

## Evaluation of Microtensile Bond Strength of Different Adhesive Systems to Dentin

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### Abstract

**Objective:** The purpose of this study was to examine the bond strengths of three adhesive systems (Prime&Bond NT, Clearfil S3 Bond and Silorane System Adhesive) to dentin by microtensile testing.

**Materials and Methods:** Fifteen non-carious human molars were selected for the study. Occlusal surfaces of the teeth were removed and flattened until dentin was exposed. The teeth were randomly divided into three groups of five teeth each. Group I was treated with total etch adhesive Prime&Bond NT (Dentsply), group II with self-etch adhesive Clearfil S3 Bond (Kuraray), and group III was treated with self-etch adhesive Silorane System Adhesive (3M ESPE). Polyester matrix was placed and according to the manufacturers' instructions bonding agents were applied. Resin composite build-ups were constructed on the bonded surfaces in increments and light cured according to the manufacturers' instructions. After storage in distilled water at 37° C for 24 hours, the specimens were longitudinally sectioned perpendicularly to the adhesive interface to obtain sticks (1mm<sup>2</sup>) which were tested under a tensile load of 0.5 mm/minute. The bond strength data were analyzed with one-way ANOVA, Tukey comparisons test and Student-Newman Keuls test at p< 0.05 level.

**Results:** The highest microtensile bond strength values were obtained with the Prime&Bond NT (Dentsply) group. No significant difference was observed among Silorane System Adhesive (3M ESPE) and Clearfil S3 Bond (Kuraray) groups (p>0.05).

**Conclusion:** Total-etch system showed significantly (p<0.05) higher dentin bond strength values than self-etching systems.

### Farklı Adeziv Sistemlerin Dentindeki Mikrogerilim Bağlanma Dayanımlarının İncelenmesi

#### Özet

**Amaç:** Bu çalışmanın amacı üç farklı adeziv sistemin (Prime&Bond NT, Clearfil S3 Bond ve Silorane System Adhesive) dentindeki mikrogerilim bağlanma dayanımlarını değerlendirmektir.

**Gereç ve Yöntem:** Bu çalışmada on beş adet çürüksüz insan molar dişi kullanılmıştır. Dişlerin okluzal yüzeyleri kaldırılmış ve dentinde düz yüzeyler elde edilmiştir. Dişler her grupta beşer diş olacak şekilde rastgele üç gruba ayrılmıştır. Örneklere 1.grupta total etch adeziv Prime&Bond NT (Dentsply), 2.grupta self etch adeziv Clearfil S3 Bond (Kuraray), ve 3.grupta self etch adeziv Silorane System Adhesive (3M ESPE) uygulanmıştır. Dentin yüzeylerine polyester matriks yerleştirilmiş ve üretici firma önerilerine göre bonding ajanlar ve rezin kompozit materyal uygulanmıştır. Hazırlanan örnekler distile su içinde 37°C'de 24 saat bekletilmiş, daha sonra longitudinal ve perpendikular olarak, bağlantı yüzeyi 1mm<sup>2</sup> olan çubuklar şeklinde kesilmiştir. Elde edilen çubuklara 0.5 mm/dakika'lık çekme kuvveti uygulanmış ve sonuçlar one-way ANOVA, Turkey comparisons test ve Student-Newman Keuls test ile analiz edilmiştir (p<0.05).

**Bulgular:** En yüksek mikrogerilim bağlanma dayanımı değeri Prime&Bond NT (Dentsply) grubunda elde edilmiştir. Silorane System Adhesive (3M ESPE) ve Clearfil S3 Bond (Kuraray) grupları arasında anlamlı fark bulunmamıştır (p>0.05).

**Sonuç:** Total-etch adeziv sistemin dentine bağlantı kuvveti değerleri, self-etch sistemden belirgin olarak daha yüksektir (p<0.05).

#### Introduction

One of the major trends in today's dentistry is tooth colored materials. This observation is attributed to higher esthetic demands of the patients.<sup>1</sup> Therefore resin composites are used extensively in tooth restoration. These materials have the following advantages; similar color of a real tooth, good physical properties and enabled to be used in conservative cavity preparations.<sup>2</sup>

