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Title: Shear Bond Strength of Aged Composite Restorations Repaired With a Universal Injectable Composite

Running Head: Repair bond strength of novel composites

Authors: Nihan Gönülo¹, Lena Almasifar¹, Özge Gizem Cabadağ¹, Tuğba Misilli²,

Elif Kalyoncuoğlu³

Institutions: ¹Department of Restorative Dentistry, Faculty of Dentistry, Ondokuz Mayıs University, Samsun, Turkey

²Department of Restorative Dentistry, Faculty of Dentistry, Çanakkale Onsekiz Mart University, Çanakkale, Turkey

³Department of Endodontics, Faculty of Dentistry, Ondokuz Mayıs University, Samsun, Turkey

Address for correspondence: Özge Gizem Cabadağ

E-mail: gizemyndny@outlook.com

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Abstract

Purpose: The purpose of this study was to analyze the shear bond strength of a universal injectable composite used in the repair of aged composites.

Materials and Methods: 100 disc-shaped specimens (8mm×2mm) were produced using 5 different composites (n=20) (Gradia Direct Posterior, Tetric N Ceram BulkFill, Filtek Z250, SonicFill and Filtek BulkFill Posterior). Specimens were polymerized using an LED light curing unit for 20 s and stored at 37°C in distilled water for 3 weeks. Specimens were subdivided into two groups per composite for repair using either the same composite used for the specimen or G-aenial Universal Flo. Following acid-etching and silane application, a universal adhesive (G-Premio BOND) was applied and light-cured. The repair materials were placed on the bonded surfaces of the specimens and polymerized in silicone molds (2mm×2mm). After thermocycling to simulate aging, shear bond strength (SBS) was tested using a universal testing machine at a crosshead speed of 1 mm/min. Failure modes were examined using a stereomicroscope at ×40 magnification.

Results: No statistically significant differences were found among the tested composites repaired with their own substrates. However, the SBS SonicFill and Filtek Bulk Fill Posterior groups had significantly lower bond strengths when repaired with G-aenial Universal Flo in comparison to repairs made with their own substrates ($p<0,05$).

Conclusion: When repaired with their own substrates, reliable bond strengths were obtained for all the composites tested.

Keywords: Dental Materials, shear bond strength, bulk fill composites, repair

Introduction

Over the last decade, dental resin based composites (RBCs) have risen in popularity in response to growing needs of patients (1,2). Dynamic changes in pH and temperature in the oral cavity caused by saliva, diet and aging result in degradation of resin composites (3). Despite recent improvements in material performance, clinical problems such as fracture, micro-leakage, chipping, discoloration, wear and other restoration defects may occur (4). When function or esthetics is compromised, the clinician must replace or repair the restoration using one of the various alternatives available. In the past, replacement was the only option available; however, this resulted in an undesirable loss of

dental structure and extension of the cavity (5). In line with the concept of minimally invasive dentistry, several clinical studies have reported that the more conservative option restoration repair is able to increase restoration longevity while preserving dental structures and reducing operative trauma (4,6).

One important factor influencing repair success or failure is the interfacial bond between the old and new composite resins (7). In clinical practice, the presence of an oxygen inhibition layer maintains the bond between two layers of composite (4,6). Various chemical and micromechanical methods such as mechanical roughening, etching with hydrofluoric or phosphoric acid, air abrasion, resin coating and silanization may be used, either alone or in combination, to improve bonding between old and new resin composites (4). Studies have reported repair strengths ranging from 25%-82% of the SBS values of the composite substrate (7).

While much data is available on the SBS of conventional composites, few studies have examined the SBS of bulk-fill composites, which were more recently introduced into clinical use to make the application process quicker and easier. In comparison to conventional composites, bulk-fill composites can be applied in deeper layers, and studies have demonstrated adequate polymerization of layers up to 4 mm in thickness (8). Various strategies have been applied to increase the depth of polymerization of bulk-fill composites, including adding a non-camphorquinone initiator and increasing translucency by changing filler size, concentration and refractive index (9).

