Flexural and Microtensile Bond Strength of Bulk Fill Materials

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Aim: *The aim of this study was to evaluate the flexural and μTBS of bulk-fill materials.* ***Study design:*** *Bulk-fill materials SDR, X-trabase (XTR) and TetricEvoCeramBulkFill (EVO) were used in this study. To test flexural strength, 25x2x2mm samples were prepared and tested with three point bending test. To test the microtensile bond strength (μTBS), two blocks (4x4x4mm) were prepared for each material. In Group A+B, acid-etching was applied to the surface of one of these blocks and no acid-etching was applied in Group B. After applying bonding agent, two blocks were placed into the mold and composite resin (COMP; Tetric N-Ceram) was applied with incremental layering. To evaluate the μTBS of primary dentin, the bulk-fill materials were applied to flat dentin up to 4mm. The new blocks and the teeth were sectioned to obtain sticks and the sticks were loaded in tension until failure. Flexural and microtensile bond strength was calculated based on failure load.* ***Results:*** *The ranking of materials with regards to flexural strength values were SDR>XTR>EVO>COMP, respectively. In GroupA+B, the μTBS values were XTR>SDR>EVO and were XTR>EVO>SDR in GroupB (p>0.05). The μTBS values of these materials to dentin were XTR>EVO>SDR (p>0.05).* ***Conclusion:*** *Within the limitations of this study, the use of a bonding agent without acid-etching showed positive interactions between base materials and composite resin and there were no significant differences in μTBS of these materials to dentin*

Key words: *SDR, X-Trabase, Evoceram Bulk-Fill, Flexural Strength, microtensile bond strength.*

INTRODUCTION

Since first reported in the early 1960s[1], resin based composites (RBC) are becoming more popular in dentistry day by day. RBCs have been used for many purposes. Along with their clinical use, some problems such as polymerization shrinkage, lack of adaptation to cavity walls, microleakage resulting with secondary caries, the loss of restorations, pulp inflammation, post-operative sensitivity, micro or macro cracks both in RBC and tooth surface have emerged[2-5]. To overcome these problems, manufacturers have attempted to improve the physical and mechanical properties of RBC materials. Many techniques and many developments have been introduced accordingly. Incremental layering, soft-cure or pulse- delay cure methods, application of an intermediate layer are used to minimize the polymerization shrinkage and to have a tight marginal seal[1-3,5-8]. Among these, the most widely accepted technique is incremental layering[9]. However, this technique has some disadvantages including the possibility of incorporating voids or contamination between layers, bond failures between increments, difficulty in placement because of limited access in conservative preparations, and the increased time required to place and polymerize each layer as opposed to delivering the resin in a single bulk layer[6,9-11]. It was also reported that the technique produced higher polymerization shrinkage stresses than bulk filling[3].

Mostly, the clinicians perform successful treatments in dental practice although RBC is a technique-sensitive procedure involving acid etching, bonding and placing to cavities. The researches on minimizing the polymerization shrinkage stress introduced a new class of restorative material called “bulk-fill materials”. SDR (*SDR*: Dentsply Caulk, Milford, DE, USA), Tetric EvoCeram Bulk-fill (*EVO*: Ivoclar Vivadent, Schaan, Liechtenstein) and X-tra Base (*XTR*: Voco, Cuxhaven, Germany) were marketed as bulk-fill materials and composites for using beneath the conventional RBC materials with a reported depth cure in an excess of 4 mm. According to the manufacturers, the reduced filler particle amount shows slow polymerization resulting with less polymerization shrinkage stress[1].

Deep cavities of primary teeth and the polymerization shrinkage of RBC require pediatric dentists to be more cautious. The bulk-fill materials may be an alternative to avoid the polymerization

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shrinkage and also recurrent caries, post-operative pain, swelling and abscess.

The aim of this study was to evaluate the flexural and μTBS of bulk-fill materials regarding; the flexural strength, the μTBS to primary dentin and the μTBS to RBC.

MATERIALS AND METHOD

Ethical approval was obtained from Cumhuriyet University Clinical Research Ethic Committee to collect samples for this study (2012-09/10).

