



Effect of Self-cured Universal Adhesive System on Shear Bond Strengths of Conventional and Bulk-fill Composites

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ABSTRACT

Aim: The aim of this study was to investigate the effect of self-cured universal adhesive system on shear bond strengths of two conventional and two bulk-fill composites to dentin.

Materials and Methods: In this study, four groups were formed: two conventional condensable composites[G-aenial posterior(GC), Palfique Estelite Paste(Tokuyama)], and two bulk-fill composites[Estelite Bulk Fill Flow(Tokuyama), Beautifil Bulk Restorative(Shofu)]. Each group was divided into two subgroups; G-premio bond (control group) ve self-cured Universal Bond (Tokuyama). 32 human third molar human teeth were prepared to expose dentin surfaces. After the preparation, the teeth were randomly divided into 4 main groups and 2 subgroups (n = 8), composites were applied to the surfaces with the help of cylindrical silicone transparent mold with a diameter of 4 mm and a height of 4 mm.

Results: In this study, when both adhesive systems are evaluated; the highest shear bond strength value was Palfique Estelite Paste, while the lowest shear bond strength value was Beautifil Bulk Restorative. While the difference between Palfique Estelite Paste and Estelite bulk-fill flow, Beautifil Bulk Restorative was found to be statistically significant, the difference with G-aenial posterior was not significant in both adhesive groups. The difference between G-aenial posterior and Beautifil Bulk Restorative was found to be statistically significant in both adhesive groups. When adhesive systems are compared; G-premio bond was found to be higher in all composite groups compared to self-cured Universal Bond, but the difference between them was statistically insignificant.

Conclusions: While conventional composites exhibited high shear bond strengths, bulk-fill composites showed values close to these values. The self-cured adhesive system we used showed near values of shear bond strength to the light-polymerized adhesive system. It is advisable to use self-cured adhesive systems for use in areas where light can be difficult to access in clinical applications.

Keywords: Shear Bond Strength, Bulk-Fill Composites, Self-Cured Adhesive Systems.

Geleneksel ve Bulk-fill Kompozitlerin Makaslama Bağlanma Dayanımlarına Işıksız Adeziv Sistemin Etkisi

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ÖZ

Amaç: Çalışmamızın amacı, iki geleneksel ve iki bulk-fill kompozitlerin dentine makaslama bağlanma dayanımlarına ışiksiz adeziv sistemin etkisinin incelenmesidir.

Materyal-metod: Çalışmamızda iki geleneksel kondanse edilebilen kompozit [G-aenial posterior(GC), Palfique Estelite Paste(Tokuyama)], kütlele yerleştirilebilen iki bulk-fill kompozit [Estelite Bulk Fill Flow(Tokuyama), Beautifil Bulk Restorative(Shofu)] olarak 4 grup oluşturuldu. Her grup iki alt gruba ayrıldı; G-premio bond (Kontrol grubu) ve ışiksiz Universal Bond (Tokuyama). 32 adet insan üçüncü molar insan dişi dentin yüzeyleri açığa çıkacak şekilde prepare edildi. Preparasyonun ardından dişler rastgele 4 ana, 2 alt gruba ayrıldı(n=8), yüzeylere 4 mm çapında ve 4 mm yüksekliğindeki silindirik silikon şeffaf kalıp yardımıyla kompozitler uygulandı. Tüm grupların makaslama bağlanma değerleri universal test cihazında ölçüldü, kuvvet birimi ise "newton" (=N) olarak kalibre edildi.

Bulgular: Çalışmamızda her iki adeziv sistem kullanıldığında da, en yüksek makaslama bağlanma dayanım değerini Estelite Paste gösterirken, en düşük makaslama bağlanma dayanım değerini Beautifil Bulk Restorative göstermiştir. Her iki adeziv sisteminde de Estelite paste ile Bulk-fill flow ve Beautifil Bulk Restorative arasındaki fark istatistiksel olarak anlamlı bulunurken, G-aenial posterior ile arasındaki fark anlamsız bulunmuştur. Adeziv gruplarının her ikisinde de G-aenial posterior ile Beautifil Bulk Restorative arasındaki fark istatistiksel olarak önemli bulunmuştur. Adeziv sistemler kıyaslandığında; tüm kompozit gruplarında G-premio bond, Işıksız Universal Bond'a göre daha yüksek bağlanma sağlarken, aralarındaki fark istatistiksel olarak anlamsız bulunmuştur.

