

BOND STRENGTH OF THE BULK FILL COMPOSITE MATERIALS

ЈАЧИНА НА ВРСКАТА КАЈ BULK FILL КОМПОЗИТНИ МАТЕРИЈАЛИ

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Abstract

INTRODUCTION. The Bulk Fill composites possess specific features, including increased flow rates in order to achieve an appropriate cavity adaptation. An improved depth of at least 4 mm eliminates the need for layered application techniques, thereby reducing working hours. **AIM.** The aim of this study is to compare the shear bond strength (SBS) of the recently introduced three different brands of Bulk Fill composites, with one conventional micro hybrid and one nano hybrid composite. **MATERIALS AND METHODS.** In order to realize the set goals in our in vitro study, we included 35 extracted, non-carious molars in male and female patients of different ages as research samples. Three commercial Bulk Fill composites (TetricEvoCeram Bulk Fill, TetricEvoCeram Bulk Flow, SureFil® SDR® Flow) have been tested and used to control two conventional composites with a layered application technique of 2 mm (Filtek Z250, Gradia posterior). Randomly selected teeth were divided into 5 groups, with 7 teeth each. **RESULTS.** The average values and standard deviations in all groups are presented in Table 2 and Figure 1. The highest average SBS ($6.24 \pm 1.65\text{MP}$) has been noted for the Filtek nano hybrid composite (Group IV) bound to a dentine sample, while the lowest average SBS ($4.52 \pm 0.65\text{MP}$) has been registered for full filling SDR composite (Group I). **CONCLUSION.** The results indicate that the application of bulk fill composite material results in an acceptable SBS comparable to that achieved through conventional RBC. As such, bulk fill composites can provide reliable alternatives to conventional composites. **KEY WORDS:** composit, tehniqe, strength.

Апстракт

ВОВЕД. Bulk Fill композити поседуваат специфични карактеристики, вклучувајќи зголемена проточност за да се постигне соодветна адаптација на кавитетит. Подобрната длабочина од најмалку 4 мм ја елиминира потребата за слоевита техника на нанесување, а со тоа се намалува работното време. **ЦЕЛ.** Целта на оваа студија е да се спореди јачината на силата на смолкнување (shear bond strength - SBS) на неодамна воведените три различни брендови на Bulk fill композити, со еден конвенционален микро хибриден и еден нано хибриден композит. **МАТЕРИЈАЛ И МЕТОД.** За да ги реализираме зададените цели во нашата ин витро студија како истражувачки примерок вклучивме 35 екстрахирани, некариозни, молари кај пациенти од машки и женски род со различна возраст. Беа тестирани три комерцијални Bulk fill (TetricEvoCeram Bulk Fill, TetricEvoCeram Bulk Flow, SureFil® SDR® Flow) композити и беа користени како контрола два конвенционални композити со слоевита техника на нанесување од 2 mm (Filtek Z250, Gradia posterior). Забите по случаен избор беа поделени на 5 групи, со по 7 заби. **РЕЗУЛТАТИ.** Највисоката средна SBS ($6,24 \pm 1,65\text{MPa}$) е забележана за Filtek нанохибриден композит (група IV) врзан за примерок од дентин, додека најнискиот просечен SBS ($4,52 \pm 0,65\text{MPa}$) е регистриран за SDR композитот со целосно пополнување (група I). **ЗАКЛУЧОК.** Резултатите покажуваат дека примената на bulk fill композитниот материјал резултира со прифатлив SBS кој е споредлив со оној постигнат преку конвенционалните RBC. Како такви, bulk fill композитите може да претставуваат сигурни алтернативи на конвенционалните композити. **КЛУЧНИ ЗБОРОВИ:** композити, техника, јачина.

Introduction

The quest for restorative material having the optimum properties of fusion with solid dental substances leads to numerous dental materials, adhesive systems and restorative techniques. The main disadvantage of resin-based materials is the contraction while bonding. The polymerization contraction of the composite breaks the bond between the dentine adhesive and the tooth tissue, especially in the insufficiently well-conditioned surfaces, and this leads to oblique micro cracks. The prevention of these micro cracks is in the creation of a stronger bond between the dentine adhesive and the composite with the tooth tis-

sues. The strength of the polymerization contraction depends on:

1. The formula of the resin matrix and the amount of the filler into the composite;
2. The appearance of the cavity;
3. The type of the base;
4. The size, shape and location of the composite layer;
5. Light or chemical polymerization;
6. Module of elasticity (factor C);
7. The intensity of the connecting light, and
8. Degree of inbuilt porosity^{1, 2, 3, 4, 5}.