G-aenial Universal Flo (GC Corporation, Tokyo, JAPAN) is a high-fill injectable composite that has come into recent clinical use. Due to its high viscosity and improved mechanical properties, it is assumed to be similar to conventional composites (10). According to the manufacturer, G-aenial Universal Flo consists of a revised formulation of strontium glass whose filler particles have been reduced in size to 200 nanometers. The application of silane to the nano-sized glass surface enhances the adhesion between the glass particles and the resin matrix to provide greater durability and hydrolytic stability (11).

The aim of this *in vitro* study was to analyze the shear bond strengths of composites used in the posterior region when repaired using their own substrates or with G-aenial Universal Flo. The null hypothesis tested was that there would be no difference in the shear bond strength of composites to their own substrates and to an injectable universal composite.

Materials and Methods

The compositions of the materials and manufacturer details are listed in *Table 1*, and a study flowchart is shown in *Figure 1*.

Sample Preparation

Using a Teflon mold (8mm x 2mm), 20 disc-shaped specimens were created from 5 different resin composites (Filtek Bulk Fill Posterior, Filtek Z250, Gradia Direct Posterior, SonicFill and Tetric N-Ceram Bulk Fill), for a total of 100 specimens. The resin composite was condensed with a filling instrument and covered with a Mylar strip and pressed with glass coverslips to create a smooth surface. Polymerization was performed with a third-generation light curing unit (VALO, Ultradent, Utah, USA) for 20 seconds in standard mode. Light intensity was periodically checked by a radiometer (LED Radiometer, SDI, Australia) after the processing of every five specimens and it was verified to be higher than 1000 mW/cm².

Specimens were removed from the molds, roughened with 600 and 1,200 grit silicon carbide paper and then cleaned with an ultrasonic device for 10 minutes. Similar to the previous studies, aging was simulated by storing all samples in distilled water at 37°C for 3 weeks (12-14). Prior to repair procedure, as stated in some studies the samples were etched with 37% phosphoric acid (Bisco Select HV® etch, Schaumburg, USA) for 15 s, rinsed with water and air-dried (15,16). A silane coupling agent (GC Ceramic Primer II- GC Corporation, Tokyo, JAPAN) was then applied and air-dried for 10 s, and a universal adhesive (G-Premio BOND- GC Corporation, Tokyo, JAPAN) was applied in accordance with the manufacturers' recommendations and polymerized with the same LED unit (VALO) for 10 s in standard mode.

Each group of composite specimens was then divided into two subgroups according to repair material (either their own substrate or G-aenial Universal Flo). A silicone mold (2mm×2mm) was placed over the composite sample, which was filled with the repair material and vertically photopolymerized for 10 s. Samples were then stored in distilled water at 37°C for 48 hours and they were subjected to 500 thermocycles in water between 5°C and 55°C at a dwell-time of 30 s as stated in previous studies (17,18).

Bond Strength Testing

Bond strengths of samples were tested using a universal testing machine (Llyod Instruments, LRX Plus 6, Amates, Leicester, UK) (*Image-1*). Specimens were screwed to the lower compartment of the testing machine and subjected to a load of 5 kN at a 90° angle and a cross-head speed of 1 mm/min

until fracture. The load required to dislodge each specimen was recorded in Newtons and then converted into megapascals (MPa) by dividing the fracture load (Newton) by the repair surface area. Failure type was identified using a stereomicroscope (Nikon SMZ 1500, Tokyo, Japan) at a magnification of $\times 40$ and classified as adhesive failure (fracture between the composite and adhesive), cohesive failure (fracture within the composite), or mixed adhesive and cohesive failure (both composite and adhesive residue detected on the surface).

