

Effect of the Application Procedure on The Bond Strength of Universal Adhesive in Restoration Repair

Uygulama Prosedürünün Restorasyon Onarımında Üniuersal Adezivin Bağlanma Dayanımına Etkisi

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ABSTRACT

Objective: This study aimed to evaluate the effect of reduced application time on the bond strength of a universal adhesive used for the repair of hybrid ceramic and composite resin restorations.

Methods: In the study, a total of 48 rectangular prisms of sizes 7×6×2 mm hybrid ceramic (Vita Enamic, Zahnfabrik, Germany) and composite resin (Solare X, GC, Japan) specimens were prepared. All samples were aged with 5000 thermal cycles. Then, the upper surfaces of the samples were roughened with a diamond fissure bur (10–36µ, 852 FG Meisinger; Hager & Meisinger GmbH, Germany) with 5 strokes under water cooling. The samples were divided into 2 subgroups according to the different application times of the adhesive system (G-Premio Bond, GC, Japan) (n=12). Then, composite resin (Solare X, GC) was placed using plastic cylinder molds with 3 mm inner diameter and height and cured with light for 20 seconds. All samples were thermal aged and then a shear bond strength test was conducted. The data were analyzed with Two-way ANOVA and post-hoc Tukey HSD tests ($P<.05$).

Results: Two-way ANOVA revealed that the repair bond strength of the 10 seconds of waiting was significantly higher than the quick application for both hybrid ceramic and composite resin ($P<.05$). It was determined that there was no significant difference between hybrid ceramic and composite resin in the both quick application group and 10 seconds waiting group ($P>.05$).

Conclusion: Quick application procedure showed lower shear bond strength than 10 seconds waiting group for the repair of both composite resin and hybrid ceramic ($P<.05$).

Keywords: Quick application, Application time, Universal adhesive, restoration repair

ÖZ

Amaç: Bu çalışmanın amacı hızlı uygulama prosedürünün bir üniuersal adezivin, hibrit seramik ve kompozit rezin restorasyonların onarımında bağlanma dayanımı üzerine etkisinin değerlendirilmesidir.

Yöntem: Çalışmada 7×6×2 mm boyutlarında dikdörtgen prizması şeklinde toplam 48 adet hibrit seramik (Vita Enamic, Zahnfabrik, Almanya) ve kompozit rezin (Solare X, GC, Japonya) örnekler hazırlandı. Tüm örnekler 5000 termal döngü ile yaşlandırmaya tabi tutuldu. Daha sonra örneklerin üst yüzeyleri su soğutması altında elmas fissür frez (10–36µ, 852 FG Meisinger; Hager & Meisinger GmbH, Almanya) ile 5 kez yüzeyden geçilerek aşındırıldı. Örnekler kendi içlerinde adeziv sistemin (G-Premio Bond, GC, Japonya) farklı uygulama sürelerine göre 2 alt gruba ayrıldı (n=12). Daha sonra 3 mm iç çapa ve yüksekliğe sahip plastik silindirik kalıplar kullanılarak kompozit rezin (Solare X, GC) tabakalı olarak yerleştirildi ve polimerize edildi. Termal yaşlandırma sonrasında makaslama bağlanma dayanımı test edildi. Çift-Yönlü varyans analizi (ANOVA) ve post-hoc Tukey HSD testleri ile verilerin istatistiksel analizi yapıldı ($P<.05$).

Bulgular: Çift-Yönlü ANOVA sonuçlarına göre, hibrit seramik ve kompozit rezin onarımında 10 sn bekletilerek uygulama hemen uygulamaya göre anlamlı ölçüde daha yüksek bağlanma dayanımı gösterdi ($P<.05$). Hibrit seramik ile kompozit rezin arasında her iki uygulama grubunda da anlamlı farklılık olmadığı belirlendi ($P>.05$).

Sonuç: Hızlı uygulama prosedürü, kompozit rezin ve hibrit seramik onarımında 10 sn bekletilerek uygulamaya göre daha düşük makaslama bağlanma dayanımı göstermiştir.

