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Evaluation of Shear Bond Strength of Universal Adhesives to Dentin: in Vitro Study

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ABSTRACT

Aim: Universal adhesives are widely used in restorative dentistry. The aim of this study was to evaluate the shear bond strength (SBS) of universal adhesives to dentin in different application modes.
Materials and Methods: The study utilized seven universal adhesives and one total-etch adhesive. The sixty-four extracted intact human molars used in the study were embedded in acrylic blocks to the enamel-cement junction. The occlusal surface was removed to expose the dentin surface. After applying self-etch and total-etch adhesives to the dentin, flowable composite (Clearfil Majesty Flow, Kuraray) was placed in a transparent mold (2.38 mm diameter and 2 mm height) and polymerized. After thermal aging (1000 cycles), a SBS test has been carried out on a universal testing machine. The fracture types formed on the surface of the teeth were examined under a microscope. Data were analyzed by two-way analysis of variance (ANOVA) test ($p<0.05$).
Results: SBS values of universal adhesives on dentin showed statistically significant differences according to adhesive and application mode ($p<0.05$). Total-etch application produced statistically higher SBS than self-etch application ($p<0.05$). The two-step universal adhesive (G2 Bond Universal) had the highest SBS in both the self-etch and total-etch application modes ($p<0.05$). When the bond surfaces of the universal adhesives were examined, fractures occurred most frequently in the adhesive type.
Conclusion: The use of universal adhesives in the total-etch mode results in greater bond strength to dentin tissue. Universal adhesive type influences bond strength.

Üniversal Adezivlerin Dentine Makaslama Bağ Dayanımının İncelenmesi: İn Vitro Çalışma

Makale Bilgisi

Makale Geçmişi

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Anahtar Kelimeler:
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Makaslama dayanımı.

ÖZET

Amaç: Üniversal adezivler restoratif diş hekimliğinde yaygın olarak kullanılmaktadır. Bu çalışmanın amacı, üniversal adezivlerin dentine makaslama bağlanma dayanımını farklı uygulama modlarında değerlendirmektir.
Gereç ve Yöntemler: Çalışmada 7 adet üniversal ve 1 adet total-etch adeziv kullanıldı. Çalışmada kullanılacak 64 adet çekilmiş sağlam insan molar dişi mine-sement sınırına kadar akrilik bloklara gömüldü. Dişler, dentin yüzeyini açığa çıkaracak şekilde okluzal yüzeyden aşındırıldı. Dentin dokusu üzerine adezivler self-etch ve total-etch modda uygulandıktan sonra şeffaf bir kalıp (2,38 mm çap ve 2 mm yükseklik) içerisinde akıcı kompozit (Clearfil Majesty Flow, Kuraray) konularak polimerize edildi. Termal yaşlandırma (1000 siklus) işleminden sonra üniversal test cihazında makaslama bağ dayanım testi yapıldı. Dişlerin yüzeyinde oluşan kırık tipleri mikroskop altında incelendi. Çalışmada veriler iki yönlü varyans analiz (ANOVA) testi kullanılarak değerlendirildi ($p<0,05$).
Bulgular: Üniversal adezivlerin dentin üzerindeki makaslama bağ dayanım değerleri adeziv ve uygulama modduna göre istatiksel anlamlı farklılıklar gösterdi ($p<0,05$). Total-etch uygulama self-etch uygulamadan istatiksel olarak daha fazla makaslama bağ dayanım gücü oluşturdu ($p<0,05$). İki aşamalı üniversal adeziv (G2 Bond Universal) self-etch ve total-etch uygulama modunda en fazla makaslama bağ dayanımı gösterdi ($p<0,05$). Üniversal adezivlerin bağlantı yüzeyleri incelendiğinde en fazla adeziv tipte kırık görüldü.
Sonuç: Üniversal adezivlerin total-etch modda kullanılması dentin dokusu üzerinde daha fazla bağlantı gücü oluşturmaktadır. Üniversal adeziv tipi bağlantı gücü üzerinde etkilidir.

