Cumhuriyet Dental Journal, 25(1): 54-59, 2022
DOI: https://doi.org/10.7126/cumudj.1012777

Cumhuriyet Dental Journal

| cdj.cumhuriyet.edu.tr | Founded: 1998 Available online, ISSN: 1302-5805 Publisher: Sivas Cumhuriyet Üniversitesi

Effect of Adhesive on Micro Shear Bond Strength of a New Bioactive Restorative Material on Normal and Caries-Affected Dentine

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Research Article

History

Received: 01/11/2021
Accepted: 17/01/2022

ABSTRACT

Objectives: Activa BioActive-Restorative is defined as a self-adhesive dual-cured resin-modified glass ionomer. The suggested application method includes only conditioning to the dentine with acid and keeps bonding optional. This study aims to evaluate the micro shear bond strength in the presence and absence of the bonding agent application after acid conditioning for different dentin surfaces.

Materials and Methods: 30 posterior molars having occlusal decay were used. The dentin surfaces involving normal dentin (ND) and caries affected dentin (CAD) were prepared and prepared three groups: Activa BioActive-Restorative with only 10 seconds of acid application (Act), Activa with acid and bonding application (Act B), GC G-aenial universal posterior composite with acid and bonding application. A total of 6 groups were formed, as each group had normal and caries-affected dentin surfaces. Universal test machines were used to calculate micro shear bond strength at a crosshead speed of 0.5 mm/min, and failure modes were checked by stereomicroscope. One-way ANOVA and Student t-test was applied for statistical analysis.

Results: There was no statistically significant difference between test results of each group neither for ND nor for CAD. There was no statistically significant difference between μ-SBS values of the ND and CAD subgroup for any of the groups. Adhesive failure dominated the other failure modes.

Conclusions: There is no difference between adherence of the material to ND and CAD. Acid application is sufficient for adequate bonding. However, an optional bond application can be recommended, especially for dentin cavities.

Keywords: Micro Shear Bond Strength, Bioactivity, Adhesion

Yapıştırıcının Normal ve Çürükten Etkilenen Dentin Üzerindeki Yeni Bir Biyoaktif, Restoratif Materyalin Mikro Kaydırma Bağ Dayanımı Üzerine Etkisi

ÖZ

Amaç: Activa BioActive-Restorative, self-adeziv, dual-cure, rezin ile modifiye edilmiş bir cam iyonomer olarak tanımlanır. Materyalin uygulama talimatlarında asit kullanılması önerilirken, bonding ajan kullanılması opsiyonel olarak kullanıcıya bırakılmıştır. Bu çalışma, farklı dentin yüzeyleri için asitleme sonrası bonding ajan uygulama ve uygulamama durumlarında mikro makaslama bağlanma dayanımını değerlendirmeyi amaçlamaktadır.

Gereç ve Yöntemler: 30 adet okluzal çürüğü olan molar diş kullanıldı. Normal ve çürükten etkilenmiş dentin içeren düz dentin yüzeyleri hazırlandı ve rastgele üç gruba ayrıldı (n=10): İlk gruba sadece 10 saniyelik asit uygulamasıyla Activa BioActive-Restorative (Act) uygulandı, ikinci gruba asit ve bonding ajan uygulaması ardından Activa uygulandı (Act B), üçüncü gruba asit ve bonding ajan uygulaması ardından GC G-aenial Universal posterior kompozit uygulandı. Her bir diş üzerinde hem normal hem çürükten etkilenmiş dentin yüzeyleri olduğu için toplamda 6 grup oluşturuldu. Bağlanma değerlerini hesaplamak için 0,5mm/dk hızında universal test cihazında makaslama bağlanma dayanımı testi uygulandı ve kırılma modları stereomikroskop ile belirlendi. İstatistiksel analiz için One-way ANOVA ve Student t testi uygulandı.

Bulgular: Gruplar kendi aralarında normal ve çürükten etkilenmiş dentin için test edildiğinde istatistiksel olarak anlamlı bir fark bulunmadı. Her üç grup için normal ve çürükten etkilenmiş dentin alt grubunun μ-SBS değerleri arasında istatistiksel olarak anlamlı bir fark yoktur. Örneklerde en fazla adeziv kırılma gözlendi.

