# Evaluation of Microtensile Bond Strength of Conventional and New Generation Flowable Composite Resins to Dentin With Different Adhesive Systems

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#### Abstract

**Objective:** The purpose of this study was to evaluate the effect of conventional and new generation two flowable composite resins with three different adhesive systems on bond strenght to dentin by using the microtensile bond test.

**Material and Method:** Fourty two non-carious human third molars were sectioned parallel to the occlusal plane to expose occlusal dentin. The dentin surfaces were ground with 600-grid silicon carbide (SIC) paper. Teeth were randomly divided in to six groups (n=7). Group 1-Clearfil S<sup>3</sup>Bond+ Clearfil Majesty Flow, Group 2-Clearfil SE Bond+Clearfil Majesty Flow, Group 3-Prime&Bond *NT* +Clearfil Majesty Flow, Group 4-Clearfil S<sup>3</sup>Bond+ReFil SDR Flow, Gorup 5-Clearfil SE Bond+ReFil SDR Flow, Group 6-Prime&Bond *NT*+ReFil SDR Flow. The restored teeth were serially sectioned to obtain 1mm<sup>2</sup> sticks. Each stick was submitted to the microtensile test performed at a crosshead speed of 1mm/minute. One-way ANOVA, and Tamhane's tests were used to compare the data.

**Results:** The results indicated that Clearfil SE Bond showed higher microtensile bond strength when compared to the other adhesives in Clearfil Majesty Flow group (p < 0.05). Clearfil SE Bond and Clearfil S<sup>3</sup>Bond showed similar microtensile bond strength (p > 0.05) whereas microtensile bond strength of Prime&Bond NT was significantly decreased (p < 0.05). However, in the comparision of the microtensile bond strength values of Clearfil Majesty Flow and ReFil SDR Flow groups adhesive, it was determined a statistically significant difference between two groups for the only Clearfil S<sup>3</sup>Bond (p < 0.05).

**Conclusion:** In the experimental conditions of this study it was seen that adhesive systems may have different effects on the bond strenght to dentine tissue.

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Keywords: Flowable composites, adhesive systems, microtensile bond strength, dentin.

## Geleneksel ve Yeni Nesil Akışkan Kompozit Rezinlerin Farklı Adeziv Sistemler ile Dentine Mikrogerilim Bağlanma Dayanımlarının Değerlendirilmesi

# Özet

**Amaç:** Bu çalışmanın amacı, geleneksel ve yeni nesil iki akışkan kompozit rezinin üç farklı adeziv sistem ile dentine bağlanma dayanımı üzerine etkisini mikrogerilim bağlanma dayanımı test yöntemi ile değerlendirmektir.

**Gereç ve Yöntem:** Kırk iki adet çürüksüz insan üçüncü büyük azı dişin okluzal minesi kesilerek okluzal dentin açığa çıkarıldı. Dentin yüzeyleri, 600-grid silikon karbid kâğıtla (SIC) zımparalandı. Dişlerden rastgele altı grup oluşturuldu (n=7). Grup 1-Clearfil S<sup>3</sup>Bond+ Clearfil Majesty Flow, Grup 2-Clearfil SE Bond+Clearfil Majesty Flow, Grup 3-Prime&Bond *NT* +Clearfil Majesty Flow, Grup 4-Clearfil S<sup>3</sup>Bond+ReFil SDR Flow, Grup 5-Clearfil SE Bond+ReFil SDR Flow, Grup 6-Prime&Bond *NT*+ReFil SDR Flow. Restore edilen dişler, 1mm<sup>2</sup>'lik çubuklar elde edilecek şekilde kesildi. Her bir çubuk, dakikada 1 mm hızla hareket eden cihazla mikrogerilme testine tabi tutuldu. Sonuçların karşılaştırılmasında tek-yönlü ANOVA, t ve Tamhane testleri kullanıldı.

**Bulgular:** Clearfil Majesty Flow gruplarında Clearfil SE Bond daha yüksek mikrogerilim bağlanma dayanımı göstermiştir (p< 0.05). ReFil SDR Flow grubunda ise, Clearfil SE Bond ve Clearfil S<sup>3</sup>Bond'un mikrogerilim bağlanma dayanım değerlerinin, Prime&Bond NT'ye göre yüksek olduğu bulunurken (p<0.05), kendi aralarında bir fark bulunamadığı belirlenmiştir (p>0.05). Bununla birlikte, Clearfil Majesty Flow ve ReFil SDR Flow gruplarındaki adezivlerin mikrogerilim bağlanma dayanım değerlerinin karşılaştırılmasında yalnızca Clearfil S<sup>3</sup>Bond için iki grup arasında istatistiksel olarak anlamlı bir farklılık gösterdiği tespit edilmiştir (p<0.05).