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INTRODUCTION

Today, restorative dentistry tends to focus on the complete removal of carious tissue while avoiding the removal of healthy tooth structure to improve mechanical retention. Adhesion is the cornerstone of modern restorative dentistry.[1] Adhesive systems are used to provide a long-lasting bond between the tooth and resin-based materials.[2] These systems act as a mediating material in the restoration, increasing retention, marginal sealing and tooth-restoration interface resistance.[3]

Adhesive systems can be divided into two main categories based on the method of application to the tooth surface: total-etch and self-etch.[4,5] Acidifying enamel partially demineralizes hydroxyapatite, thus exposing enamel prisms that facilitate micro-attachment. For dentin, superficial hydroxyapatite is dissolved and the smear layer and smear plugs are removed after rinsing. Phosphoric acid (35-37%) is often used as an etching agent for total etch systems [2] and these systems involve the prior application of phosphoric acid.[5,6] Therefore, total-etch adhesives can be applied in three steps (etching, primer and adhesive) or in two steps (primer and adhesive combined in one material).[7]

Although total etch adhesives are still the gold standard, the current trend is towards the development of simplified self-etch materials.[7] Self-etch adhesives can be two or one-step depending on the primer and adhesive resin.[5,8] The application is simplified by combining all components (acid, primer and adhesive resin) in a single material.[9,10] Functional and base monomers, solvents and initiators, and optional fillers are included in these "universal" adhesives. It is difficult to stabilize all these different components in a single bottle while maintaining their bonding ability.[2] These systems are designed by the manufacturers to allow the dentist to decide which bonding technique to use in relation to the choice of the bonding procedure and the number of steps.[7] The term “universal” indicates that these adhesives can be used in all modes of application, such as self etch, total etch and selective etch (selective enamel etching).[2]

Despite the advances made in adhesive technology, there are still unresolved issues regarding the durability of the adhesive interface.[11] It is still difficult to achieve a sealed connection between the resin and the dentin substrate, and it is doubtful that ideal interdiffusion of the adhesive system within the collagen scaffold can be achieved.[12,13] Although an adequate resin-dentin connection is usually achieved immediately, a decrease in the strength of the bond occurs over time.[14] As a result, the actual mechanisms of failure are not fully understood. In this sense, it is necessary to follow the recommendations of each company; otherwise, adhesive degradation may occur.[15]

Adhesion performance depends on many factors and may vary depending on the adhesive system used. Universal adhesives have the advantages of being simple, fast to apply and requiring less precision in technique. The stability and durability of the dentin-adhesive interface created by universal adhesive systems continues to be questioned. One of the major concerns with these systems is the increase in microleakage after aging, leading to limited bond strength. The lack of a standardized protocol to ensure stable and optimal adhesion of universal adhesive systems to dentin is a major problem. Furthermore, improvement of bond strength can be achieved by a multitude of methods.[13]

Despite the improved materials, it is still an important question whether clinicians should consider using these new adhesives with total-etch rather than self-etch.[7] Which adhesive protocol is optimal for universal adhesives cannot be answered definitively

based on the available clinical evidence and the short follow-up times evaluated.[13]

Furthermore, manufacturers use different combinations of components in universal adhesives; therefore, comparisons of available adhesives are difficult. Furthermore, studies comparing the SBS of different commercially available universal adhesives are scarce in the literature.[2]

The aim of this study was to contribute to the literature by investigating the SBS of universal adhesives to dentin in self-etch and total-etch mode.

MATERIALS AND METHODS

Ethical approval fort his study was obtained from Gülhane University of Health Sciences Pharmaceutical and Medical Received by off-device ethics committee (2024/284).

The SBS of a composite (Clearfil Majesty Flow, Kuraray) bonded to human dentin using seven different universal adhesive systems will be tested. A total-etch adhesive system (Prime&Bond NT, Densply) was used as the control group (Table 1). Sample size was determined using G-Power analysis software with a large effect size, 0.05 error level, and 80% power.