The information about the tested restorative materials in this study is shown in Table 1.

Ten specimens were prepared in a stainless steel mold (2x2x25 mm). The materials were applied into the mold carefully to avoid air bubbles and light cured with LED curing light (with three over-lapping exposures of 20s per side, Light Intensity 1,200 mW/cm^2, Bluephase, Ivoclar Vivadent). Tetric N-Ceram composite was used to compare flexural strength of these materials. Specimen edges were manually finished with an 800-grit SiC-paper. The specimens were stored in distilled water at 37°C for 24 h. The three-point bending test was used to measure the flexural strength on a computer-controlled Universal Testing Machine (LF Plus, LLOYD Instruments, Ametek, Inc., England) at a crosshead-speed of 0.5 mm/min. The data were collected and recorded using the software Statistical Packages for Social Sciences for Windows 15.0 (SPSS Inc., Chicago, IL, USA).

Microtensile Bond Strength Testing:

Two cubes (4x4x4mm) were prepared in a stainless steel mold from each tested material and divided into two subgroups shown in Figure 1.

For the first group (Group ***A+B***, ***A***cid-etch+***B***ond), the surface of the specimens were etched with phosphoric acid (FineEtch 37, Spident Co., Ltd, Korea) for 20s and Prime&Bond NT (Dentsply Detrey, Konstanz, Germany) was applied for 20 s, gently dried, and light-cured with an LED curing light (Bluephase, Ivoclar Vivadent) for 15s.

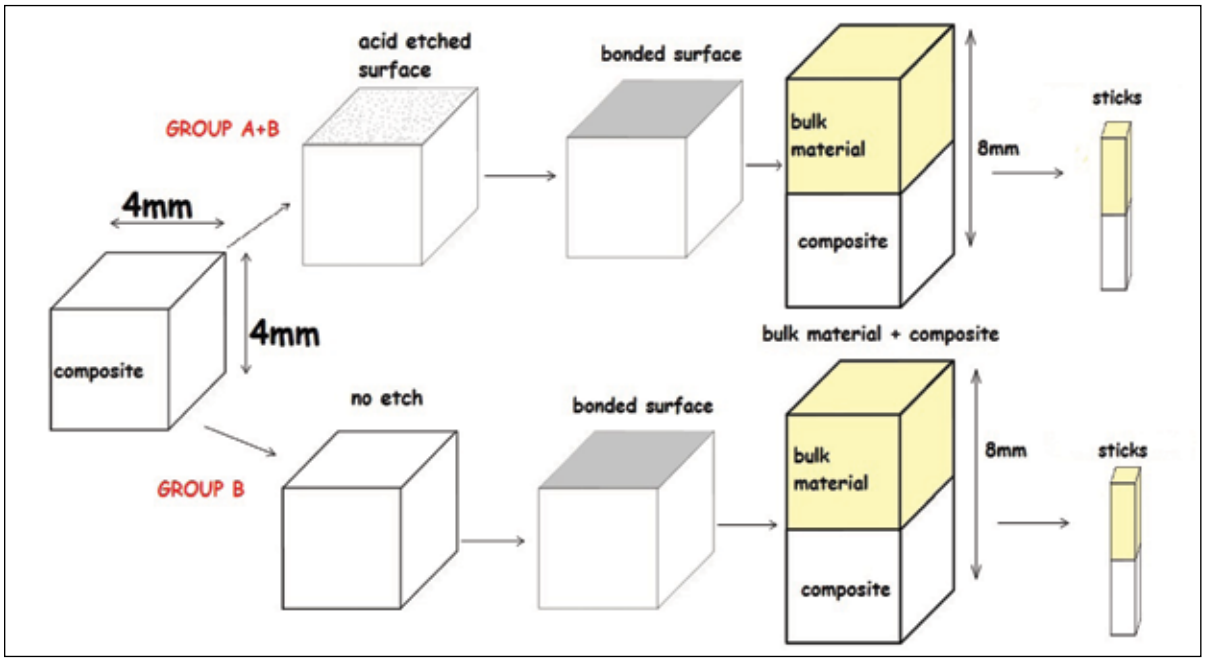
For the second group (Group ***B***, ***B***ond), Prime&Bond NT was applied for 20s, gently dried, and light-cured with an LED curing light (Bluephase, Ivoclar Vivadent) for 15s without etching.