Sonuç: Malzemenin normal ve çürükten etkilenen dentine bağlanması arasında fark yoktur. Yeterli bağlanma için asit uygulaması yeterlidir. Ancak özellikle dentin kavitelerinde isteğe bağlı bir bond uygulaması önerilebilir.

Anahtar Kelimeler: Mikro makaslama Bağlanma Dayanımı, Biyoaktivite, Adezyon.

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How to Cite: Sonkaya E, Süsgün Yıldırım Z. (2021) Effect of Adhesive on Micro Shear Bond Strength of a New Bioactive Restorative Material on Normal and Caries-Affected Dentine, Cumhuriyet Dental Journal, 25(1): 54-59.

Introduction

The most important purpose of restorative dentistry is to clean a carious lesion and restore the anatomy, function, and aesthetics of the tooth with the most appropriate restorative material. With the advancing technology, new concepts in minimally invasive dentistry have been discovered and new materials supporting them have been found.[1] Despite successful advances in the composition and adhesion of resin composites, materials science has always maintained its innovative perspective and worked to develop permanent fluoride-releasing restoration materials.[2] There are many fluoride-releasing materials on the market, but most of them do have not as strong mechanical properties as permanent composite resins. Recent innovations are the production of restorative resin composites with strong mechanical properties or claimed bioactivity.[3]

Repair, reconstruction, and regeneration are aimed with these special materials used. Bioactive materials, in the most general definition, are compounds that create a chemical bond between tissue and material by creating a special biological response when applied to living tissues.[4] Bioactive materials can strengthen the tooth structure by creating natural remineralization by releasing the calcium and phosphate minerals they contain.[5] At the same time, they cover the material and tooth surface with a hydroxyapatite layer to prevent the formation of secondary caries.[6]

Activa BioActive-Restorative is a bioactive composite resin and leads to more fluoride than glass ionomers residual. It claims to contain a shock-absorbing rubberized ionic-resin component and sol-gel derived bioactive glass (BAG) fillers in the bioactive resin matrix[4]. It does not contain BPA derivative monomers such as Bisphenol A, Bis-GMA.[7] Studies have shown bioactive materials have acceptable diametral tensile strength and flexural strength values[8], microleakage[9], and wear resistance.[10] Although the number of long-term randomized clinical studies conducted specifically to address the self-adhesion success of the material is insufficient, these studies have shown inconsistent results. One of these studies claimed that fillings applied with Activa showed 98% acceptable performance, while another study found that fillings showed a very high failure rate.[11,12] This composite is placed in the cavity with a dual barrel automix syringe. Phosphoric acid conditioning is recommended. A bonding agent may or may not be applied. Although there are multiple reasons for the high failure rate, the main factor is thought to be the weak initial bond at the filling-tooth interface.[12] Clinical results and manufacturer's instructions gave researchers the idea that Activa self-adhesion ability should be studied. A considerable amount of literature has not been published on these. The objectives of this research are to determine whether using a bonding agent is necessary or not in Activa adhesion, *in vitro*, by calculating micro shear bond strength in comparison to a conventional composite. Our null hypothesis is that when a bonding agent is not applied, it is also showed adequate micro shear bond strength as a conventional composite.

Materials and Methods

The present *in vitro* study was undertaken in Cukurova University Faculty of Dentistry from May 2021 to August 2021 and was authorized by the Non-Invasive Clinical Research Ethics Committee of Cukurova University No. 44 dated May 21, 2021. The present study applies to the CRIS guidelines. Extracted carious human molars (n=30) were kept in 0.2% sodium azide solution added phosphate-buffered saline. Later, they were embedded in epoxy resin until the tooth enamel was exposed. To access normal dentin's (ND) and caries-affected dentin's (CAD) flat surfaces, their enamel was trimmed horizontally. 30 molars with moderately involved occlusal caries in the dentin were used according to Mount's classification and ICDAS II classification.