Table1: Contents of the adhesive systems used

Adeziv Sistemler	İçindekiler	pH	Lot No
G2-Bond Universal (GC, Tokyo, Japan)	Two-step Universal Adhesive Primer: 4-MET, 10-MDP, 10-MDTP, dimethacrylate monomer, acetone, water, initiators, fillers Adhesive: dimethacrylate monomer, Bis-GMA, filler, photoinitiator	1.5	1110251
Optibond Universal (Kerr, Schaumburg, USA)	GPDM, glycerol dimethacrylate, HEMA, acetone, ethanol	2.3	9448958
Bond Force II (Tokuyama, Japan)	Phosphoric acid monomer, Bis-GMA, TEGDMA, 2-Hydroxyethyl methacrylate HEMA, Camphorquinone, alcohol and purified water.	2.8	138E52
Prime&Bond NT (Dentsply Sirona, Germany)	Urathane dimetacrylate (UDMA), trimethacrylate, phosphoric acid modified acrylate resin (PENTA), highly dispersed silicon dioxide, camphorquinone, ethyl-4(dimethylamino) benzoate (photoaccelerator), butylated hydroxy toluene (BHT), cetylamine hydrofluoride, acetone	2.1	2205000548
G-Premio Bond Universal (GC, Tokyo, Japan)	MDP, 4-MET, MEPS, methacrylate monomer, acetone, water, initiator, silica	2.1	2202021
Clearfil Tri-S Bond Universal (Kuraray Noritake, Tokyo, Japan)	10-MDP, Bis-GMA, HEMA, colloidal silica, silane, sodium fluoride, camphoquinone, ethanol, water	2.5	1P0065
Prime&Bond Universal (Dentsply Sirona, Germany)	10-MDP, bisacrylamide monomers, PENTA, isopropanol, water, initiator, stabilizer	2.5	2112000750
Gluma Bond Universal (Kulzer, Hanau, Germany)	UDMA, MDP, 4-META, HEMA, acetone, water, photo initiators, stabilizers	1.5	M010058

Preparation of Samples

Sixty-four extracted caries-free wisdom teeth were used in the study. The teeth were kept in +4 degree distilled water after removal of calculus and soft tissues. The teeth embedded in acrylic blocks up to the enamel-cementum boundary were abraded from the occlusal surface perpendicular to the long axis of the tooth, leaving the dentin surface exposed. A 600-grit silicon carbide (SiC) abrasive was applied to the abraded tooth surface for 1 minute under water to obtain a standardized smear layer on the specimens and to smooth the dentin surface. This surface was divided into two sections by marking the center mesially and distally with a bur, then rinsed with water and gently dried with oil-free air. The mesial half of the surface was treated with 35% phosphoric acid (K-ETCHANT Syringe, Kuraray) for 15 s while the distal half was not treated with acid. It was then rinsed for 15 s and dried again

using oil-free air. Adhesives were applied on the prepared surface in total-etch mode on the mesial part and self-etch mode on the distal part and polymerized with light (Q-Light, Woodpecker, Germany) for 30 s.

As shown in the figure, this study planned eight sample groups according to material type and two subgroups within each group including adhesive procedures (Figure 1).

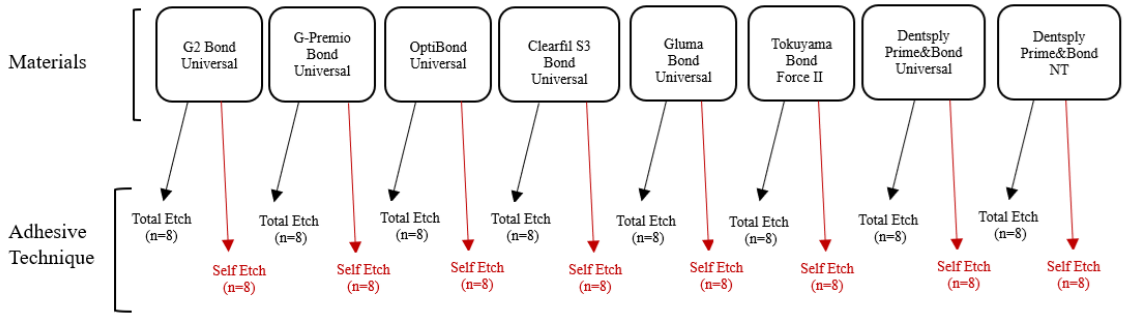

Figure 1: Groups, sample numbers and adhesive application methods

Then, a flowable composite (Clearfil Majesty Flow, Kuraray) was placed in a transparent mold (2.38 mm diameter and 2 mm height) and light polymerized for 30 seconds. Adhesive systems have been applied according to user instructions. The specimens were post-polymerized in water at 37 degrees for a day and then aged. (Moddental- 1000 cycles) The temperature settings for the ageing baths were 5°C and 55°C. Immersion time in each bath was planned as 30 s and transfer time as 5 s.