The cubes were placed in mold again and a composite resin

Table 1: the codes, product names, manufacturers, lots and filler systems of the bulk fill materials

Code	Product	Manufacturer	Lot	Filler System
SDR	Surefil SDR flow	Dentsply Caulk, Milford, DE, USA	10028	Barium/strontium-alumino-fluoro-borosilicate glass (68% by wt and 44% by vol)
EVO	Tetric EvoCeram	Bulk-fill Ivoclar Vivadent, Schaan, Liechtenstein	PM0213	Ba glass, YbF3, oxides and prepolymers (80% by wt and 60% by vol)
XTRA	X-tra base	Voco, Cuxhaven, Germany	V 45252	Inorganic fillers (75% by wt and 58% by vol
COMP	Tetric N-Ceram	Ivoclar Vivadent, Schaan, Liechtenstein	P35843	Paste of dimethacrylates, inorganic fillers, YbF3, initiators, stabilizers and pigments

Fig 1: Schematic view of the etched and non-etched groups



(COMP; Tetric N-Ceram, Ivoclar Vivadent, Liechtenstein) was applied on the cubes with incremental layering technique in a thickness of 2 mm and light cured. The new cubes were then stored in distilled water for 24h.

For testing the microtensile bond strength of primary dentin, three human primary molar teeth were used in this study. The teeth were stored in distilled water and used within one month. The coronal one-third of the tooth and surrounding enamel was removed using an Isomet low-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA). A stereomicroscope was used to check for the absence of enamel and pulp tissue on the resultant substrate. Prime&Bond NT was applied to dentin for 20 s, gently dried, and light-cured with an LED curing light for 15s. The bulk fill materials were applied to dentin with a metal matrix band up to 4 mm and light cured. Then the teeth were stored in distilled water for 24 h (Fig 2).

At the end of 24h, the cubes and the teeth were longitudinally sectioned in both "x" and "y" directions with a slow-speed saw under water cooling to obtain bonded sticks with a cross-sectional area between 0.7 mm^2-1 mm^2. For each group, 10 sticks were obtained. The sticks were stored in distilled water for 24h. Then the sticks were fixed to the universal testing machine with cyanoacrylate adhesive plus an accelerator. The specimens were stressed in tension until failure using a universal testing machine (LF Plus, LLOYD Instruments, Ametek, Inc., England) at a crosshead speed of 0.5 mm/min, and the μTBS was calculated and expressed in MPa.

Statistical analysis:

Shapiro-Wilk test was performed to evaluate suitability of normal distribution for flexural strength and normal distribution was found. Therefore, One-way Anova and Tamhane tests were used to analyze the data collected from flexural strength.

Shapiro-Wilk test was performed to evaluate suitability of normal distribution for μTBS and normal distribution was found without homogeneous variances. Therefore, two-way Anova and Fisher's LSD test were made for comparing μTBS values. The level of significance was set at $p<0.05$.

RESULTS

Flexural strength

The mean flexural strength values (in MPa) of the sticks were SDR 45.00, XTR 40.96, EVO 24.90 and COMP 22.34 (Table 2a). There were no significant differences between SDR and XTR ($p>0.05$). The comparison of SDR with EVO and COMP revealed that SDR showed values significantly higher than EVO and COMP ($p<0.05$) (Table 2b). No significant differences were found between XTR and EVO ($p>0.05$), whereas significant differences between XTR and COMP were observed ($p<0.05$). There were no significant differences between EVO and COMP ($p>0.05$).