Anahtar Kelimeler: Hızlı uygulama, Uygulama süresi, Üniuersal adeziv, Restorasyon onarımı

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INTRODUCTION

Development of the dental adhesive systems has brought important innovations into clinical dentistry practice. One of these innovations, universal adhesives, was introduced to the market in 2011 and continues developing.¹ Universal adhesives are designed as the "all-in-one" concept of currently used single-step self-etch adhesives, and offer the advantages of being easier, less technical sensitivity and quick application, different from previous adhesive systems.^{2,3}

Manufacturers state that universal adhesives can be used for the placement of direct and indirect restorations with total-etch, self-etch, and selective etching procedures.² Universal adhesives contain various functional monomers such as 4-META (4 methacryloyloxyethyl trimellitate anhydrides), 10-MDP (10 methacryloyloxydecyl dihydrogen phosphate) or GPDM (glycerol phosphate dimethacrylate).⁴ These multi-directional functional monomers; enables bonding to different surfaces such as tooth surface, resin-containing cement, noble and non-precious metals, zirconium, porcelain, and composite.^{2,4} These properties are the reason for extensive use of universal adhesives in clinical practice.^{1,5}

Marginal gap formation, development of secondary caries, or fracture of the restoration are the main reasons of restoration failure.⁶ In this cases, there are two treatment options: complete restoration renewal or repair of the restoration. If failures consist of minor deformities such as anatomical deterioration, restoration fracture, or external discoloration, repair of the restoration can be preferred.⁷ In addition complete restoration renewal is a costly and time-consuming procedure, and can cause the unintentional removal of the sound tooth structures.^{8,9} Fernandez et al.¹⁰ showed that renewal or repair of the restorations demonstrated similar features in terms of deterioration of marginal adaptation, development of secondary caries, maintaining anatomical form, and restorations that were completed with both of the procedures were clinically acceptable after 10 years.

Development in adhesive dentistry has not only reduced the size of the cavity preparation but also provided retention to the restoration repair without the need for extensive preparation. Restoration repair can be quickly performed with the help of composite and adhesive systems.⁹ Recently, the use of universal adhesives with reduced application time seen as clinically attractive in restoration repair. However, it is thought that the reduced application time may cause the risks of not removing the solvent completely in the adhesive layer and deterioration of the layer after water absorption.¹¹ It has been reported that micromechanical retention and chemical bonding are important factors in the repair process.¹² However, it is doubtful whether the reduced application time will allow chemical bonding. It is indicated by the manufacturer of G-Premio Bond (GP; GC, Tokyo, Japan) that is one of the currently developed universal adhesives, can be used with a waiting time of 10 seconds after the application or air application and polymerization can be done without waiting after the application of the adhesive in restoration repair.

In light of this information, the aim of this study was to evaluate the effect of reduced application time on the bond strength of a universal adhesive in the repair of hybrid ceramic and composite restorations. Tested null hypothesis; 'Different application times of the universal adhesive do not affect repair bond strength of the hybrid ceramic or composite resin restorations.

METHODS

The contents and manufacturer information of the restorative materials and universal adhesive used in this study are given in **Table 1**.

From the hybrid ceramic blocks (Vita Enamic, Zahnfabrik, Bad Säckingen, Germany) used in the study, 24 rectangular prisms of 7×6×2 mm in size were prepared with a precision cutting device (Micracut 125, Metkon, Bursa, Turkey) under water cooling. For the preparation of the samples in the composite resin group, 7×6×2 mm rectangular prism-shaped negative cavity molds were created using silicone impression material (Zeta plus, Zhermack, Rovigo, Italy). Micro-hybrid composite resin (Solare X GC, Tokyo, Japan) was placed in two layers and light-cured for 20 seconds (Valo; standard mode, Ultradent Products Inc., South Jordan, UT, USA) and 24 composite resin specimens were obtained. Hybrid ceramic and composite resin specimens were placed in acrylic molds with their tops parallel to the ground plane, and these surfaces were polished using silicon carbide papers of 600, 800, and 1.200 grit, respectively. The specimens were subsequently thermo-cycled (SD Mechatronic Thermocycler, SD Mechatronic GMBH, Westerham, Germany) with a transfer time of 5 s and a dwell time of 30 s for 5.000 cycles in distilled water (5 ±2°C / 55 ±2°C).