Statistical Analysis

Statistical analysis was performed using SPSS software, version 17.0 (Statistical Package for Social Sciences, SPSS Inc., Chicago, IL, USA). SBS means and standard deviations were calculated for all groups. Differences in mean SBS between groups were compared by two-way analysis of variance (ANOVA) and a post-hoc Tukey test, and differences in failure mode distribution were identified by chi-square test, with the level of significance set at $p < 0.05$.

Results

SBS values of the tested materials are presented in *Table 2*. No statistically significant differences were found among the tested composite groups when repaired with their own substrates. However, when repaired with G-aenial Universal Flo, the Filtek Z250 Group exhibited the highest SBS values (34.14 ± 14.89), and the Sonic Fill Group had the lowest SBS values (21.12 ± 7.95), and the difference between the two groups was statistically significant ($p < 0.05$).

Intragroup comparisons showed the mean SBS of Sonic Fill and Filtek Bulk Fill Posterior to decrease significantly when repaired with G-aenial Universal Flo as compared to their own substrates ($p < 0.05$), whereas the bond repair material did not significantly affect the mean SBS values of Gradia Direct Posterior, Tetric N-Ceram Bulk Fill, or Filtek Z250.

The distribution of failure modes for all groups is shown in *Table 3*. Adhesive failures were more frequent in both the Sonic Fill group repaired with its own substrate and the Sonic Fill group repaired with G-aenial Universal Flo ($p < 0.05$).

Discussion

This study assessed bond strengths of new composites used in the posterior region when repaired with their own substrates and an injectable universal composite. Both Sonic Fill and Filtek Bulk Fill had mean bond strengths that were significantly lower when repaired with G-aenial Universal Flo as compared to their own substrates; thus, the null hypothesis that there would be no differences in the shear bond strength of the tested composites when repaired with their own substrates or with an injectable universal composite was partially rejected.

The repair of composite restorations is considered a conservative option that offers the advantages of increased durability and longevity, preservation of dental structure, faster treatment and less strain on the patient during treatment (2). However, besides these advantages, repair entails the risk of weakening the restoration. Different studies have reported interfacial bond strengths ranging between 25% and 80% of the cohesive strength of the substrate materials (4,19). Factors such as chemical differences between different resins used in repair process, surface treatment, and the length of time between initial restoration and repair have a significant effect on bond strength of repaired restorations (1,4). Under clinical conditions, the type of composite used for the initial restoration is generally unknown to the operator performing the repair. For this reason, this study examined repairs made with the same material as the original composite substrate as well as repairs made with a high-fill flowable composite (G-aenial Universal Flo).

Although shear bond strength testing is frequently criticized for its unhomogeneous stress distribution at the interface (20), it is still the most widely used method for evaluating the bonding effectiveness of restoration repairs (7,20) because it is easy to prepare samples and implement the test protocol (5,21) and because it imitates oral clinical conditions better than other methods (22,23).

Flowable composites are widely used in clinical practice today. High-fill flowable composites are particularly recommended for posterior restorations. Kitasako *et al.* (10) reported the clinical performance of the high-fill flowable composite G-aenial Universal Flo used in the posterior region to be comparable to that of a conventional composite after 36 months.

Bulk-fill materials have grown in popularity due to their ease of application (24). The particular advantages offered by bulk-fill composites when used in the posterior, stress-bearing region make their mechanical properties especially important. However, there is little information available in the literature about their clinical performance and repair (25,26).

Previous studies have shown that the original filling material has a greater effect on bond strength than the repair material (27). As the composite ages, the number of free radicals within the resin

structure that provide adhesion between the different composite layers decreases (28). Successive changes in temperature that occur in the oral environment also weaken the bond between the resin matrix and filler (5). Due to the differences in thermal expansion coefficients, the composite resin matrix and inorganic fillers are affected at different rates, resulting in weaker interfacial bond strength (2). Because the length of time and environmental conditions of clinical service also affect the outcome of composite repair, *in vitro* studies need to take these conditions into consideration (5).