**Sonuç:** Bu in vitro çalışma koşullarında, adezivlerin dentine bağlanma dayanımı üzerinde farklı etkilere neden olabileceği görüldü.

Anahtar Kelimeler: Akışkan kompozitler, adeziv sistemler, mikrogerilim bağlanma dayanımı, dentin.

# Introduction

In today's dentistry; increasing emphasis on aesthetic has brought a lot of research which is based on composite resins and adhesives systems.

The importance of the bonding between hard tissue of teeth with dental materials is great at the success of composite resins which has a wide application area in the posterior and anterior region. As opposed to the bonding to the enamel the bonding to the dentine continues to cause problem for dentists due to its tubular structure and the formation of smear layer during cavity preparation.<sup>1,2</sup>

Composite resins are being bonded to the dental tissues micromechanically by using new generation of adhesive systems with two different techniques; the total-etch and self-etch.<sup>3,4</sup> Producers tended to simplify the three-step total-etch adhesives, and have developed a two-step total-etch system due to difficulty of moisture control and surplus of application steps.<sup>5,6</sup> Self-etch adhesives have been developed to eliminate operator errors which occur during the usage of the total-etch adhesives by reducing the number of phases of the adhesive aplication and eliminating technique sensitivity. The mixing step is eliminated with the self-etch adhesives which have been developed in recent years, resin monomer, photoinitiator, tertiaryamine accelerator was collected in a singe bottle.<sup>7,8</sup>

The edge compliance has a critical importance for composite resin restoration to continue its performance for a long time. Polymerization shrinkage of the resin restorations can cause accumulation of stress in the bonding surfaces and separation between the tooth surfaces and adhesives. Gaps that are formed between the cavity walls and restoration material, can cause postoperative problems such as sensitivity, pulp damage and recurrent caries.<sup>9,10</sup> The application of flowable composite resins as a thin layer to cavity flour, is one of the proposed method to provide a full sealing between the cavity wall and composite restorations during the polymerization shrinkage.<sup>11</sup> In addition, the usage of flowable composite resin under the composite resin, has been determined to significantly increase the bonding strength.<sup>12,13</sup>

Researchers have used different test methods to measure the bonding strength of restoration materials and adhesive systems. Sano et al.<sup>11</sup> have introduced the microtensile bonding strength test for the first time in 1994. Stress can be measured in the 0.25 and 1mm<sup>2</sup> sample size with microtensile bonding strength test. In addition, higher bonding strength and a very low variation coefficient are measured with conventional shear and tensile tests. Thus the more reliable results are expected to occur. Many studies indicated that the bond strength could be measured in different regions and depths of the tooth at and also multiple samples could be tested from the same tooth.<sup>11,14-16</sup>

The purpose of this study was to evaluate the effect of conventional and new generation two flowable composite resins with three different adhesive systems on bond strenght to dentin by using the microtensile bond test.

#### **Materials and Methods**

Fourty two non-carious human third molars extracted with periodontal reasons were used in this study. Teeth were stored in distilled water until to be tested after cleaning tissue debris on the teeth. Occlusal enamel was cut to be perpendicular to the long axis of tooth under water cooling with diamond bur by operating at low speed. 600 grid silicon carbide paper (SIC) was applied to the surface for one minute to obtain homogeneous smear layer on dentin surfaces that was uncovered. Teeth were randomly divided in to six groups (n=7). The adhesive systems and flowable composites which is used in the research are shown in Table 1.

Group 1: After Clearfil S<sup>3</sup>Bond which is one-step self-etch adhesive is applied in accordance with manufacturer recommendations to dentin surfaces, it was polymerized with LED (HS LED 1500, Henry Schein Inc, USA) light-curing unit.

Materials	Туре	Manufacturer		
Clearfil S3	one-step self-etch adhesive	Kuraray Medical Inc., Tokyo, Japan		
Clearfil SE Bond	two-step self-etch adhesive	Kuraray Medical Inc., Tokyo, Japan		
Prime&Bond NT	two-step total-etch adhesive	Dentsply, Konstanz, Germany		
Clearfil Majesty Flow	flowable composite	Kuraray Medical Inc., Tokyo, Japan		
ReFil SDR Flow	flowable composite	Dentsply, Konstanz, Germany		

Table 1. The adhesive systems and flowable composites which is used in the research

Group 2: After Clearfil SE Bond which is two-step self-etch adhesive is applied in accordance with manufacturer recommendations to dentin surfaces, it was polymerized with LED light-curing unit. Group 3: After 34% phosphoric acid gel applying to the occlusal dentinal surface, Prime & Bond NT which is the total-etch adhesive is applied in accordance with manufacturer recommendations to dentin surfaces, it was polymerized with LED light-curing unit.