Caries detecting dye (Snoop, Pulpdent Corp, USA) was used to clearly define the difference between ND and CAD. Red caries detector dye was applied with 10 seconds interval (sec) to the dentin surface, rinsed, and then dried. Three different colors were observed after the procedure: dark-red, pink, and yellow respectively denoting caries infected, caries affected, and normal dentin. Caries-infected dentin was removed partially by a round steel bur. The dentin surface was flattened using 600-grit silicon carbide paper for 10 sec. Samples (n=30) were unplanned divided into 3 different groups according to tested restorative materials (n=10) (Table 1).

Table 1. Tested materials

Material	Chemical composition	Brand
Activa Bio-Active Restorative	Modified diurethane, other methacrylate monomers, modified polyacrylic acid, silica, amorphous, sodium flouride	Pulpdent Corp, Watertown, MA, USA
GC G-aenial Universal posterior composite	Urethane dimethacrylate, Inorganic and Pre-polymerized filler, Fluoroaluminosilicate, Silica, Trontium and Lanthanoid fluoride	GC Corp. Tokyo, Japan
Gel Etchant	37,5 % orthophosphoric acid gel	Kerr, CA, USA
Optibond All in One	Glycerol phosphate dimethacrylate, acetone,water, ethanol, Triethylene Glycol Dimethacrylate, ytterbium fluoride, photo initiators, accelerators, stabilizers, water	Kerr, CA, USA

Group 1: According to the manufacturer of Activa Bioactive-Restorative's instruction, dentin was etched with acid gel for 10 sec and then washed with water for 15 sec, air-dried slightly (Kerr, USA). Then, Activa BioActive-Restorative (Act) was applied (Pulpdent Corp, USA). It was placed with an applicator gun and polymerized for 20 sec (Valo, Ultradent Products Inc, USA) after allowing it to settle for three-four sec. Its full polymerization continued for three-four minutes.

Group 2: Dentin was etched with acid for 10 sec and washed with water for 15 sec, air-dried slightly. Then, the bonding agent (OptiBond All-In-One, Kerr, USA) was applied to the entire dentin during 20 sec and dried during 5 sec and polymerized for 10 sec. Then, Activa BioActive-Restorative was applied. It was placed with an applicator gun and polymerized for 20 sec after allowing it to settle for three-four sec. Its full polymerization continued for three-four minutes.

Group 3: Two-step total etching procedure was applied for a conventional composite. The dentin surface was etched with acid for 10 sec and then washed for 15 sec, air-dried slightly. Then, the bonding agent was applied to the dentin for 20 sec, dried for 5 sec and polymerized for 10 sec. Then, GC G-aenial Universal posterior composite was applied and polymerized for 20 seconds.

Starch tubes (pasta, Oba Pasta, Turkey) were applied for the application of the restorative material. The tubes were 1 millimeter (mm) in diameter-1 mm in height. One starch tube was placed for each dentin substrate (ND and CAD).[13] Microcylinders were left a room temperature for 24 hours to entire polymerization. During this time, the starch tubes softened, and the softened tubes were removed with a scalpel tip. All specimens were tested immediately. Universal test machines (MOD Dental MIC-101, Esetron Smart Robototechnologies, Turkey) were used to calculate micro shear bond strength. This machine utilizes a chisel-shaped metal blade inserted parallel to the dentin surface into the composite-dentin interface at crosshead speed of 0.5 mm/min until the stick's failure.[14]

The micro shear bond strength (μ-SBS) was calculated to the equation:

$s=p/\pi.r^2$

(s=μ-SBS (MPa), p=load at sample failure (N), π=3.14, r=radius of bonded sample (mm^2)).

Each fractured stick was controlled using a stereomicroscope (Leica Microsystems, Germany) at 50× magnification and was classified into the following failure types:

Type 1: Adhesive link between dentin and material or cohesive in adhesive

Type 2: Cohesive in dentin or resin

Type 3: Mixed.[15]

Statistical Analysis

The normality of the data was controlled with the Kolmogorov-Smirnov test. Two-way ANOVA was used. One-way ANOVA was applied to show the significant difference and a Student t-test was applied to determine the bond strength values among the tested restorative material bonded to normal or caries affected dentin. In all statistical analyses, the level of significance was determined as 95%. All statistical analyses were finalized using SPSS Statistics 23.