Table 2a: the mean strength values (in MPa) and standard deviation *(sd)* for flexural strength

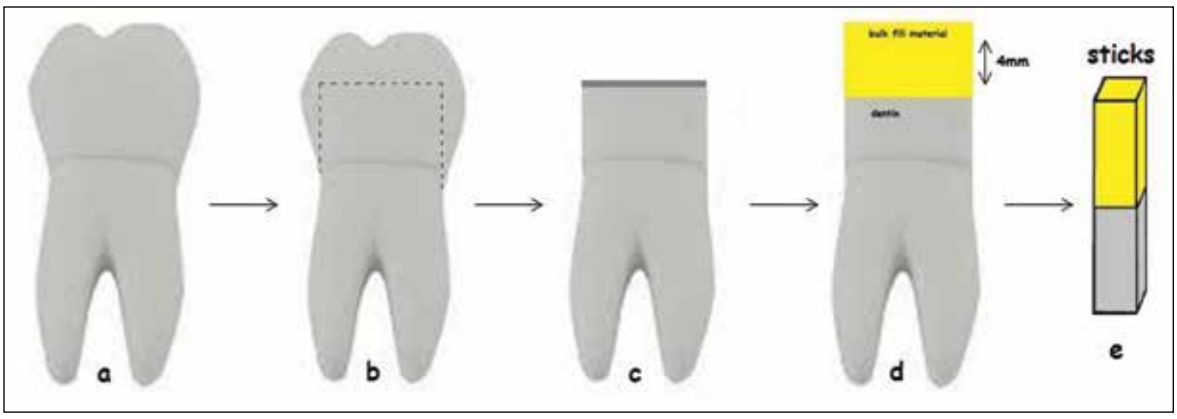
	n	Mean± sd
SDR	10	45,00±3,58
XTRABASE	10	40,96±15,98
EVOCERAM	10	24,90±7,34
COMPOSİTE	10	22,34±3,00
Total	40	33,30±13,24

Table 2b: the table shows multiple comparisons of the groups.

Groups		Mean Difference	Sig.
SDR	Composite	22,66*	,000
	XTRABASE	4,03	,973
	EVOCERAM	20,09*	,000
XTRABASE	Composite	18,62*	,030
	SDR	-4,03	,973
	EVOCERAM	16,05	,076
EVOCERAM	Composite	2,56	,907
	SDR	-20,09*	,000
	XTRABASE	-16,05	,076
Composite	SDR	-22,66*	,000
	XTRABASE	-18,62*	,030
	EVOCERAM	-2,56	,907

* the mean difference is significant at the .05 level
***sig**: significance*

Fig 2: *schematic view of obtaining sticks:* **a:** the primary teeth, **b:** surrounding enamel removed from the dashed line, **c:** adhesive applied to dentine, **d:** bulk fill material applied to surface up to 4mm **e:** the sticks obtained from



Microtensile Bond Strength

The mean μTBS values were seen in Table 3a and the defination of each factor were seen in table 3b. For each material tested in this study, inter group comparison of the groups were seen in table 3c. There were no significant differences between SDR and XTR both in Group A+B and Group B ($p>0.05$). With regards to EVO, Group A+B showed significantly lower μTBS values than Group B ($p<0.01$). For the materials tested in this study, only bonding application seemed to be sufficient when placing to cavities. Multiple comparisons revealed that there were no significant differences between the groups XTR-SDR (p=0.262), XTR-EVO (p=0.231) and SDR-EVO (p=0.938) (Table 3d).

In Group A+B, the μTBS values were XTR>SDR>EVO and EVO showed the lowest μTBS values. There were no significant differences between SDR-XTR (p=0.556) but there were significant differences between SDR-EVO ($p<0.01$) and XTR-EVO ($p<0.001$) (Table 3d).

The μTBS values of Group B were XTR> EVO> SDR, respectively ($p>0.05$) and also there were no significant differences between the groups XTR-SDR (p=0.262), XTR-EVO (p=0.231) and SDR-EVO (p=0.938).

The μTBS values of the tested materials to primary dentin were shown in table 4 and the highest values were XTR>EVO>SDR. There were no significant differences between the materials ($p>0.05$).

DISCUSSION

Although there is a huge demand for esthetic restorative materials in the posterior teeth, polymerization shrinkage is the most important problem for dental practitioners and the use of the bulk-fill materials will be easier and more convenient in deep cavities.