At present, dental composites are expected to have optical and mechanical properties comparable to those of tooth enamel and dentin and provide a service life of 10 years or more.<sup>3</sup> For a successful resin composite restoration, the adhesive resin forming a strong and stable bond between the resin composite and the dentin is required.<sup>2</sup> However in the mouth, the interface between restoration and tooth is exposed to diverse forces that act simultaneously.<sup>3</sup> Also when placing direct resin-

based posterior composite restorations, polymerization shrinkage occurs which is one of the dental clinician's primary concerns.<sup>4</sup> Already during setting of composite, polymerization shrinkage stresses occur within seconds of the polymerization process beginning<sup>2</sup> and resin shrinkage puts stress on the bond, eventually pulling it away from the cavity wall,<sup>5,6</sup> which may form a gap.<sup>5</sup> Gaps which contain bacteria, fluids, molecules, and ions may cause micro-leakage, staining, secondary caries and post-operative sensitivity. Also it may initiate micro-cracking of the restorative material (cohesive failure) that causes the bond to fail in a short period of time.<sup>2</sup>

High bond strength and a complete marginal seal at the resin composite-dentin interface are required for clinically successful restorations. Dentin bond strength and marginal integrity are

used by clinicians as important selection criteria for dental adhesives.<sup>7</sup>

The aim of this study was to examine the bond strengths (microTBS) of three different adhesive systems, Prime&Bond NT (Dentsply), Clearfil S3 Bond (Kuraray), and Silorane System Adhesive (3M ESPE) to dentin surface by microtensile testing.

### Materials and Methods

Fifteen extracted, caries free human molars were used in this study. These teeth were stored in distilled water after extraction and cleaned of debris and remnants of the periodontal ligament with scalers. The teeth were individually fixed to a sectioning block using acrylic resin, they were embedded until cervical line. The occlusal surfaces of these teeth were removed and flattened until dentin was exposed. The selected teeth were randomly divided into three groups of five teeth each.

Polyester matrix was placed and each adhesive system [total etch adhesive Prime&Bond NT (Dentsply), self-etch adhesive Clearfil S3 Bond (Kuraray), self-etch adhesive Silorane System Adhesive (3M ESPE)] was applied according to the manufacturer's instructions, following application of the adhesives same manufacturer's resin

composites were built on the bonding surface, sequential application of 2 mm thick layers of material (up to 4 mm) was done and each specimen was cured according to the manufacturers' instructions. The completed specimens were stored in distilled water at 37° C for 24 hours. After incubation, each tooth was serially sectioned into rectangular beams with mean cross-sectional area of 1 mm<sup>2</sup> using a slow speed, water-cooled diamond saw (Medkom Microcut, Turkey). The sections were cut parallel to the long axis and perpendicular to the adhesive interface. Due to the occurring fractures of specimens in the microcut machine, some teeth were excluded in terms of equalizing the number of samples. A total of twenty dentin-composite specimens were obtained from each group. The specimens were then individually attached to a testing apparatus with Pattex (Henkel), which was placed in a universal testing machine (Microtensile Tester, USA) for tensile testing at a cross-head speed of 0.5 mm/min. Statistical analysis of tensile bond strengths and the comparisons between the groups were performed using one -way ANOVA, Turkey comparisons test and Student-Newman Keuls test. The components and composite resins of each bonding agents used in the study are showed in Table I.

**Table I. Composition of bonding agents and composite resins used.**

Group	Bonding Agent	Composition of bonding agent	Composite Resin
I	Prime&Bond NT (Dentsply DeTrey GmbH, Konstanz, GERMANY)	PENTA, R5-62-1, acetone, camphoroquinone, UDMA resin, cetylamine hydrofluoride, initiators, stabilizer, acetone, nanofillers and two proprietary elastomeric resins	Quixfil (Dentsply DeTrey GmbH, Konstanz, GERMANY)
II	Clearfil S3 Bond (Kuraray Medical inc., Tokyo, JAPAN)	10-MDP, Bis-GMA, HEMA,DMA, camphoroquinone, ethanol, water, silanated colloidal silica	Clearfil Majesty Posterior (Kuraray Medical inc., Tokyo, JAPAN)
III	Silorane System Adhesive (3M ESPE, Dublin, IRELAND)	<i>Primer:</i> phosphorylated methacrylates, BisGMA, HEMA, water, ethanol, camphoroquinone, silane treated silica filler <i>Adhesive:</i> hydrophobic bifunctional monomer, silane treated silica filler camphoroquinone	Filtek Silorane (3M ESPE, Dublin, IRELAND)

### Results

Table II presents mean microtensile bond strengths and standard deviations of each group.

Mean bond strength in group I (34.96±16.36) is significantly higher than the mean bond strength in

group II (25.21±7.79) and group III (20.39±8.95) (p<0.05). However there is no statistically significant difference in mean values between group II and group III (p>0.05).