Table 1. Compositions and manufacturer details of the tested restorative materials

Material	Composition	Manufacturer
Vita Enamic (Hybrid Ceramic CAD/CAM Block)	CAD/CAM ceramic (86% by weight feldspathic ceramic, 14% by weight polymer)	Vita Zahnfabrik, Bad Säckingen, Germany
GC Solare X (Composite Resin)	UDMA, Silica powder, Organic fillers, Dimethacrylate, Photoinitiator	GC, Tokyo Japan
G-Premio BOND (Universal Adhesive)	10-MDP, 4-META, 10-methacryloyloxydecyl dihydrogen thiophosphate, methacrylate, and ester, distilled water, acetone, photoinitiators, silica	GC, Tokyo Japan
Porcelain Primer	Silan, ethanol	Bisco, Schaumburg, IL, USA

10-MDP: 10-methacryloyloxydecyl dihydrogen phosphate; 4-META: 4-methacryloyloxyethyltrimellitate anhydride; UDMA: urethane dimethacrylates

Surface Treatments and Repair Procedure

The upper surfaces of the hybrid ceramic and composite resin specimens were ground with a diamond fissure bur (10–36µ, 852 FG Meisinger; Hager & Meisinger GmbH, Neuss Germany) under water cooling with a high-speed aerator under stable pressure for 5 times. After using four samples, the bur was replaced with a new one, in case the grains were changed. Hybrid ceramic and composite resin samples were divided into 2 subgroups according to the different application procedures of the adhesive agent (n=12). The following procedures were applied to the groups.

Group HC-A (Hybrid ceramic (HC) + quick application (A)): Silane was applied to the surfaces of the hybrid ceramic samples according to the manufacturer's instructions. After the universal adhesive application, adhesive was immediately dried with maximum air pressure for 5 s and light cured for 10 s.

Group HC-B (Hybrid ceramic (HC) + 10 seconds waiting group (B)): Silane was applied to the surfaces of the hybrid ceramic samples according to the manufacturer's instructions. After the universal adhesive application, waited for 10 s, followed by drying with maximum air pressure for 5 s and light-cured for 10 s.

Group CR-A (Composite Resin (CR) + quick application (A)): Immediately after the application of the universal adhesive to the surfaces of the composite resin samples, dried with maximum air pressure for 5 s and light-cured for 10 s.

Group CR-B (Composite Resin (CR) + 10 seconds waiting group (B)):

After the application of the universal adhesive to the surfaces of the composite resin samples, waited for 10 s, then dried with maximum air pressure for 5 s and light-cured for 10 s.

After the adhesive application, a micro-hybrid composite resin (Solare X, GC) was applied to the hybrid ceramic and composite surfaces at a height of 3 mm by layering technique using 3 mm diameter plastic cylinder molds and cured with light-cured for 20 s. Then, all samples were subsequently thermo-cycled with a transfer time of 5 s and a dwell time of 30 s for 5000 cycles in distilled water (5 ±2°C / 55 ±2°C).

Shear Bond Strength Test

The shear bond strength (SBS) test was performed via a universal testing device (AGS-X Series, Shimadzu Europa GmbH, Germany) at a crosshead speed of 0.5 mm/min until failure occurred. The obtained values were converted into units of MPa through division by bonding surface area (N/mm²).

Fracture Mode Analysis

The fracture types of the samples were evaluated with a stereomicroscope (Stemi C-2000; Zeiss, Oberkochen, Germany) under ×40 magnification and classified as indicated; Adhesive fracture: Between restorative materials and adhesive, Cohesive fracture: Fracture in restorative materials, Mixed fracture: Fracture in which adhesive and cohesive fracture¹³.