Clearfil Majesty Flow as flowable composite was light cured after being placed with a thickness of 2 mm to the dentin surface which was applied adhesive in all three in the group.

Group 4: After Clearfil S<sup>3</sup>Bond which is one-step self-etch adhesive is applied in accordance with manufacturer recommendations to dentin surfaces, it was polymerized with LED light-curing unit. Group 5: After Clearfil SE Bond which is two-step self-etch adhesive is applied in accordance with manufacturer recommendations to dentin surfaces, it was polymerized with LED light-curing unit. Group 6: After 34% phosphoric acid gel applying to the occlusal dentinal surface, Prime & Bond NT which is the total-etch adhesive is applied in accordance with manufacturer recommendations to dentin surface is applied in accordance with manufacturer recommendations to dentin surface is applied in accordance with manufacturer recommendations to dentin surface is applied in accordance with manufacturer recommendations to dentin surface is applied in accordance with manufacturer recommendations to dentin surface is applied in accordance with manufacturer recommendations to dentin surface is applied in accordance with manufacturer recommendations to dentin surface is applied in accordance with manufacturer recommendations to dentin surface is applied in accordance with manufacturer recommendations to dentin surface is applied in accordance with manufacturer recommendations to dentin surfaces.

ReFil SDR Flow as flowable composite was light cured after being placed with a thickness of 2 mm to the dentin surface which was applied adhesive in all three in the group.

Then the teeth were kept in the incubator for 24 hours in the saline solution. After each storage period, the bonded teeth were vertically sectioned into serial slabs and further into beams with cross-sectional areas of approximately 1 mm<sup>2</sup>. Totally 30 rod was obtained for each group. Specimens were attached to microtensile testing apparatus (Micro Tensile Tester, Bisco, USA) with the cyanoacrylate adhesive (Zapit, Dental Ventures of America, Corona, CA, USA) and stressed to failure in tension at a crosshead speed of 1 mm/min. The load recorded in Newtons was retrieved in MPa.

Normal distribution assumption of conformity of the data were analyzed with the Kolmogorov-Smirnow test, it's homogeneity was examined by Levene test. Statistical differences were examined using ANOVA, Independent Samples test and Tamhane test at a significance level of 5% with SPSS 11.0 for Windows (SPSS Inc., USA)

#### Results

Microtensile bond strength values of the experimental group are shown in Table 2.

As a result of evaluation of the data that is obtained, a statistically significant difference was observed in the microtensile bond strength values of adhesives that is applied Clearfil Majesty Flow (F=7.825; p=0,001) (Figure 1). In multiple comparison of microtensile bond strength values, bond strength values of Clearfil SE Bond were determined to be statistically significantly higher (p<0.05).



Figure 1. Mean microtensile bond strength values of adhesives that is applied Clearfil Majesty

When microtensile bond strength values of adhesive that is applied ReFil SDR Flow are examined to of adhesive it was observed a statistically significant difference. (F=13,960; p=0,000) (Figure 2). In multiple comparison of microtensile bond strength values, bond strength values of Clearfil SE Bond and Clearfil S<sup>3</sup>Bond was observed statistically significant higher than Prime & Bond NT (p <0.05). However, no statistically significant difference was shown in the bond strength values between the Clearfil SE Bond and Clearfil S<sup>3</sup>Bond (p>0.05).



Figure 2. Mean microtensile bond strength values of adhesives that is applied ReFil SDR Flow

In the comparison microtensile bond strength values of the adhesives that is applied Clearfil SE Bond and ReFil SDR Flow was determined a statistically significant difference between two groups for Clearfil S<sup>3</sup>Bond (t=-4,909; p=0,000); there is no statistically significant difference for Clearfil SE Bond and Prime&Bond NT (p>0.05).