Sonuçlar: Geleneksel kompozitler, yüksek makaslama bağlanma dayanımları sergilerken, bulkfill kompozitler bu değerlere yakın değerler göstermiştir. Kullandığımız ışiksiz adeziv sistem, ışıkla polimerize edilen adeziv sisteme yakın bağlanma değerleri sergilemiştir. Işıksız adeziv sistemler, klinik kullanımlarda ışığın zor olabileceği bölgelerde bağlanma dayanımı açısından kullanılması önerilebilir.

Anahtar Kelimeler: Makaslama Bağlanma Dayanımı, Bulkfill Kompozitler, Işıksız Adezivler.

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Introduction

Newly developed dental materials should be evaluated by in vitro studies before they are put into clinical use. Laboratory tests are similar to clinical trials and are important for gaining insight about mechanical properties. The most commonly used tests to investigate the effectiveness of adhesive systems are bond strength tests. Test methods such as tensile, microtensile, shear are used in vitro to measure the bond strength of materials to dental tissues.^{1,2} Shear bond strength test is one of the most widely used test methods defined by ISO 11405 standard.³ The standard method of shear strength tests is routinely used to determine the adhesion of dental materials to the tooth structure due to its advantages such as ease of sample preparation, reliability, and simple and feasible test protocol.^{4,5}

Universal adhesives have been advanced to rule out complications in etch and rinse and self-etch adhesive practices and to use an adhesive with all types of adhesive application.⁶⁻⁹ Universal adhesives contain acidic functional monomers such as MDP (methacryloxydecyl dihydrogen phosphate).¹⁰ MDP has polymerizable methacrylate groups, phosphate groups capable of chemical bonding with calcium in hydroxyapatite. It is an acidifying monomer due to the dihydrogen phosphate groups it contains. It is highly hydrophobic due to its long carbonyl chain. Forms a stable nano layer on the adhesive interface with hydroxyapatite. The resulting MDP-Ca (calcium) salts precipitate along this layer. This ensures a high and stable bond strength.^{11,12} The hydrophilic monomer HEMA (2-Hydroxyethyl methacrylate) contained in Universal adhesives improves the bonding strength of the adhesive resin by providing better resin infiltration.¹³⁻¹⁵

Giomers are described as resin based restorative material that releases fluoride, contains S-PRG (surface pre-reacted glass ionomer particles) fillers.¹⁶ In the presence of water, an acid-base reaction occurs between fluoride-containing glass and polyacrylic acid, a wet silica hydrogel is formed, and S-PRG fillers are obtained after these events. After freeze-drying the dehydrated xerogel is ground and processed with silane to create S-PRG fillers of a certain size range. S-PRG fillers perform the release of aluminum, boron, fluorine, sodium, silicon and strontium ions. Composite resins containing S-PRG fillers show antibacterial properties thanks to the metal ions released from the composite. Strontium and fluoride transform hydroxyapatite into strontiapatite and fluoroapatite, making the tooth more acid-resistant. At the same time, S-PRG fillers have the ability to regulate the pH of the environment when they come into contact with water or acidic solutions.¹⁸

In recent years, bulk-fill composite materials have been developed to apply composites to the cavity in larger masses and to reduce application time. Due to the ease of application by reducing the time spent in the clinic, the placement of bulk-fill composites in deep and wide

cavities as 4-5 mm thick single layer, bulk-fill composites have been introduced and used with interest by clinicians.¹⁹

The purpose of this work is to analyze the effect of universal self cured adhesive system on shear bond strength of dentin to 2 conventional and 2 bulk-fill composites.

Materials and Methods

Selection of Teeth

Ethics Committee approval dated 04.07.2019 and numbered 2019-07/05 was obtained by Sivas Cumhuriyet University Non-Interventional Clinical Research Ethics Committee to start the study. In the study, 64 permanent human molars were used and these teeth were procured from the teeth extracted for orthodontic or periodontal reasons in the last 6 months in Sivas Cumhuriyet University Faculty of Dentistry, Department of Oral and Maxillofacial Surgery. No tooth extraction was performed for the study. Organic residues on the tooth surface were cleaned by keeping the teeth in 2.5% sodium hypochlorite (NaOCl) solution for 1 hour immediately after extraction. During the supply of teeth, the teeth were kept in distilled water at room temperature and the storage fluid was renewed every week. When all the teeth were collected, the work began.