The flexural strength values of bulk fill materials suggested by the manufacturers (XTR 133 MPa, SDR 115 MPa and EVO bulk fill 120 MPa) are generally higher than the results of the present study. In this study, the MPa values of the sticks were 45.00 MPa for SDR, 40.96 MPa for XTR, 24.90 MPa for EVO and 22.34 MPa for the Composite. Sticks of 25x2x2mm were used in the present study and the differences between this study and manufacturer's results might be due to the differences between the experimental techniques, such as crosshead speed, sample sizes, curing times and the abilities of the curing devicies[12,13]. For decreasing polymerization shrinkage, the manufacturers changed the filler systems of these materials and the reason for higher flexural strength values of SDR and XTR than composite and EVO should probably be the effect of the different filler systems and the filler volumes of these materials. The authors stated that increased filler content promotes increased flexural strength, increased elasticity modulus and decreased polymerization shrinkage[13,14], but they remain uncertain[14]. The size of the filler particles of these materials may have an effect on their flexural strength. Scanning Electron Microscope (SEM) images of these materials revealed that SDR had the biggest particle size when compared with

Table 3a: the mean microtensile bond strength values and standard deviation *(sd)* of the groups

MATERIAL	GROUPS	n	Mean ± sd (MPa)
XTRABASE	GROUP A+B	10	34,16±8,98
	GROUP B	10	36,42±6,95
	TOTAL	20	35,29±7,90
SDR	GROUP A+B	10	32,22±7,42
	GROUP B	10	32,70±9,22
	TOTAL	20	32,46±8,15
EVOCERAM	GROUP A+B	10	29,09+4,06
	GROUP B	10	33,08+6,13
	TOTAL	20	26,67+7,79

Table 3b: Interaction of the factors (material- acid-etching)

Source	Type III Sum of Squares	df	Mean Square	F	Sig
Corrected Model	1466,180[a]	5	293.236	5,438	,000
Intercept	59453,758	1	59453,758	1102,648	,000
Material	772,897	2	386,449	7,167	,002
Acid-etching	340,388	1	340,388	6,313	,015
Material-acid-etching	352,895	2	176,447	3,272	,046
Error	2911,630	54	53,919		
Total	63831,567	60			
Corrected Total	4377,810	59			

Table 3c: inter group comparison of the groups

MATERIAL	GROUPS	Mean Difference	Sig.
XTRABASE	GROUP B GROUP A+B	2,26	,494
SDR	GROUP B GROUP A+B	,48	,883
EVOCERAM	GROUP B GROUP A+B	11,54	,001

***sig**: significance*

Table 3d: the mean difference and significance (sig) of the groups

GROUPS MATERIAL	MATERIAL		Mean Difference	Sig.
GROUP A+B	XTRABASE	SDR	1,947	,556
		EVOCERAM	13,267*	,000
	SDR	XTRABASE	-1,947	,556
		EVOCERAM	11,320*	,001
	EVOCERAM	XTRABASE	-13,267*	,000
		SDR	-11,320*	,001
GROUP B	XTRABASE	SDR	3,724	,262
		EVOCERAM	3,982	,231
	SDR	XTRABASE	-3,724	,262
		EVOCERAM	,258	,938
	EVOCERAM	XTRABASE	-3,982	,231
		SDR	-,258	,938

XTR, COMP and EVO and this might be the possible explanation of the higher flexural strength values of these materials (Fig 3).

In this study, Prime & Bond NT was preferred, which is a universal bond, and commonly used by pediatric dentists under compomer restorations. Another reason for choosing only one type of bonding agent was to standardize the effects of bonding agents. Microtensile bond strength values were studied several times in primary teeth. According to the authors, when Prime & Bond NT is used with a composite in primary teeth, the μTBS values were found 45.9 MPa[15], 40.8 +/- 13.4 MPa[16], 38.4MPa[17], 22 MPa[18] and 12.9 MPa[19]. In the present study, μTBS values were found 16,63 MPa for SDR, 17.65 MPa for XTR and 17.26 MPa for EVO. The results were similar to the previous studies and these findings indicate that these materials could be used as composites.