FE-SEM Evaluation

Two samples from each type of fracture were randomly selected and observed with field emission scanning electron microscopy (FE-SEM) (Mira 3 XMU, Tescan, Brno, Czech Republic) to determine the surface properties. The samples were sputter coated with Pt-Pd and examined at ×250 and ×1000 magnifications.

Statistical Analysis

Statistical analyzes were performed using the SPSS v.19 package program (ISPSS Inc, IBM Corp, IL, USA). The normal distribution of the data was determined using Shapiro-Wilk's test. Two-way analysis of variance (ANOVA) and post-hoc Tukey HSD tests was used to evaluate the effect of reduced application time of the universal adhesive on bond strength in the repair of hybrid ceramic and composite resin restorations (P<.05).

RESULTS

Shear Bond Strength Test

As a result of the statistical analysis, it was seen that the different application times of the universal adhesive had a significant effect on the bond strength (P < .001) and the material type did not have a significant effect (P > .05) on the repair of hybrid ceramic and composite resin specimens.

The mean shear bond strength and standard deviation values of the study groups are shown in Table 2. In hybrid ceramic and composite resin repair, the 10 seconds waiting group showed a significantly higher bond strength than the quick application (P < .05). It was determined that there was no significant difference between hybrid ceramic and composite resin in the quick application group and the 10 seconds waiting group (P > .05).

Table 2. Mean shear bond strength and standard deviation values of the study groups

	Quick application (A)	10 seconds waiting group (B)	p value
Composite Resin(CR)	13,41±2,93	16,19±1,92	0,006
Hybrid Ceramic(HC)	13,28±2,37	17,51±2,61	<0,001
p value	0,900	0,181	

Fracture Type Analysis

The distribution of the fracture types of the tested groups is shown in Table 3 and the sample of fracture-type images are shown in Figure 1. The adhesive fracture was common in all study groups. It was seen that the cohesive type fractures increased in the 10 seconds waiting group.

Table 3. Distribution of fracture types according to study groups.

Fracture Types	Composite Resin (CR)		Hybrid Ceramic (HC)	
	Quick application (A)	10 seconds waiting group (B)	Quick application (A)	10 seconds waiting group (B)
Adhesive	8	7	10	7
Cohesive	0	2	2	3
Mix	4	3	0	2

FE-SEM Analysis

When the adhesive fracture surfaces were examined at higher magnifications (×250 and ×1000), pores were observed in the adhesive layer. These pores were more in number and larger in the quick application group compared to the 10 seconds waiting group (Figure 2).

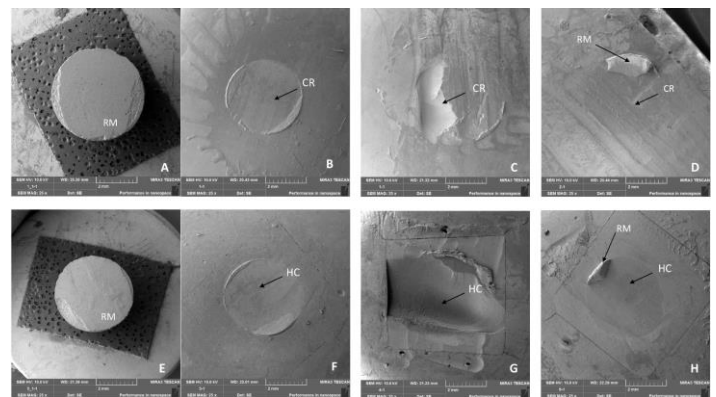


Figure 1. FE-SEM images of the fracture types (x25). A: Adhesive fracture in composite resin group (repair material: RM), B: Adhesive fracture in composite resin group (composite resin surface) C: Cohesive fracture in composite resin group D: Mixed fracture in composite resin group E: Adhesive fracture in the hybrid ceramic group (repair material) F: Adhesive fracture (hybrid ceramic surface) in the hybrid ceramic group G: Cohesive fracture in the hybrid ceramic group H: Mixed fracture in the hybrid ceramic group