Results

The mean±standart deviation of all experimental group is shown in the tables (Table 2-3). No significant difference was observed between test values of each group neither for normal dentin (p:0.065; $p > 0.05$) nor for caries-affected dentin (p:0.110; $p > 0.05$).

No significant difference was observed between μ-SBS values of the ND and CAD subgroup for any of the groups (Act p:0.933; Act B p:0.684; GC p:0.863; $p>0.05$). According to pairwise comparison results, μ-SBS Act B and μ-SBS GC values are higher than μ-SBS Act values.

Regarding the percentages of observed failure modes, adhesive failure dominated the other failure modes (adhesive at dentin side) (Figure 1).

Discussion

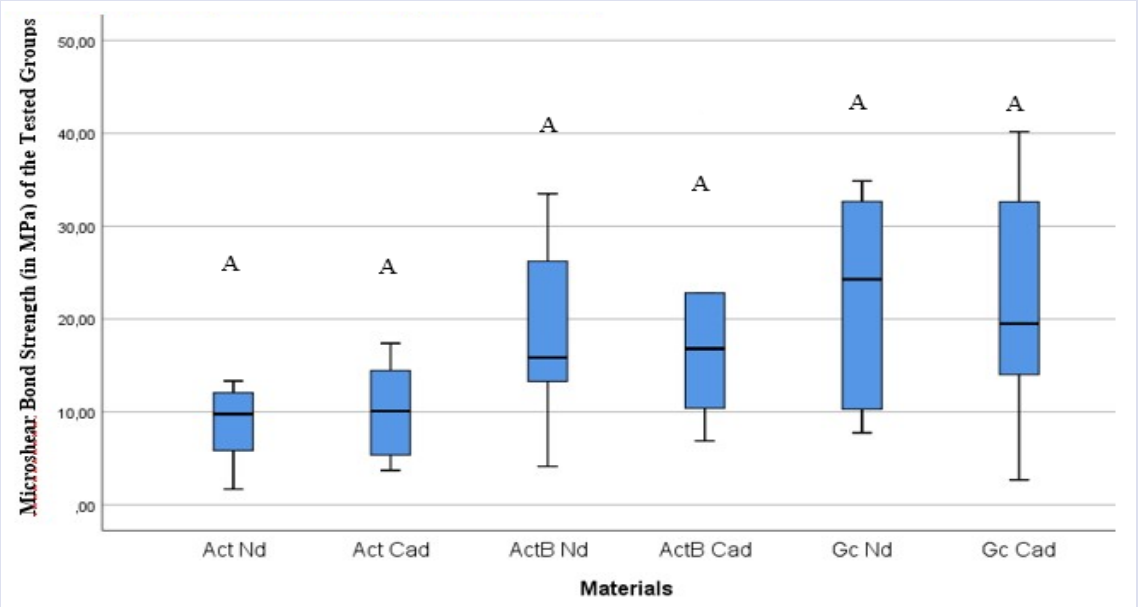
A relatively new material character in restorative dentistry is bioactivity. Bioactive materials can be used with or without an adhesive agent. How Activa bonds to the dentin are not fully clear. This bonding is thought to be a combination of chemical interaction, and micromechanical infiltration. The ionic interaction between the carboxyl group of the material and the hydroxyapatite of the tooth provides chemical bonding. The self-etch feature of the material creates porosity on the tooth surface, also creates surface roughness in the dentin, and these provide micromechanical bonding.[16] The manufacturer suggested the use of acid and expressed the dentin-bonding procedure as optional. Therefore, in this study, the bonding of Activa to dentin with acid and applying acid + bond as compared with the bonding of a conventional composite.

Table 2. μ-SBS (in MPa) of the Tested Materials

	Materials			
	Activa Bio-Active Restorative only acid (Act)	Activa Bio-Active Restorative acid+bond (Act B)	GC posterior acid+bond	P-value
ND (Normal dentin)	10.42±7.82[A] (Ptf/tnt:3/10)	18.24±10.21[A] (Ptf/tnt:0/10)	22.55±10.90[A] (Ptf/tnt:0/10)	0.065
CAD (Caries-affected dentin)	10.11± 5.52[A] (Ptf/tnt:3/10)	20.48±13.74[A] (Ptf/tnt:0/10)	21.66±11.75[A] (Ptf/tnt:0/10)	0.110
P-value	0.933	0.684	0.863	

Ptf/tnt: Pre-test failures/total number of tested samples. [A] Same superscript uppercase letter denotes an insignificant difference.