The preferred methods for simulating aging and interfacial bond stresses are storage in water and thermal cycling (2,29), which tests thermal stress caused by contact with liquid and temperature changes between 5°C-55°C. In this study, samples were stored in 37°C in distilled water for three weeks (4,30) and thermocycled for 500 cycles to simulate thermal strain caused by exposure to liquids and temperature changes.

Papacchini *et al.* (1) stated that higher composite-to-composite bond strength is obtained with a flowable resin, and thus a flowable resin is recommended for use as an intermediate agent in composite repair. For this reason, G-aenial Universal Flo, a highly filled flowable composite, was tested as a repair material in this study.

Repair of a composite-resin restoration generally requires partial removal of both the restoration and adjacent enamel and dentin (7). In clinical practice, acid etching is performed to remove the smear layer and expose the filler and underlying surface, increasing the surface area so that the stress is distributed across the interface of the two bonded substrates (6). For this reason, phosphoric acid etching was performed to roughen the specimen surfaces.

In the present study, a silane solution was applied to specimens after surface treatments. Various studies have reported that treatment with silane improves surface wettability and promotes chemical bonding between the resin matrix and fillers (7,20). The silane molecule contains both silanol, which bonds to the silica particles of the composite, and an organofunctional group, which bonds to the methacrylate of the bonding agent (6, 23).

Brosh *et al.* (31) have mentioned three important mechanisms of the repair process for achieving an ideal bonding between the old and new composite: 1) micromechanical bonding of the treated surface, 2) chemical bonding of the organic matrix, and 3) chemical bonding of the exposed filler particles. In the present study, given that the surface treatment of the specimens was standardized, differences in micromechanical bonding cannot explain the differences in bond strengths of the tested composites; rather, the differences can be explained by differences in chemical bonding between the organic matrix and/or filler particles.

According to Baur and Ilie (2), the weak bond demonstrated by adhesive failure may be related to technical faults such as voids and porosities, the chemical structure of the adhesive system, and low

wettability of the composite. These authors also state that the low repair bond strength of high-filled composites is due to their low wettability. The present study's finding that Sonic Fill, which contains the highest amount of inorganic fillers of all the composites tested, had a lower mean bond strength when repaired with G-aenial Universal Flo in line with this assertion. The lower repair bond strength values of both Sonic Fill and Filtek Bulk Fill when repaired with G-aenial Universal Flo may also be related to differences in the monomer composition of the composite and the repair material. Filtek Bulk Fill contains high-molecular-weight resin monomers such as AUDMA and additional fragmentation monomer (AFM), which could be responsible for the low bond strength values obtained when Filtek Bulk Fill specimens were repaired with G-aenial Universal Flo. Conversely, the similarities in bond strength of the Gradia Direct Posterior, Tetric N Ceram Bulk Fill and Filtek Z250 specimens repaired with their own substrates and those repaired with G-aenial Universal Flo may be due to the similarity in the monomer structures of the composite substrates and repair material. Despite the fact that previous studies have reported that superior interfacial coupling in bonding does not require similarities in chemical composition between substrate and repair material (1), the fact that all the composites in the study had higher bond strengths when repaired with the same substrate as compared to repair with another material suggests that similarity in composition of the resin matrix and inorganic fillers in the original and repair materials is important for achieving a strong bond. Future *in vitro* studies are needed to better understand how different monomers affect repair bond strength of composites.

Conclusions

Within the limitations of this *in vitro* study, it can be concluded that substrate has a greater effect than repair material on the bond strength between an aged composite restoration and its repair. While the use of homologous repair materials generally offers more reliable results in terms of bond strength, the fact that clinicians are seldom aware of the substrate material makes it difficult to predict the success of the repair process. However, based on the data obtained from our study, the use of a universal injectable composite is not recommended for the repair of Filtek Bulk Fill and Sonic Fill restorations.

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Table 1: Chemical compositions and manufacturers of the tested composites.