**Table II. Mean microtensile bond strengths of each group**

Group	n	Bond Strength (MPa ± S.D.)
I	20	34,96 (16,36) <sup>a</sup>
II	20	25,21 (7,79) <sup>b</sup>
III	20	20,39 (8,95) <sup>b</sup>

### Discussion

It has been recommended that adhesives be tested using a resin-based composite recommended by the same manufacturer to avoid possible adverse chemical interactions.<sup>8,9</sup> So in the current study, the microtensile bond strength of three bonding agents to dentin surface was evaluated with the posterior composite resins of the same manufacturers'.

In our study due to the occurring fractures of specimens in the microcut machine, some teeth were excluded. Okuda et al.<sup>10</sup> stated that when fracture occurs during the preparation of samples of 50% of a tooth, the other 50% does not reflect the real bond strength. As a result, we have excluded the fractured samples in terms of equalizing the number of samples.

Measured bond strengths values depend on numerous factors including the bonding system used<sup>11-15</sup>. The bonding agent which we used in group I, is a total-etch adhesive Prime&Bond NT. On the other hand the adhesives used in group II (Clearfil S3 Bond) and group III (Silorane System Adhesive) are self-etch adhesive systems. The results that we obtained in the study display that group I had the highest mean bond strength. The smear layer and superficial dentin are demineralized and the collagen fibers are exposed with the application of 37% phosphoric acid. The exposed collagen may provide reactive groups that can chemically interact with bonding primers.<sup>16,17</sup> Also nanoparticles of Prime&Bond NT, may provide to establish a thicker more uniform resin film thickness that stabilizes the hybrid layer.<sup>17, 18</sup> These factors may be the reason that group I had the highest bond strength in our study.

The bond strength difference between self-etch and total-etch systems in our study is in agreement with those of Neelima et al.<sup>16</sup>, Soares et al.<sup>20</sup> and Purk et al.<sup>21</sup> Also Brackett et al.<sup>22</sup> reported that single phase self-etching resins do not produce as a good bond with enamel as etch-and-rinse adhesives and Bowen and Cobb<sup>23</sup> reported that etching dentin with acidic solutions also increased bond strengths to dentin. However in the study of Guzmán-Armstrong et al.<sup>7</sup> a self-etch adhesive system Clearfil SE Bond (Kuraray) showed higher bond strength value than total-etch systems.

Yesilyurt and Bulucu<sup>24</sup> represent in their study that comparisons among total-etch and self-etch systems except Clearfil SE Bond (self-etch adhesive, Kuraray), total-etch systems bonding strength was found statistically higher than other self-etch adhesive systems.

In the present study we used one-step self-etch adhesive (Clearfil S3 Bond) in group II and two-step self-etch adhesive (Silorane Adhesive System) in group III. We reported that there is no statistically significant difference in mean values between group II and group III ( $p>0,05$ ). For the self-etch adhesives, smear layer removal is dependent on the pH of the primer used.<sup>5, 25-27</sup> Both self-etch adhesives used in our study have a pH around 2.7. It is thought that these adhesives had similar bond strength values because of similar effects on the smear layer.

One of the factors effecting the microtensile bond strength might be the polymerization shrinkage of composites tested.<sup>28</sup> According to the manufacturers of the tested composites', volumetric shrinkage were ranked as follows; Group I (Quixfil), 1.7 %; Group II (Clearfil Majesty Posterior), 1.5 % and Group III (Filtek Silorane), 0.66 %. On the contrary, in our study polymerization shrinkage could not be the main factor acting on the microtensile bond strength. As Group I had the highest bond strength despite the high percentage of volumetric shrinkage.

In the Buonocore Memorial Lecture of Meerbeek et al.<sup>5</sup> notified that two-step etch-and-rinse and two-step self-etch bonded significantly much better to dentin than one-step self-etch adhesives. Our study results are only in agreement with two-step etch-and-rinse bonded significantly better to dentin than one-step self-etch adhesives.

The use of microtensile bond strength test to evaluate bond strength of adhesive systems has been widely accepted.<sup>10</sup> Bond strength values can be used for comparing the effectiveness of the adhesives however they cannot present directly what might happen under clinical situations.<sup>18</sup>

Our study concluded that, microtensile bond strength of two-step total-etch system is better than one-step and two-step self-etch systems.

Additionally, one-step self-etch has a greater mean value than two-step self-etch adhesive. But the difference is not statistically significant. To investigate the in vivo effects of the materials further clinical studies should be undertaken.

### Conclusion

Despite total-etch adhesive systems have a disadvantage of requiring multiple application steps; based on the study results total-etch adhesives have higher bond strength values than the self-etch adhesives.

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