The Bulk Fill composites possess specific features, including increased flow rates in order to achieve an appropriate cavity adaptation. Elasticity and low polymerization lead to stress reduction and micro-flow, and thus to a reduction in postoperative sensitivity and the emergence of secondary caries. An improved depth of at least 4 mm eliminates the need for layered application techniques, thereby reducing working hours⁶. Today, traditional techniques of placing composite resins include this technique⁷. Most physicians recommend layered placement of composites with a layer thickness of 2 mm. However, every dentist will prefer composite material that can be used with Bulk Fill technique especially for posterior restorations. Therefore, the purpose of this study is to compare the shear bond strength (SBS) of the recently introduced three different brands of Bulk Fill composites, with one conventional micro hybrid and one nano hybrid composite.

Material and methods

In order to realize the set goals in our in vitro study, we included 35 extracted, non-carious molars in male and female patients of different ages as research samples, which after extraction have been stored at 4°C in a 0.5% solution of sodium hypo chloride for 24 hours before being washed with a saline solution and stored in distilled water at room temperature during the test period.

The teeth used for this study were extracted for a period not longer than 6 months. Teeth extracted more than 6 months before use may be subject to degenerative changes in dentinal protein.

We used the occlusion surface of the extracted molars. Each tooth was placed in a 25 mm internal diameter and 25 mm height mold, made of PVC (poly - vinyl - chloride). The mold was filled with mixed self-binding acrylic - ORTOpoli-PoliDent, Slovenia. When the molds were filled, they were placed in a cold water bath during

the polymerization of the acrylic because the heat from the polymerization could adversely affect the samples. Immediately after hardening of the acrylic we opened the hoods and removed the prepared samples from the mold.

The coating enamel was cut by IsoMet 1000 (precision cutting machine with diamond discs of Buehler Ltd, Lake Bluff, IL, USA) with water cooling, ensuring to reach only the surface dentine.

We used surface dentine as close as possible to the enamel, in order to reduce the test variations. We created a standard, repetitive, flat surface. We kept the surfaces of the teeth wet at any time, because dentine is particularly sensitive to dehydration. Exposing the surface of the teeth in the air for several minutes can cause irreversible changes in the adhesive bond.

Three commercial Bulk Fill composites were tested and used to control two conventional composites with a layered application technique of 2 mm. The materials used in the study are presented in Table 1.

Randomly selected teeth were divided into 5 groups, with 7 teeth each.

- **Group I:** After the adhesive application of each sample with G Bond-GS, using a technique of self-etching, we set a pier with SDR Flow (Dentsply. Konstanz. Germany) composite resin with a height of 3.0 mm and a diameter of 3.0 mm. Each composite excess was carefully removed and then polymerized for 20 seconds using a diode for transmitting polymerizing light (LED) with intensity of 1000 mW/cm² (Elipar S10, 3M ESPE, Seefeld, Germany).
- **Group II:** After the adhesive application of each sample with G Bond-GS, a pier with a composite resin TetricEvoCeram Bulk Flow (Ivoclar. Vivadent. AG. Liechtenstein) with a technique of self-etching was placed with a height of 3.0 mm and a diameter of 3.0 mm. Each composite excess was carefully removed and then polymerized for 20 seconds using a light emitting polymerizing light (LED) with a power of

Table 1. Materials used in the study.

Type of material	Name	Manufacturer	Volume of filler (%) (wt)
Nanohybride composite	Filtek Z250	3 M ESPE	73,5%
Nanohybride composite	TetricEvoCeram Bulk Fill	Ivoclar. Vivadent. AG. Liechtenstein	79% - 81%
Flow composite	TetricEvoCeram Bulk Flow	Ivoclar. Vivadent. AG. Liechtenstein	
Flow composite	SureFil® SDR® Flow Dentsply	Konstanz. Germany	68%
Microhybrid composite	Gradia posterior	GC Corp. Tokyo. Japan	81%

1000 mW/cm² (Elipar S10, 3M ESPE, Seefeld, Germany).