Preparation of Specimens

Before starting the test, 64 human molars were molded using silicone self curing acrylic to be subjected to shear bond strength testing. The occlusal enamel of the teeth was removed using a low-speed diamond saw until the dentinal surfaces were exposed. Then a 600 grit silicon carbide paper was applied to the surfaces to obtain a standard smear layer. 2 adhesive systems [G-premio bond (Control group, GC, Japan), chemical cured Universal Bond (Tokuyama, Japan)] and 2 conventional composites [G-aenial posterior (GC, Japan), Palfique Estelite Paste (Tokuyama, Japan)] 2 bulk-fill composites [Estelite Bulk Fill Flow (Tokuyama, Japan), Beautifil Bulk Restorative (Shofu, Japan)] were used for the preparation of samples. Materials tested and their compositions are showed in Table 1. In the control group G-premio bond was applied to the surface using applicator, After waiting for 10 s, it was air dried and polymerized using a 10 s LED light device. After the self-cured Universal Bond A and B were mixed, the application was completed within 1 minute and no light application was performed. After adhesive applications, composites were placed using 4 mm diameter, 4 mm high transparent cylindrical mold. In traditional composite groups, 2 layers of 2 mm were inserted by incremental technique. The layers were polymerized (G-aenial posterior 20 sec, Palfique Estelite Paste 30 sec) with LED light device (Valo Cordless, Ultradent, USA). In Bulk-fill composite groups, both bulk fill composite resins were applied without layering and cured for 10 seconds.

Table 1. Materials tested and their composition

Material	Composition
G-premio bond (GC, Japan) (One-stage universal adhesive)	10-MDP, 4-META, 10-methacryloyloxydihydrogen thiophosphate (MDTP), methacrylate adic ester, distilled water, acetone, photoinitiators, silica
Universal Bond (Tokuyama, Japan) (One-stage two-component universal adhesive)	Primer A: Acetone, 3D-SR monomer, MTU-6 (tiourasil monomer), Bis-GMA, TEGDMA, HEMA Primer B: Acetone, isopropanol, purified water, borate catalyst, peroxide, silane coupling agent
G-aenial posterior (GC, Japan) (microhybrid composite)	UDMA, dimethacrylate co-monomers, strontium and lanthanoid, fluoroaluminosilicate glass, silica (Bis-GMA-free)
Palfique Estelite paste (Tokuyama, Japan) (microfill composite)	Bis-GMA, TEGDMA,
Estelite Bulk Fill flow (Tokuyama, Japan) (flowable bulk-fill composite)	Bis-GMA, TEGDMA, Bis-MPEPP, mequinol, dibutyl hydroxyl toluene, uv adsorber, silicon oxide, zirconium oxide
Beautiful Bulk Restorative (Shofu, Japan) (packablegiomer bulk-fill)	Bis-GMA, UDMA, Bis-MPEPP, TEGDMA, fluoro-silicate glass (S-PRG filler based on fluoroboroaluminosilicate glass) polymerization initiator, pigments and others

Shear Bond Strength Test

After the samples were stored in distilled water at 37 °C for 24 hours, they were subjected to shear bond strength test using the universal test machine. The crushing apparatus was placed at an angle of 90 ° with the dentin-composite interface of the samples and shear bond strength test was applied to the samples at a head speed of 0.5 mm/min. The forces applied during the crushing process were measured in Newtons (N) and then converted to Megapascals. After shear bond strength test, fracture surfaces of all samples were enquired under a stereomicroscope with X25 magnification.

SEM Analysis

After all samples were examined by stereomicroscope, SEM analyzes were performed to evaluate the fracture surfaces in detail. The samples were analyzed with SEM device.

Statistical analysis

Variation data of were analyzed using the SPSS statistical software program (22.0 version, SPSS Inc., Chicago, USA). The data were subjected to statistical analysis with using one-way analysis of variance and

Tukey's post hoc test to examine pairwise differences at a significance level of 0.05.

Results

In Table 2, we presented the minimum, maximum values, mean and standard deviations of the composite groups tested, and the differences between the groups. In this study, when both adhesive systems were used, Palfique Estelite Paste showed the highest shear bond strength value, while Beautiful Bulk Restorative showed the lowest value. Palfique Estelite Paste, which showed the highest bond strength, was followed by G-aenial posterior and Estelite Bulk-fill flow, respectively. The difference between Palfique Estelite paste and bulk-fill composites and the difference between G-aenial posterior and Beautiful Bulk Restorative were also statistically significant ($p < 0.05$). If we look at the comparison of adhesives; In all composite groups, G-premio bond showed higher values than self cured Universal Bond, while the differences between them were insignificant ($p > 0.05$). SEM images of the groups are given in figure 1-8.

