress: incremental or bulk filling? *Dent Mater*, 24(11), 1501-1505.
- Roggendorf, M. J., Kramer, N., Appelt, A., Naumann, M. & Frankenberger, R. (2011). Marginal quality of flowable 4-mm base vs. conventionally layered resin composite. *J Dent*, 39(10), 643-647.
- Rueggeberg, F. A. & Margeson, D. H. (1990). The effect of oxygen inhibition on an unfilled/filled composite system. *J Dent Res*, 69(10), 1652-1658.
- Scougall-Vilchis, R. J., Hotta, Y., Hotta, M., Idono, T. & Yamamoto, K. (2009). Examination of composite resins with electron microscopy, microhardness tester and energy dispersive X-ray microanalyzer. *Dent Mater J*, 28(1), 102-112.
- Wieczkowski, G., Jr., Joynt, R. B., Klockowski, R. & Davis, E. L. (1988). Effects of incremental versus bulk fill technique on resistance to cuspal fracture of teeth restored with posterior composites. *J Prosthet Dent*, 60(3), 283-287.
- Yap, A. U. (2000). Effectiveness of polymerization in composite restoratives claiming bulk placement: impact of cavity depth and exposure time. *Oper Dent*, 25(2), 113-120.