Table 3. Boxplot For µ-SBS (in MPa) of the tested materials. Median and 25–75% quartiles are displayed within the boxes.



[A] Same superscript uppercase letter denotes an insignificant difference

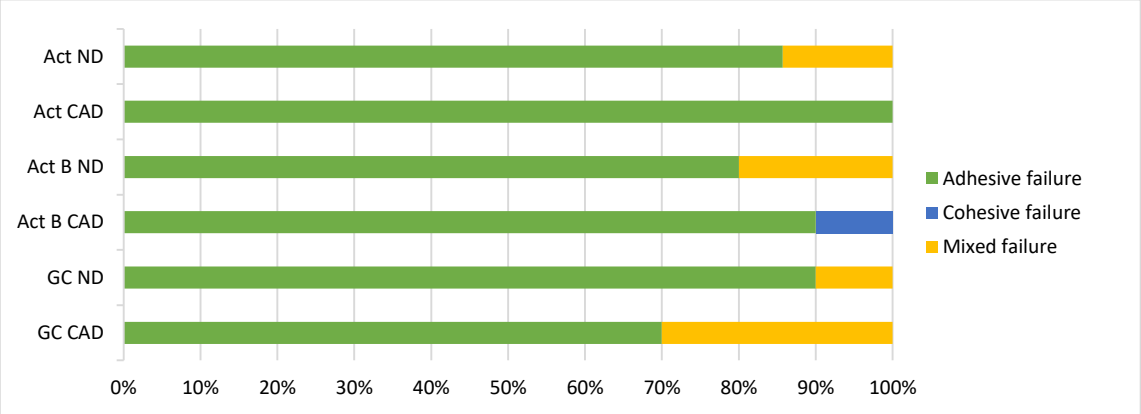


Figure 1. Percentage of failure modes in groups.

The current laboratory-based study is built on the hypothesis that Activa Bio-Active Restorative (Act) without bonding agents shows adequate bond strength in CAD and ND compared to conventional composites. The suggested hypothesis is not rejected.

The durability and adhesive strength evaluation of Activa and conventional composite was performed using the µ-SBS test as it is simple and easy to apply with broad acceptability and cost-effectiveness.[17]

Restoration prior to surface treatment on the enamel and dentin plays a major role in increasing the durability of resin-based restorations. With the introduction of the etching technique by Bonoucore is considered the most effective method to improve adhesive bonding for conditioning the tooth surface.[18] The dentin surface which was conditioned with the total-etch technique and conventional composite applied to show the highest bond strength in ND and CAD (22.55± 10.90 MPa, 21.66± 11.75 MPa, respectively) compared to all test groups, as expected. However, no significant difference was detected between the groups. This outcome can be explained using 37% phosphoric acid, which completely dissolves the smear layer and demineralizes intratubular and peritubular dentin and that would lead to hybridization and resin tags formation.[19]

Initially, in the current study, only Act-applied specimens were prepared without any preliminary preparation on the dentin. However, since all sample composites placed in this group were dropped, the group had to be excluded from the sample space. Thereupon, by following the manufacturer's instructions, Activa composite with 37.5% phosphoric acid was applied instead. There were only 3 failures in the acid-applied groups. These losses are quite significant for a material where the manufacturer promises a strong resin-hydroxyapatite complex and low microleakage.[20] However, no significant bond strength difference was noticed between the Act group in which we applied only