Table 2. The maximum, minimum, mean and standard deviation values of the shear bond strength tests of the composite resins used in the study

Composites	G-premio bond	Universal bond
Palfique Estelite paste	18.02 (1.49) ^{a,b}	15.97 (1.14) ^{d,e}
G-aenial posterior	17.14 (1.53) ^c	15.07 (1.49) ^f
Bulk-fill flow	15.10 (1.19) ^a	13.23 (0.76) ^d
Beautiful Bulk Restorative	14.68 (1.64) ^{b,c}	12.69 (1.37) ^{e,f}

* F=14.231 p=0.000, p<0.05

a,b,c,d,e,f,g there is a statistical difference between the groups shown with the same lower case letters(p<0.05).

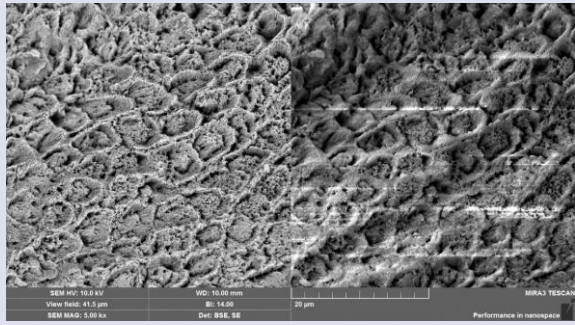


Figure 1. SEM image of G-premio bond + G-aenial posterior

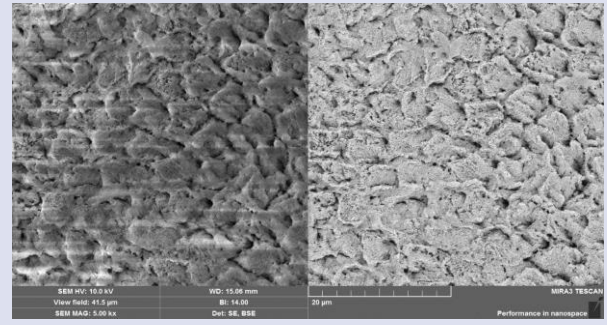


Figure 5. SEM image of G-premio bond + Estelite Bulk-fill flow

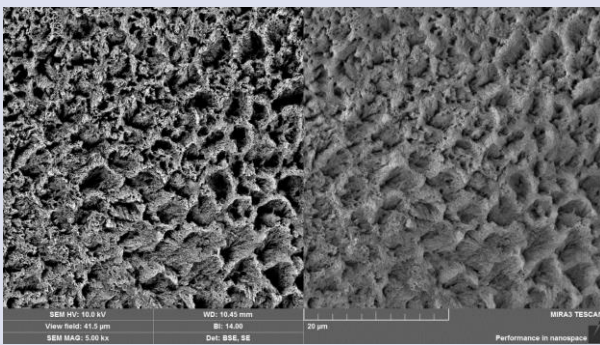


Figure 2. SEM image of Universal bond + G-aenial posterior

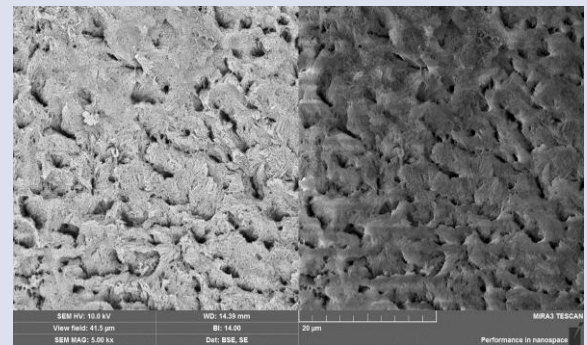


Figure 6. SEM image of Universal bond + Estelite Bulk-fill flow

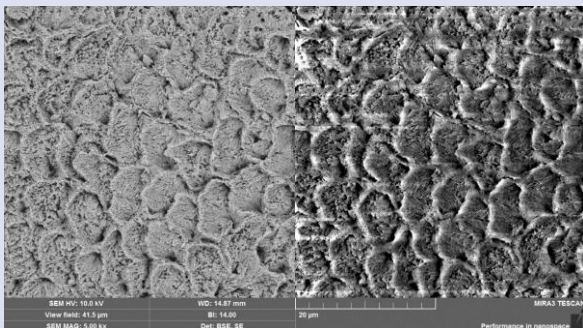


Figure 3. SEM image of G-premio bond + Palfique Estelite Paste

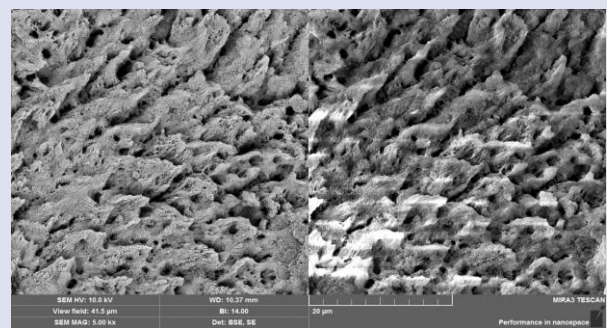


Figure 7. SEM image of G-premio bond + Beautiful Bulk Restorative

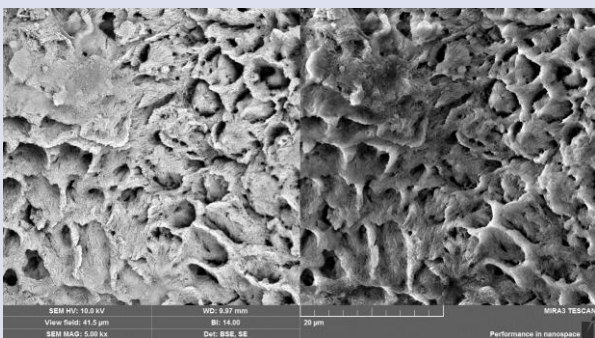


Figure 4. SEM image of Universal bond + Palfique Estelite Paste

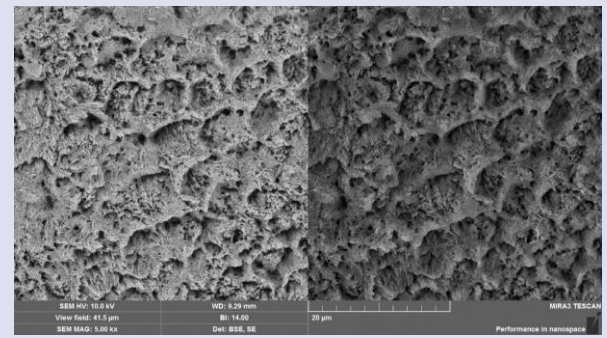


Figure 8. SEM image of Universal bond + Beautiful Bulk Restorative

Discussion

In the field of dentistry, minimally invasive dentistry has greatly increased the use of resin-based materials. The bonding of these materials with the teeth has shown more successful results with the development of adhesive systems. Effective bonding is necessary to reduce microleakage, prevent susceptibility, increase retention and increase caries resistance.²⁰