SBS Test

The SBS test was performed on a universal testing machine (H5KS Redhill, UK) at a crosshead speed of 1 mm/min until specimen fracture. The force required for debonding was obtained in Newtons (N) and converted to megapascals (MPa) by dividing by the surface area of the composite material. (Figure 2)

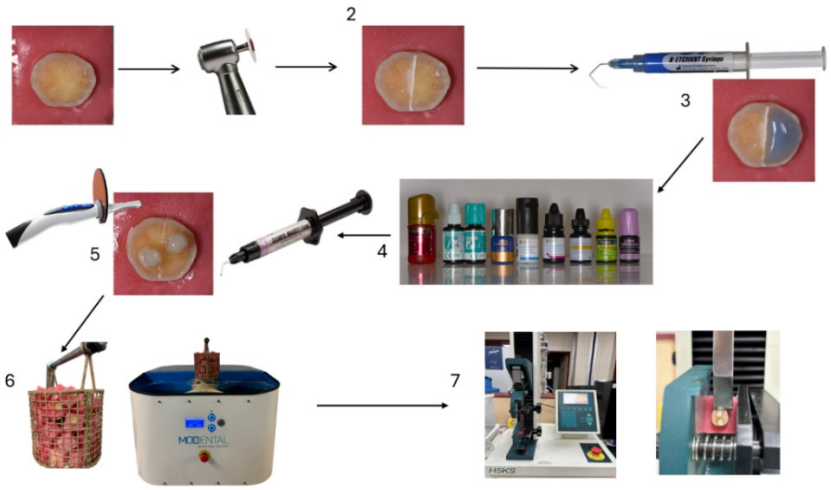

Figure 2: Schematic representation of sample preparation

Microscopic Analysis

Fracture surfaces were examined with (Leica MZ 12, Leica, Germany). If the fracture line is between the tooth and the composite cylinder, the fracture mode is classified as adhesive. If the fracture line partially extends across the adhesive interface and penetrates one of the substrates, the fracture mode is classified as mixed, so we distinguish mixed fracture mode in dentin or composite (depending on which substrate it

covers). If more than 75% of the adhesive area contains dentin or composite, the fracture mode is classified as cohesive.

Statistical Analysis

SPSS 22.0 (SPSS Inc., Chicago, IL, USA) program was used to analyze the SBS data. The normality distribution of the SBS data obtained from the study was performed by Kolmogorov-Smirnov test. Normally distributed SBS data were evaluated using two-way analysis of variance (ANOVA) and post hoc Tukey test ($p<0.05$).

RESULT

SBS findings are presented in Table 2. SBS values of universal adhesives on dentin showed statistically significant differences according to adhesive and application method ($p<0.05$). Total-etch application produced statistically higher SBS than self-etch application ($p<0.05$). Two-step universal adhesive (G2 Bond Universal) showed the highest SBS in all application methods ($p<0.05$). The SBS values for OptiBond Universal, Clearfil S3 Bond Universal, Gluma Bond Universal and Prime&Bond Universal were not statistically significantly different ($p > 0.05$) in the total-etch application method. The single bottle adhesive Bond Force II was the adhesive system with the lowest values in both methods ($p<0.05$).

Table 2: SBS values (MPa) of the adhesives used in the study

Adhesive/Application	Self-Etch	Total-Etch	p
G2-Bond Universal	$10.67\pm4.9^{a,A}$	$18.5\pm2.8^{a,B}$	0,000
G-Premio Bond	$5.7\pm1.4^{b,A}$	$8.5\pm2.3^{b,B}$	0.040
OptiBond Universal	$5.4\pm1.7^{b,A}$	$10.8\pm2.7^{c,B}$	0.000
Clearfil S3 Bond Universal	$6.3\pm0.9^{bc,A}$	$10.8\pm1.8^{c,B}$	0.000
Gluma Bond Universal	$8.1\pm2.1^{c,A}$	$11.6\pm2.5^{cd,B}$	0.003
Bond Force II	$3.1\pm0.5^{d,A}$	$7.6\pm1.2^{b,B}$	0.000
Prime&Bond Universal	$4.6\pm1.7^{b,A}$	$13.3\pm3.2^{d,B}$	0.000
Prime&Bond NT	$6.1\pm1.8^{bc,A}$	$14.9\pm1.5^{d,B}$	0.000
p	0.000	0.000	

* Statistically significant difference between self-etch application and total etch application is shown with A-D, statistically significant difference between adhesives is shown with a-d ($p<0.05$).