	Clearfil Majesty Flow				<b>ReFil SDR Flow</b>					
	Ν	Mean(MPa)	Standard	Min.	Max	Ν	Mean(MPa)	Standard devi-	Min.	Max
			deviation					ation		
Clearfil S3	30	22,116	8,2884	8,1	41,4	30	34,556	11,1343	11,4	53,6
Clearfil SE Bond	30	30,150	12,4529	10,5	53,2	30	38,060	19,7934	7,7	83,7
Prime&Bond NT	30	20,716	8,6193	4,1	37,4	30	19,373	10,9710	3,7	50,0

#### Table 2. Microtensile bond strength values of the experimental group

## Discussion

Clinical trials are the most appropriate way to evaluate the effectiveness of restorative materials. However, clinical research of the developing adhesive systems or resins is very difficult technically and ethically. Therefore, laboratory studies are often preferred in dentistry.<sup>17</sup> One of the commonly used methods are microtensile bond strength tests for the evaluation of clinical performance of resin-adhesive systems. In addition to conventional tensile testing methods, the micro test methods which is using 1 mm<sup>2</sup> surface area are also used to determine the bond strength between the dental tissues and restorative materials. It can be stated that the non-uniform stress distribution at the interface of the dental tissue and an adhesive system can be eliminated through the use of samples which have smaller surface areas in test methods. <sup>11,17,18</sup> In this study, microtensile bond strength test method was used by obtaining bars that have an average of 1 mm<sup>2</sup> bonding surface from samples, as in the study Sano et al<sup>11</sup> and Phrukkanon et al<sup>19</sup>.

Clearfil SE Bond which we used as a two-step self-etch adhesive, has acidic primer in middle strength (pH=1.9). This adhesive system showed high bond strength to normal dentin in many studies.<sup>20-22</sup> Clearfil SE Bond contains filler particles that are thought to increase the adhesive's tensile capacity against shrinkage stress (silicon dioxide).<sup>23</sup> It was declared that theoretically, simultaneously of the emerging of collagen fibrils and the occurring of the monomer infiltration were sufficient for micromechanical bonding. Also, carboxyl and phosphate groups of Clearfil SE Bond monomers may be chemically bonded to the residual hydroxyapatite. In this way, it is claimed to exhibit high bond strengths.<sup>20,24,25</sup>

Clearfil S<sup>3</sup>Bond that is used as one-step self-etch adhesive, has acidic primer in low strength (pH= 2.7). <sup>26</sup> This type of adhesive systems allow to remain hydroxyapatite around collagen fibrils by demineralizing dentin fairly shallow, and it creates a superficial hybrid layer. These adhesives behave like a permeable membrane and absorb a significantly water after polymerization due to its hydrophilic nature. Therefore, it is claimed to showed lower bond strength values from two-step self-etch adhesives. <sup>25,27,28</sup>

In the two-step total-etch system is the first step in creating the acid application, the second step primer and adhesive Prime & Bond NT, which constitutes one bottle united version. Although the bonding mechanism of this type of adhesive systems are same as three-step total-etch systems, in many studies, it is claimed that the application of the adhesive and the primer in one-step may reduce the hybridization. The fact that two-step "etch-and-rinse" adhesive systems are more sensitive to water and oxygen contamination, may lead to the incomplete polymerization for adhesive resin and lower bond strengths.<sup>6,29</sup>

This type of adhesive before depolymerization, nano-filler particles can form clusters which are large enough to prevent the infiltration come together to inter-fibrillar space of the hybrid layer of adhesive. In addition, the aqueous monomer which is the main component of the adhesive can prevent the infiltration of these particles as previously infiltrated to demineralized intertubuler matrix . This situation affects bonding negatively. <sup>30</sup> It is reported that may have reduced the bond strength values as a result of lack of access to the adhesive to these regions and having the greater demineralization depth which is occured in dentin.<sup>5</sup>

We believe that all this obtained data in our study explains the different bond strength results.

It has been reported in many studies to reduce microleakage and seen in the restoration edge spacing formation by preventing the polymerization shrinkage, as a linear usage under composite restorations of flowable composite resins having a low elasticity coefficient.<sup>31,32</sup> Not only the usage of flowable composite under composite resins play a role in compensating stresses caused by polymerization shrinkage, but also it has been reported that stress absorber is doing against the accumulated tension and compressive stress in this region task during chewing forces. <sup>33,34</sup> A new generation of flowable composite which has more filling rate, is claimed that find wide application due to the increased mechanical properties. <sup>22,35</sup> Although higher bond was observed in the new generation of flowable composite group, a statistically significant difference was detected only for Clearfil S<sup>3</sup>Bond, when bond strength of the flowable composite resins is compared.

Nowadays, while many new developments occurs at the system of adhesive dentistry; following these developments on a regular basis and selecting suitable materials of dentist will improve the clinical success of restorative treatment. In this context, we believe that our study results will contribute to the different studies on the subject.

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