There are many factors that negatively affect the bonding to dentine tissue. Some of these factors; increasing dentin tubule diameters and numbers from enamel dentin border to pulp, reduction of remaining dentin thickness, dentin sclerosis, dentin fluid causing humidification in the cavity, smear layer, smear plug formation, in deep dentin decreasing of intertubular dentin area and increasing the water content.^{21,22} As the depth of dentin increases, changes in the chemical structure of dentin, increase in diameter and number of dentin tubules, increase in dentin moisture and change in dentin permeability are observed. These factors affect bonding to dentin.²³ In this study, enamel was removed from the occlusal parts of the teeth 2 mm below the dentin border, in order to ensure standardization. In laboratory tests, 600 grit silicon carbide paper is one of the most preferred abrasives in order to standardize the degree of roughness of the dentin surface and the resulting smear layer.²⁴ In this study, 600 grit silicon carbide sander was used to prepare tooth surfaces.

It is important to appraise the bonding performance of newly developed adhesives before they are placed on the market. The most preferred bond strength test method is shear bond strength test.²⁵ Shear bond strength test is a reliable test method used in the assessment of bond strength of materials to different dental tissues. It is frequently preferred especially because it mimics the load distribution in the clinical setting better and achieves significant results in the evaluation of bonding to homogeneous surfaces.²⁶

In order for the use of bulk-fill composite resins to become widespread, it is necessary to be aware of their physical mechanical features and to research their bonding to dental tissue. In this study, we aimed to assess the shear bond strength of 1 condensable, 1 flowable bulk-fill composite resin and 2 conventional composites with using 2 different adhesives.