When the fracture types were analyzed, the most fracture surface was seen in the adhesive type. (Figure 3) Cohesive and mixed type fractures were mostly seen in the total-etch application of G2 Bond Universal adhesive. In the self-etch application of all universal adhesives, only adhesive type fracture was observed. Bond Force II and G-Premio Bond showed only adhesive type fracture in both self-etch and total-etch application.

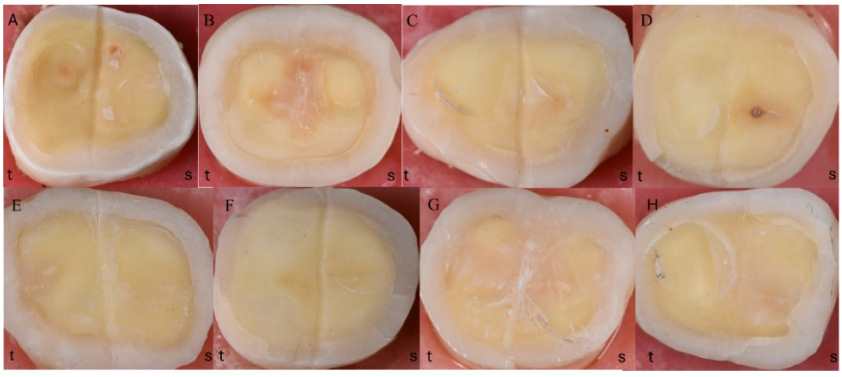

Figure 3: Fracture types between material and tooth (A: G2 Bond Universal, B: G-Premio Bond, C: OptiBond Universal, D: Clearfil S3 Bond Universal, E: Gluma Bond Universal, F: Tokuyama Bond Force II, G: Densply Prime&Bond Universal, H: Densply Prime&Bond NT, t:total etch, s:self etch)

DISCUSSION

The performance of a restorative material in clinical practice is essential for material selection. Clinical trials are more reliable than in vitro studies in determining the success of restorative treatment and the durability of the material However, it is not easy to determine the cause of a failed restoration in clinical studies. Clinical studies, especially when testing the durability of the material, require more time than laboratory studies. Given the progressive evolution of materials, often the tested material is no longer in daily use by the time the study is completed.[16] This is why adhesive systems are often selected based on the results of laboratory tests, but these tests are affected by many variables, including specimen characteristics, specimen preparation, handling, storage and testing technique.[3]

Adhesive systems are an important component of restorative treatment outcomes. It enables the interaction between the resin and the dental substrate.[17] Without proper mechanical properties of the adhesive, the choice of resin is irrelevant because failure of the restoration is inevitable. Therefore, it is useful and important to compare dentin bond strengths and to test the bond strength of various new adhesive systems.[3]

Both the treatment modalities and materials used in dentistry are constantly changing and evolving. While the oldest adhesives on the market, total-etch adhesives, are still the gold standard for dental bonding, the trend is to develop self-etching materials that are easier to apply.[7] This simplifies manipulation by combining all components into a single dental material, reducing the number of steps to a one-step system.[9,10] Universal adhesives are easy to use, faster to apply, and less sensitive to changes in operator technique than multi-step etch-and-rinse adhesives.[5,18] There is also a wide variety of commercially available and widely used universal adhesives.