Material	Resin Matrix	Fillers	wt.% /vol. %	Manufacturer
Filtek Z-250	Bis-GMA,Bis-EMA, UDMA, TEGDMA	Zirconia /silica	82/60	3M ESPE (St Paul, MN, USA)

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G-ænial Universal Flo	UDMA, Bis-MEPP, TEGDMA	Silicon dioxide, Strontium glass	69/50	GC Corp. (Tokyo, Japan)
Gradia Direct Posterior	UDMA co-monomer matrix	Silica, prepolymerized fillers, fluoroaluminosilicate glass	80/-	GC Corp. (Tokyo, Japan)
Tetric N-Ceram Bulk Fill	Modified Bis-GMA, UDMA, Bis-EMA	Barium, Ytterbium, spherical mixed oxide, prepolymer fillers	79-81/60-61	Ivoclar Vivadent AG, Schaan, Lichtenstein
Filtek™ Bulk Fill Posterior Restorative	AUDMA, UDMA, DDDMA	Silica, zircon, YbF ₃	76.5/58.4	3M ESPE (St Paul, MN, USA)
SonicFill™	Bis-GMA, TEGDMA, Modified Bis-EMA	SiO ₂ , glass, oxide	83,5/66	Kerr (Orange, CA, USA)
G-Premio Bond	Main Components: MDP, 4-MET, MEPS, Methacrylate monomer, Acetone, Water, Initiators, Silica			GC Corp. (Tokyo, Japan)
GC Ceramic Primer- II	Main Components: Silane, phosphate monomer, methacrylate, ethanol			GC Corp. (Tokyo, Japan)
Bisco Select HV® etch	35% Phosphoric acid			BISCO Inc., Schaumburg, USA

All informations were supplied by the manufacturers.

Abbreviations: BIS-GMA, bisphenol A dimethacrylate; BIS-EMA, bisphenol A polyethylene glycol diether dimethacrylate; UDMA, urethane dimethacrylate; TEGDMA, triethyleneglycol dimethacrylate; BIS-MEPP, 2,2-bis(4-methacryloxyethoxyphenyl) propane; DDDMA, 1,12-dodecane dimethacrylate; YbF₃, ytterbium trifluoride; SiO₂, silicon dioxide; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; 4-MET, 4-methacryloxyethyl trimellitic acid; MEPS, methacryloyloxalkyl thiophosphate methylmethacrylate.

Table 2: Mean±Standard Deviation of Microshear Bond Strength (MPa) of the Study Groups.

n = 10	Composites' Own Substrates	G-aenial Universal Flo
	Mean± Standard Deviation	Mean± Standard Deviation
Gradia Direct Posterior	29,25 ^a ± 6,91	33,16 ^{a,AB} ± 10,99
Tetric N-Ceram Bulk Fill	33,00 ^a ± 13,81	31,95 ^{a,AB} ± 4,87
Filtek Z-250	33,43 ^a ± 9,62	34,14 ^{a,A} ± 14,89
SonicFill™	31,52 ^a ± 7,72	21,12 ^{b,B} ± 7,95
Filtek™ Bulk Fill Posterior Restorative	41,01 ^a ± 11,59	29,73 ^{b,AB} ± 6,58

*Different lowercase letters in rows and and uppercase letters in columns indicate statistically significant differences.

Table 3: Distribution of failure modes for all experimental groups.

	FAILURE MODE					
	Composites' Own Substrate			G-Aenial Universal Flo		
	ADHESIVE	COHESIVE	MIXED	ADHESIVE	COHESIVE	MIXED
Gradia Direct Posterior	5	4	1	3	5	2
Tetric N-Ceram Bulk Fill	3	3	4	2	5	3
Filtek Z-250	3	5	2	5	5	0
SonicFill™	9	1	0	10	0	0
Filtek™ Bulk Fill Posterior Restorative	5	2	3	2	5	3

Figure 1:Flowchart of the study