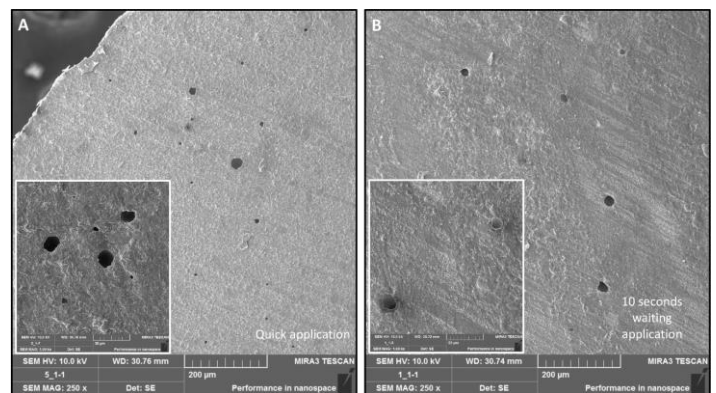


Figure 2. High magnification (x250 and x1000) FE-SEM images of the surface of the fractured repair material for different application times. A: Quick application, B: 10 seconds waiting group.

DISCUSSION

As a result of this study, it was determined that the application of universal adhesive with reduced application time has a significant effect on shear bond strength in hybrid ceramic and composite resin repair. Therefore, the null hypothesis of 'Different application times of the universal adhesive do not affect repair bond strength of the hybrid ceramic or composite resin restorations' was rejected.

The need of restoration repair usually occur after a certain period of clinical use of the restoration. The aging of pre-repair materials is important for simulating oral conditions in laboratory studies that examining the repair potential of restorative materials. For this purpose, various *in vitro* aging protocols such as thermal cycle,¹⁴ boiling in water,¹⁵ and water storage¹⁶ are available. However, there is no consensus on aging protocols. In this study, the effect of the application time of the adhesive agent on the bond strength was investigated rather than the effect of the aging protocol. Therefore, to provide comparability, all samples were thermally cycled to simulate pre- and post-repair clinical conditions.

Universal adhesives can be used as primers on various silica-based ceramics, noble metals, non-precious metals, composite resins, and substrates such as zirconium, as well as adhesion to dental tissues. These adhesives make it possible to adhere to surfaces without the need for different products such as silane, metal, and zirconia primers.¹ Manufacturer of the GP, one of the universal adhesives available in the market, states that it can be used in composite restoration repair without the need for any additional material or primer application, but if the surface to be repaired contains glass ceramic or hybrid ceramic, additional silane should be applied to these surfaces before the adhesive application. Therefore, in this study, an additional silane application was applied in hybrid ceramic repair, while no additional silane application was applied in composite resin repair.

Restoration repair is more economical and easier to apply in a shorter time than renewal of the restoration. Despite these advantages, there is no standard application protocol for restoration repair. In some of the previous studies, it was stated that macro- or micro-mechanical roughening of the substrate surface is more important than the adhesive technique used;⁷ some of them showed the importance of the adhesive technique used.¹⁷ On the other hand, it is preferred by clinicians that restoration repair procedures are more practical and can be quickly applied.

Recently, universal adhesives with the 'quick bond' concept have been introduced by different manufacturers. In such universal adhesive systems, after the adhesive application, it can be polymerized without the need for waiting and active application for solvents to evaporate or adhesive to interact with the adherent surface. G-Premio Bond is one of the first adhesive systems to be used with this concept. Therefore, the effect of the short application time of GP on the repair bond strength of composite and hybrid ceramic restorations was evaluated in this study.

In this study; It was determined that GP for the 10-second waiting without active application provided significantly higher shear bond strength values on both hybrid ceramic and composite resin surfaces. Prolonging the time between the application of adhesive systems to the surface and their curing; is known to contributes to the reduction of the water permeability of the adhesive layer,¹⁸ the separation of acidic functional groups from the structure, and the increase in the infiltration of resin monomers.¹⁹ Adhesive systems used today are generally formulated using solvents such as acetone, ethanol, and water with hydrophilic and hydrophobic resin monomers dissolved in their combination.²⁰ The removal of solvents and water from the adhesive