- **Group III:** After the adhesive application of each sample with G Bond-GS, a pier with composite resin TetricEvoCeram Bulk Fill (Ivoclar. Vivadent. AG Liechtenstein) with a height of 3.0 mm and a diameter of 3.0 mm was set with a self-etching technique. Each composite excess was carefully removed and then polymerized for 20 seconds using a light emitting polymerizing light (LED) with a power of 1000 mW/cm² (Elipar S10, 3M ESPE, Seefeld, Germany).
- **Group IV:** After the application of the adhesive asset, G Bond-GS, a column of conventional nano hybrid composite resin Filtek Z250 (3M ESPE) with a height of 3.0 mm and a 3.0 mm diameter was placed. Each layer was two millimeters thick and polymerized with LED polymerizing light with intensity of 1000 mW/cm² for 20 seconds (Elipar S10, 3M ESPE, Germany).
- **Group V:** Following the application of the adhesive asset, G Bond-GS, a column of conventional mono-hybrid composite resin Gradia posterior GC (Corp. Tokyo. Japan) was installed with a height of 3.0 mm and a 3.0 mm diameter. Each layer was two millimeters thick and polymerized with LED polymerizing light with a intensity of 1000 mW/cm² for 20 seconds (Elipar S10, 3M ESPE, Germany).

Once prepared, the samples were stored in a 37 °C incubator with a 100% humidity over 24 hours before testing the shear bond strength (SBS) of the resin bond using a universal test machine (INSTRON 4301) at a speed of 0.5 mm/min. SBS of the dentin composite resin was recorded in Newton and calculated in MP, and the cross-sectional area of the composite composition was taken into account.

Statistical analysis was carried out with SPSS Statistics v. 22 software (IBM, NY, USA). One-way ANOVA was used for group comparison of the relation strength. Shapiro-Wilk and Levene tests were carried out in order to assess the assumptions on the data normality and the difference homogeneity. Post hoc testing for multiple comparisons among groups was carried out by the Tukey's HSD test. P-values were adjusted by the Bonferroni correction method for multiple comparisons. The confidence level was set at 95% (p<0.05).

Results

The average values and standard deviations in all groups are presented in Table 2 and Figure 1. The highest average SBS (6.24 ± 1.65MP) was noted for the Filtek nano hybrid composite (Group IV) bound to a

dentine sample, while the lowest average SBS (4.52 ± 0.65 MP) was registered for full filling SDR composite (Group I). From Table 3 we can see that this difference between Group IV and I is statistically significant (p=0.031). Although Group III showed higher bonding strength than group I, group II, and group V, these differences were not considered statistically significant (p values between group III and I, group III and II, group III and V, were found as p=0.196, p=0.272 and p=1.000

Table 2. Average Values and Standard Deviations respectively

Descriptive Statistics

Dependent Variable: SBS

Composite	Mean	Std. Deviation	N
SDR	4.5223	0.65986	7
Tetric EvoCeram flow	4.8716	1.03139	7
Tetric EvoCeram bulk fill	5.8462	0.72230	7
Filtek	6.2481	1.65103	7
Gradia	5.0344	0.54211	7
Total	5.3045	1.14579	35