Melkumyan *et al.*²⁷ compared two in vitro performance of two self etch adhesives (Contax, Bond Force) and two total etch adhesives (Te-Econom Bond, Swisstec SL Bond). Contax and Bond Force composite resin were used with Palfique Estelite Paste, while Te-Econom Bond and Swisstec SL Bond and were used with Swisstec composite resin. According to the their shear bond strength test results, although the bonding of Contax to enamel was not as strong as Te-Econom Bond and Swisstec SL Bond, the difference between them was found to be statistically insignificant. However, the bonding of Contax with dentin was found better than Swisstec SL Bond.

Çolak *et al.*²⁸ scored the shear bond strength of 2 bulk-fill and 2 nanohybrid composites to dentin in premolar teeth. They performed their work in the middle coronal part of the dentin. If we look at the results of the study, it has been reported that 2 nanohybrid composites show higher values than bulk-fill composites bond strength. Almeida *et al.*²⁹ evaluated the microtensile bond strength of 2 bulk-fill and 1 nanocomposite. They also performed their work by preparing Class I cavities. As the bonding technique, they preferred the etch and rinse method in all cavities. SonicFill exhibits the highest bond strength, followed by traditional nanocomposite and Tetric Bulk Fill has lowest value. This result can be associated with sonic activation technology. Fronza *et al.*³⁰ studied the microtensile bond strengths of a microhybrid composite (Herculite Classic), 2 flowable (SureFill SDR Flow, Filtek Bulk Fill Flowable Restorative) and a packable bulk-fill (Tetric EvoCeram Bulk Fill) resin in Class I cavities. While the highest microtensile bond strength was shown by conventional microhybrid composite, the highest bond strength was acquired by packable bulk-fill and the lowest bond strength by the flowable bulk-fill composite Filtek Bulk Fill Flowable Restorative.

Tavarez *et al.*³¹ examined the effect of bulk-fill, microhybrid and nanoparticulate composite resins on shear bond strength. Following the application of 37% phosphoric acid to 4 mm diameter, 2 mm thick cylindrical Filtek Z350 composite blocks, the composites were repaired and shear tested. Of these, microhybrid composite showed higher bonding strength than bulk-fill composite, but didn't find a significant difference between them. The lowest value was obtained by nanoparticulate composite. Tavarez *et al.*'s study supports our study by obtaining high bonding values in the microhybrid composite and differs from this study due to the repair of the composite surface. Although G-aenial posterior, which has a microhybrid structure, follows Palfique Estelite Paste which shows the highest bond strength; the differences between them were not statistically significant.

In our literature research, we could not find a research on the bond strength of Estelite Bulk Fill flow composite. According to the results of our study, Beautifil Bulk Restorative exhibited the lowest shear bond strength. Beautifil Bulk Restorative is different from bulk-fill composites thanks to its giomer structure. Singla *et al.*³² aimed to evaluate the polymerization depths and sub-surface microhardness of 3 bulk fill and 1 conventional composite in cylindrical blocks. SonicFill exhibited the highest surface hardness. When we evaluated the bulk-fills used in the study, it was reported that only giomer bulk-fill exhibited values below the acceptable limit. They suggest that the lower values of the Beautifil Bulk Restorative are associated with a softer PRG filler. Tsujimoto *et al.*³³ studied the cured depth of giomer bulk-fills and nonglomer bulk-fills. As the curing time was raised in all the materials they used, the curing depth raised. When we look at low viscosity materials, Beautifil Bulk

Flow exhibited the lowest curing depth; when we look at high viscosity materials, Beautifil Bulk Restorative exhibited the lowest curing depth. As a result, they indicated that gomers did not exhibit as much curing depth as other bulk-fills.

In the literature, there are not enough studies on the bond strength of self cured Tokuyama Universal Bond. This newly developed universal adhesive system is related to the bond strength; Katsumata *et al.*³⁴ in the study of two different universal adhesive system (Tokuyama Universal, Single Bond Universal) using different restorative materials in the dentin microtensile bond strengths were evaluated, although there isn't any statistically significant difference in terms of microtensile bond strength. It was resulted that self cured Universal Bond exhibited lower values than Single Bond Universal.

Conclusions

Conventional composites showed higher values, while bulk composites exhibited shear bond strength values close to these values. The self cured adhesive system exhibited values close to the light polymerized adhesive system. In clinical applications, the use of self-curing adhesive systems may be recommended for use in hard to light areas. As the bond strengths of the two universal adhesive systems used in the self-etch mode were similar, it is concluded that these systems should be tested in vitro using different modes.

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