layer before polymerization is extremely important for the quality of the adhesion.²¹ Shortening the time between applying the adhesive to the surface and curing may not be sufficient for solvent evaporation in quick application groups. Therefore, the solvent that cannot be removed from the environment adversely affects the adhesive polymerization and thus the bond strength obtained.^{22,23} Saikaew et al. stated that the shorter application time creates large and very prominent pores on the adhesive surface and this may be due to the insufficient removal of the solvent and water.¹¹ In this study, similarly large and prominent pores were determined in the FE-SEM images for the quick application groups. In addition, excessive air drying could be thinned the adhesive layer too much which could cause inhibition of the polymerization reaction by oxygen to form peroxide radicals^{24,25}. GP contains acetone and water as solvents. Rapid evaporation of acetone in the adhesive; causes a decrease in solvent-resin affinity and promotes the formation of a rich phase that provides cross-linking in the monomer.²⁶ If the evaporation of acetone exceeds the evaporation of water, the water accumulation increases in the adhesive layer.²⁷ For these reasons, the shortened application time may not be enough to allow water to be removed from the partially polymerized adhesive layer.²¹

In general, acetone-containing adhesives have a lower pH than ethanol-containing adhesives.¹² Slightly acidic adhesives are less prone to hydrolytic degradation than more acidic ones and promote bond stability over time.²⁸ GP has a pH of 1.5 and is considered moderately acidic adhesive. This may cause the adhesive layer to exhibit lower mechanical properties. Sai et al.²⁹, reported that five different universal adhesives applied to the enamel surface by the quick application procedure or following the manufacturer's instructions did not show a significant difference in shear bond strength. Buket K. et al.³⁰ reported that the application of three different universal adhesive systems to the dentin surface for 5 seconds, 10 seconds, and 20 seconds creates shear bond strength values that vary according to the adhesive system. Huang et al.³¹, reported that the rapid application procedure on the dentin surface with GP showed lower bond strength than waiting for 20 seconds. There is no study in the literature in which the bond strength of the quick application procedure with universal adhesives was evaluated in the restoration repair and therefore the results of this study can not be compared. However, the results of the study show parallelism with the studies evaluating the bond strength to the dentin surface.

Previous studies has been reported that the use of silane in ceramic or composite resin repairs has significantly positive effects compared to the situations in which silane is not used^{9,32} or that no beneficial effect has been observed.^{28,33} It has been reported that the application of silane increases the wettability of the surface to be repaired and creates a chemical bond between the resin matrix and silica or glass filler particles.^{34,35} In light of this information, it was thought that the higher shear bond values obtained in hybrid ceramic repair could be the result of additional silane application. The dominant failure mode was the adhesive type failure for both of the adhesive application procedures, and adhesive type failures more frequent in the quick application procedure. This may be due to the insufficient resin polymerization, incomplete removal of the solvent and the lower mechanical properties of the adhesive layer.

This study was conducted under *in vitro* conditions. Various factors such as occlusal forces and oral fluids were not taken into account and only one commercially available universal adhesive was tested. Therefore, more *in vitro* and *in vivo* studies are required to confirm the results of the present study.

CONCLUSION

Within the limits of this study; Increasing the application time of universal adhesives in hybrid ceramic and composite resin repair procedures resulted in higher bond strength values. Similar bond strength was observed for the hybrid ceramic and composite resin repair in both quick application and 10 seconds of waiting for groups.

Etik Komite Onayı: Araştırmada herhangi bir canlıya ait biyolojik doku kullanılmadığından etik kurul onayı alınmadı.

Hakem Değerlendirmesi: Dış bağımsız.

Yazar Katkıları: Fikir – G.T., H.H.; Tasarım – G.T., H.H.; Denetleme – G.T., H.H.; Kaynaklar – G.T., H.H.; Malzemeler – G.T., H.H. F.K, B.G.; Veri Toplanması ve/veya İşlemesi –G.T., H.H. F.K, B.G.; Analiz ve/veya Yorum – G.T., H.H.; Literature Search – G.T., H.H.; Yazıyı Yazan – G.T., H.H.; Eleştirel İnceleme – G.T., H.H.

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