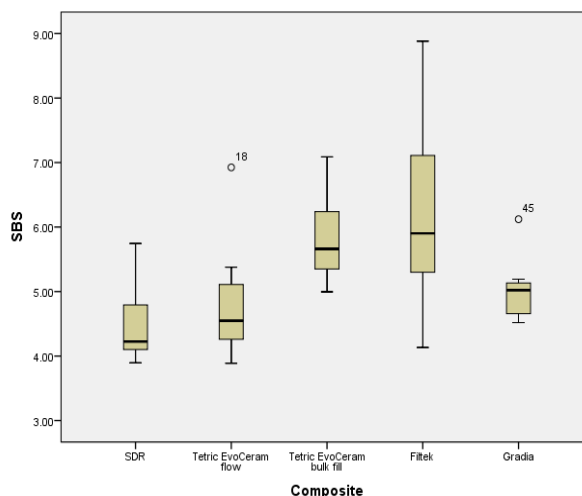


Figure 1. Average Values and Standard Deviations

Table 3. Pairwise Comparisons

Dependent Variable: SBS

(I) Composite	(J) Composite	Mean Difference (I-J)	Std. Error	Sig. ^b	95% Confidence Interval for Difference ^b	
					Lower Bound	Upper Bound
SDR	Tetric EvoCeram flow	- 0.349	0.537	1.000	-1.975	1.277
	Tetric EvoCeram bulk fill	-1.324	0.537	0.196	-2.950	0.302
	Filtek	-1.726*	0.537	0.031	-3.352	-0.100
	Gradia	-0.512	0.537	1.000	-2.138	1.114
Tetric EvoCeram flow	SDR	0.349	0.537	1.000	-1.277	1.975
	Tetric EvoCeram bulk fill	-0.975	0.537	0.794	-2.601	0.651
	Filtek	-1.377	0.537	0.156	-3.003	0.250
	Gradia	-0.163	0.537	1.000	-1.789	1.463
Tetric EvoCeram bulk fill	SDR	1.324	0.537	0.196	-0.302	2.950
	Tetric EvoCeram flow	0.975	0.537	0.794	-0.651	2.601
	Filtek	-0.402	0.537	1.000	-2.028	1.224
	Gradia	0.812	0.537	1.000	-0.814	2.438
Filtek	SDR	1.726*	0.537	0.031	0.100	3.352
	Tetric EvoCeram flow	1.377	0.537	0.156	-0.250	3.003
	Tetric EvoCeram bulk fill	0.402	0.537	1.000	-1.224	2.028
	Gradia	1.214	0.537	0.311	-0.412	2.840
Gradia	SDR	0.512	0.537	1.000	-1.114	2.138
	Tetric EvoCeram flow	0.163	0.537	1.000	-1.463	1.789
	Tetric EvoCeram bulk fill	-0.812	0.537	1.000	-2.438	0.814
	Filtek	-1.214	0.537	0.311	-2.840	0.412

Based on estimated marginal means

*. The mean difference is significant at the 0.05 level.

b. Adjustment for multiple comparisons: Bonferroni.

Univariate Tests

Dependent Variable: SBS

	Sum of Squares	Df	Mean Square	F	Sig.	Partial Eta Squared	Noncent. Parameter	Observed Power ^a
Contrast	14.392	4	30.244	3.569	0.017	0.322	14.276	0.810
Error	30.244	30	1.008					

The F tests the effect of Composite. This test is based on the linearly independent pairwise comparisons among the estimated marginal means.

a. Computed using alpha = 0.05

Table 4. Sapiro-Wilk test of normality

	Composite	Kolmogorov-Smirnov ^a			Shapiro-Wilk		
		Statistic	df	Sig.	Statistic	df	Sig.
SBS	SDR	0.245	7	0.200*	0.864	7	0.165
	Tetric EvoCeram flow	0.226	7	0.200*	0.863	7	0.161
	Tetric EvoCeram bulk fill	0.172	7	0.200*	0.949	7	0.719
	Filtek	0.208	7	0.200*	0.950	7	0.727
	Gradia	0.243	7	0.200*	0.853	7	0.131

*. This is a lower bound of the true significance.

a. Lilliefors Significance Correction

Although the binding strength of Tetric evo ceram bulk fill composites was the highest (5.84 ± 0.72) compared with the other two bulk fill composites, SDR (4.52 ± 0.65) and Tetric evo ceram flow (4.87 ± 1.03), no statistically significant differences between bulk fill composites were registered.

Table 5. Leven test of homogeneity

		Levene Statistic	df1	df2	Sig.
SBS	Based on Mean	2.291	4	30	0.083
	Based on Median	1.498	4	30	0.228
	Based on Median and with adjusted df	1.498	4	18.465	0.244
	Based on trimmed mean	2.198	4	30	0.093