To the best of the author's knowledge, adhesion of the composites to bulk materials was not a subject of any research before. When repairing a composite or bonding to another composite, the chemical composition of the composites, surface roughness and bonding agents are important factors[20,21]. The chemical composition of the bulk-fill materials and the composite used in this study were different (Table 1). In Group A+B, acid-etching was used to see the effects of acid-etched and non-etched surfaces on bond strength. Acid etching is commonly used to have a roughened surface and this will lead to a better adhesion, but in the present study, there were no significant differences between acid-etched and non-etched surfaces with the exception of EVO. In group B, acid-etching was not used and there were no significant differences between the materials. The results of the present study led to the conclusion that the use of a bonding agent solely would be sufficient prior to placing a composite resin restoration on bulk-fill materials.

CONCLUSION

The aim of this study was to compare the flexural strength and microtensile bond strength of these materials. There were no significant differences between μTBS values when these materials were applied on dentin and there were no significant differences between the groups when bonding composite to these materials. The use of bulk-fill materials will be useful to avoid the polymerization shrinkage, therefore postoperative sensitivity, recurrent caries, abscess etc., but further in vivo studies are necessary to validate these findings.

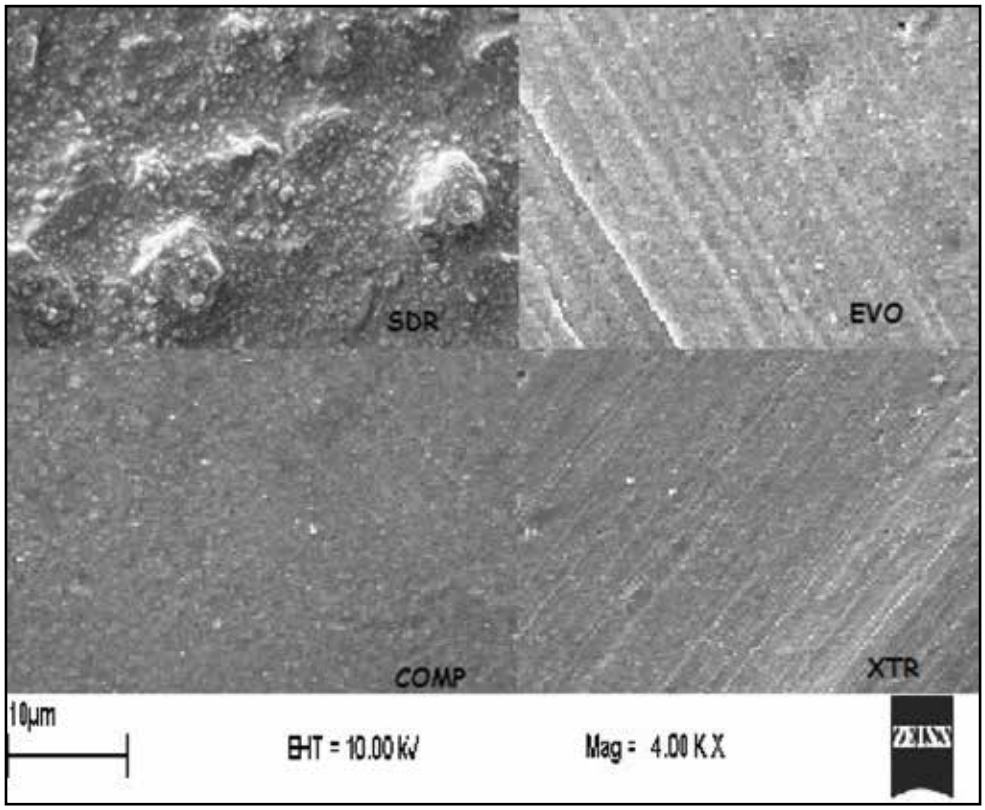
Table 4: the means and standard deviation *(sd)* of the microtensile bond strength values of the materials to dentin

Material	n	Mean±sd
SDR	10	16,63±2,69
XTRABASE	10	17,65±2,25
EVOCERAM	10	17,26±1,23

Fig 3: SEM images of the materials tested in the study *and SDR has the biggest particle size*

EVO: *TetricEvoCeramBulkFill* ***COMP:*** *Composite*

XTR: *X-trabase*



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