This study compared several new universal adhesives for shear bond strength. Shear bond testing is the most common method for determining bond strength.[19]

Acid etching of dentin removes the smear layer and demineralizes the subsurface. This is a predictable clinical procedure, but some factors inherent in the conditioning of dentin tissue can affect the bond strength of adhesives.[20,21] Dentin collagen exposed to total-etch adhesives has been shown to be highly susceptible to hydrolytic and enzymatic degradation processes.[10,22] Although adhesive systems are becoming simpler, careful management is still required, particularly with regard to the effect of substrate pretreatment on bond performance. Acceptable bond strength values can be achieved without dentin pretreatment, regardless of the adhesive system used, under less sensitive technical conditions. In the study by Poggio et all.,[23] the application of acid to universal adhesives resulted in a weakening of the bond.

According to the results of our study, the two-stage G2 Bond Universal had the highest and Tokuyama Bond Force II had the lowest SBS values. According to the results of a study by Jäggi et all.[2] it is possible that the adhesives tested, especially those with higher pH, may perform better when used with phosphoric acid. In our study, Tokuyama Bond Force II, which had the highest pH, had the lowest bond strength in both applications. This contradicts the result found by Jäggi et all.[2]

Related researches has generally not supported the notion that a lower pH will result in better bond strength. A previous study evaluating the dentin bond strength of self-etching adhesives with different pH values found that lower pH adhesives did not have higher dentin bond strength.[24,25] Other

studies have also shown that self-etching adhesives with relatively high pH values provide good dentin bond strength.[26,27]

A smooth specimen surface eliminates potential retentive elements and allows the focus to be on the chemical bonding capabilities of the adhesives. In this study, dentin surfaces were prepared using SiC disks. Of additional importance is the bond between the adhesive and the composite, which is achieved by the cross-linking of methacrylate and other functional groups during polymerization. It may make sense to combine composite materials and adhesive systems from the same manufacturer, as they often use similar base monomers and initiator systems. However, this study could not conclude that "bonding is better with the same manufacturer's products" because adhesives from other manufacturers showed better, worse, and similar results.[2]

Depending on the type of adhesive and the method of application of the same adhesive, universal adhesives can have significantly different bond strengths. According to a study by Brkanović et all.,[3] since the total-etch method has less effect on the fatigue strength of dentin than the self-etch method, a universal adhesive applied by this method will have a lower fatigue strength. However, clinical studies have shown that there is no statistically significant difference between different dentin preparations when a universal adhesive method is used.[3,28] Brkanović et all. p[3] demonstrated in a study that G2-Bond Universal has higher or equal dentin SBS compared to other adhesives, which is consistent with our study. In the same study, G2-Bond Universal was reported to be the most effective way to maintain high dentin bond strength in the self-etching approach compared to other representative adhesives. In our study, G2 Bond Universal, as well as other universal adhesives, showed better bond strength in the total etch application. A meta-analysis by Rosa et all.,[7] total-etch application of universal adhesives to dentin did not show a statistically significant difference compared to self-etch application. Only one adhesive system was reported to have better bond strength with total-etch application.

Despite the efforts of manufacturers to develop and market new materials, the question remains whether clinicians should consider using these new adhesives with pre-acid etching rather than self-etch application. Which bonding protocol is best for multimodal adhesives cannot be definitively answered with the insufficient clinical evidence available and the short follow-up times evaluated.[7]

Although the laboratory results of this study need to be further evaluated clinically, it is important to note from a materials utilization standpoint that adhesives developed for the same indication and approved for clinical use show large differences in their adhesive properties. A better understanding of these concepts could significantly improve the bonding ability of universal adhesives, making this a highly important topic for further research.[2]

CONCLUSION

Within the limitations of this study, the following conclusions were drawn:

1. Two-step universal adhesive G2 Bond Universal had the highest SBS in total-etch and self-etch modes.
2. Universal adhesives show higher SBS values when applied in total-etch mode.
3. The type of universal adhesive and the mode of application have an effect on bond strength.

Ethical Approval

Ethical approval fort his study was obtained from Gülhane University of Health Sciences Pharmaceutical and Medical Received by off-device ethics committee (2024/284).

Financial Support

No financial support was received from any institution or organization for this study.

Conflict of Interest

The authors deny any conflicts of interest related to this study.

Author Contributions

Design: IKC, FO, SK, Data collection or data entry: IKC, NA, Analysis and interpretation: IKC, SK, Literature review: IKC, NA, Writing: IKC, NA, FO.

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