Table 6. Post Hoc Tests

(I) Composite	(J) Composite	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
SDR	Tetric EvoCeram flow	-0.3493	0.53669	0.965	-1.9060	1.2074
	Tetric EvoCeram bulk fill	-1.3239	0.53669	0.126	-2.8807	0.2328
	Filtek	-1.7258*	0.53669	0.024	-3.2825	-0.1691
	Gradia	-0.5121	0.53669	0.873	-2.0688	1.0447
Tetric EvoCeram flow	SDR	0.3493	0.53669	0.965	-1.2074	1.9060
	Tetric EvoCeram bulk fill	-0.9746	0.53669	0.384	-2.5314	0.5821
	Filtek	-1.3765	0.53669	0.103	-2.9332	0.1802
	Gradia	-0.1628	0.53669	0.998	-1.7195	1.3940
Tetric EvoCeram bulk fill	SDR	1.3239	0.53669	0.126	-0.2328	2.8807
	Tetric EvoCeram flow	0.9746	0.53669	0.384	-0.5821	2.5314
	Filtek	-0.4019	0.53669	0.943	-1.9586	1.1549
	Gradia	0.8118	0.53669	0.563	-0.7449	2.3686
Filtek	SDR	1.7258*	0.53669	0.024	0.1691	3.2825
	Tetric EvoCeram flow	1.3765	0.53669	0.103	-0.1802	2.9332
	Tetric EvoCeram bulk fill	0.4019	0.53669	0.943	-1.1549	1.9586
	Gradia	1.2137	0.53669	0.186	-0.3430	2.7705
Gradia	SDR	0.5121	0.53669	0.873	-1.0447	2.0688
	Tetric EvoCeram flow	0.1628	0.53669	0.998	-1.3940	1.7195
	Tetric EvoCeram bulk fill	-0.8118	0.53669	0.563	-2.3686	0.7449
	Filtek	-1.2137	0.53669	0.186	-2.7705	0.3430

Based on observed means.

The error term is Mean Square(Error) = 1.008.

*. The mean difference is significant at the 0.05 level.

Table 7. Tukey HSD^{a,b} test
SBS

Tukey HSD^{a,b}

Composite	N	Subset	
		1	2
SDR	7	4.5223	
Tetric EvoCeram flow	7	4.8716	4.8716
Gradia	7	5.0344	5.0344
Tetric EvoCeram bulk fill	7	5.8462	5.8462
Filtek	7		6.2481
Sig.		0.126	0.103

Means for groups in homogeneous subsets are displayed.

Based on observed means.

The error term is Mean Square(Error) = 1.008.

a. Uses Harmonic Mean Sample Size = 7.000.

b. Alpha =0.05.

Discussion

The purpose of this study was to do research and compare SBS of the composite resins with the bulk application technique and the conventional composite resins with a layered material application technique. Thus, although Bulk Fill composites showed lower SBS values than the conventional composite, a significant difference between Filtek and SDR was registered. Thus, the first null-hypothesis was partially rejected.

Agarwal et al.⁸ evaluated the cervical marginal and internal adaptation of composite resins with posterior full charge with different viscosities, before and after thermo cycling. They found that liquid bulk fill composites demonstrated significantly better marginal adaptation. However, researchers found no difference between conventional composites and bulk fill composites⁸.

Previous studies have shown that samples tested with composite resins with bulk fill application technique show better polymerization depth than those treated with conventional composite resins⁹. However, in this study, there was no significant difference between the three bulk fill composites, despite the fact that tetric evo ceram bulk fill demonstrated higher SBS than the SDR and Tetric evo ceram flow, which is in line with a study by Hakan Colak et al. Thus, the second null-hypothesis was accepted.

In his in vitro study, Mirosław Orłowski et al. comparing the marginal integrity between the four types of Bulk Fill composite materials came to the conclusion

that bulk fill liquid and sonic activated liquid composites have better marginal adaptation than bulk fill composites in paste form¹⁰.

Peutzfeldt and Asmussen showed that fluidity level in the application of the composite material affects the marginal adaptation; the increased fluidity of the composite makes it better to adhere to the walls of the cavity¹¹. In our study, although there was a difference between the strength of the relationship in the three bulk fill composites, the results obtained are not extremely significant, which is correlated with the study of Alrahlah et al.¹².

In this study, bulk fill composite filling showed statistically similar SBS values. This may be because they have shown very similar mechanical properties and consistency^{13, 14}. As confirmed in various in vitro studies, resinous full-fill composite materials can be polymerized into a thicker layer of material, since the degree of polymerization and micromechanical properties can be maintained within the 4-millimeter layers at a radiation time of up to 20 seconds¹⁰.

Conclusion

Although this study has a number of limitations, the results indicate that the application of bulk fill composite material results in an acceptable SBS comparable to that achieved through conventional RBC. As such, bulk fill composites can provide reliable alternatives to conventional composites. This can be of potential benefit to dentists because the bulk fill composite materials are simpler than the conventional composites and can be more efficiently applied. However, further research in this area is required to better understand how the relation forces of these adhesion systems relate to clinically acceptable conditions.

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