acid and the Act B groups in which we applied acid and bond. However, higher micro shear bond strength results were obtained from Act B groups which we applied bond. Although the bond application on flat dentin surfaces did not show a significant difference, it was found to be applicable. François *et al.* in one of their 2021 article argued that Act had poor self-adhesion to normal dentin without surface pretreatment (4.4 MPa) even though the manufacturer claimed that Act provides high micromechanical and chemical adhesion.[15] Another finding of François's study is that all-new resin-containing fluoride-releasing materials applied to dentin with an adhesive agent have higher μ-SBS values.[21] The findings of the current study are also consistent with Benetti *et al*.'s findings. They also observed losses on the non-pretreatment surface of the enamel and obtained the highest bond strength results in the acidified group. The loss of the material on the dentin surfaces was experienced in group that did not undergo pretreatment and group that were only etched. Thus, measurement of these samples could not be made. On the other hand, the Act group applied to the dentin with acid+bond did not differ significantly from the control group. However, it is claimed that in hemi spherically shaped cavities where dentin has not been pretreated, the fact that the material is surrounded by enamel has a positive effect on bonding. However, in cases where the cavity borders are placed on the dentin, if the dentin has not been pre-treated, losses are higher in restorations due to the large volumetric shrinkage of Active.[3] The reason why we experienced loss in our samples may be that we are on the flat dentin surface. Again, the Benetti study showed that the bond to enamel was higher. [3] François *et al*. stated that in 2021, the manufacturer's instructions wrote that bonding agent application is mandatory in cavities with low retention, and it can be used optionally in retentive cavities. The reason why we experienced sample losses in our study may be our non-retentive cavity, flat dentin.[21] In the light of these findings, the use of bonding agents is supported, especially if Activa is to be applied in non-retentive cavities.

Caries-affected and caries-free dentin affects the bond between the restorative material and dentin. Because caries affected dentin are exposed to increased collagenolytic activity, the connection with the restorative material deteriorates.[22] The use of only caries-free tooth surfaces to investigate material-dentin bonding *in vitro* studies doesn't entirely mimic the clinic.[23] Although some research has been carried out on bonding strength of both normal and caries affected dentin for some bonding systems and dental composite[24,25], no studies have been found which assess the bond strength of Activa to ND and CAD. Therefore, we aimed to test this condition. In the present study, all examined groups did not show significant difference bond strength to normal and caries-affected dentin. The reason for this may be the occlusion of the tubules due to the precipitation of calcium phosphate crystals in the carious-affected dentin. These precipitates may affect the chemical binding of ions in Activa with hydroxyapatite crystals in dentin. This may explain why the tested material content recorded different bond strengths in studies on normal and caries-affected dentin.[26,27]

Ozduman *et al*. evaluated the bond strength of indirect pulp-coating materials to caries-affected dentin on dentin surfaces disinfected with and without chlorhexidine. Tricalcium silicate-based materials, whether disinfected with chlorhexidine or not, have been found to have lower shear bond strength than resin-modified glass-ionomer bioactive cement (Activa Bioactive).[28]

Besides these *in vitro* studies, van Dijken *et al***.** did in randomized controlled clinical trials, an annual failure rate of 24.1% was found in Activa restorations in which were applied only after phosphoric acid gel etching. The main causes of restoration losses were postoperative symptoms, secondary caries, and loss of restoration. It was concluded that this loss rate was unacceptable in Class II cavities and further studies using adhesive ought to be repeated.[12] Alrahlah showed bioactive materials acceptable flexural and diametral tensile strength. However, the hardness was below the expected value. They found that Activa Bulk Fill is a potential material for dentin replacement, but a restorative material must be applied over Activa.[8]

The percentage of adhesive failure between the restorative material and the dentin surface was very high. However, there was no significant difference between failure groups.

The limitation of the study:

This *in vitro* research was not performed using the standard method of thermocycling with cyclic loading to simulate the intraoral environment for checking the microleakage at the tooth- restoration interface. Mode of failure analysis information by taking SEM images is also important in micro shear tests. However, further research by conducting *in vivo* studies could authenticate these results.

Conclusions

It was found that the bond strength of the materials evaluated was not significantly influenced by applying additional bonding agents in Activa Bio-Active Restorative composites in sound dentin and caries affected dentin. However, an optional bond application can also be made, especially in dentin cavities. Further *in vivo* studies should be conducted.

Acknowledgments

The authors would like to thank Zeynep Sonkaya for her valuable help.

Conflict of Interests

The authors declare that they have no conflict of interest.

Funding Statement

The authors received no financial support for the research, authorship, and/or publication of this article.

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