Evaluation of the Bond Strength of Resin-Modified Glass Ionomer Enhanced with Bioactive Glass to Composite Resin with Different Dental Adhesive Systems

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OBJECTIVE: To investigate the bond strength of resin-modified glass ionomer enhanced with bioactive glass (Activa BioActive-Base/Liner) to composite resin using different dental adhesive systems.
STUDY DESIGN: In this study, Activa BioActive-Base/Liner (ABA/BL) was placed in cylindrical cavities formed in acrylic blocks. In blocks divided into 6 groups according to the adhesive system to be applied, two-step etch-and-rinse Gluma 2 Bond (Heraeus Kulzer, Germany), one-step self-etch Gluma Self Etch (Heraeus Kulzer), universal system Gluma Universal (Heraeus Kulzer), two-step self-etch Clearfil SE Protect (Kuraray, Japan), one-step self-etch Clearfil S³ Bond Plus (Kuraray), and universal system Clearfil S³ Bond Universal (Kuraray) adhesive systems were applied on ABA/BL. After composite resin (3M ESPE Filtek Ultimate) was applied to the prepared surfaces, the specimens were placed in a universal test device and shear bond strength test was determined. Fracture types were evaluated using a stereomicroscope and scanning electron microscope. Data were analyzed by Shapiro-Wilk, two-way ANOVA, Kruskal-Wallis, and Post-Hoc Multiple Comparisons tests.
RESULTS: In terms of bond strength values, the highest bond value was seen in the two-step self-etch (Clearfil SE Protect) group, and the lowest bond strength value was seen in the universal system (Clearfil S3 Bond Universal) group. There was no statistically significant difference between the adhesive agent groups in terms of bond strength values ($p>0.05$).
CONCLUSION: It is thought that choosing the two-step self-etch technique as an adhesive system when resin-modified glass ionomer enhanced with bioactive glass (ABA/BL) is used as the pulp capping/base material will be more appropriate in terms of bond strength. (Anal Quant Cytopathol Histpathol 2021;43:235–241)

Keywords: adhesive systems, bioactive materials, bond strength, cariostatic agents, composite resins, dental materials, fluorides, glass ionomer, glass ionomer cements, materials testing, vital pulp therapy.

One of the most important factors for teeth to stay in the oral environment for a longer time is the presence of a healthy vital pulp. Due to caries, vital pulp can be exposed mechanically during tooth preparation or in traumatic injuries. In order to maintain the vitality of the pulp that has been exposed or affected for such reasons and the func-

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tion of the tooth in the mouth, vital pulp therapies are recommended, and pulp capping treatment is the most applied vital pulp therapy procedure. The aim of pulp capping treatment is to stimulate pulp healing by inducing the pulp tissue for reparative dentin formation. Therefore, bioactive materials have an important role in the treatment of vital pulp. Bioactive material in restorative dentistry is defined as "the material that forms the surface layer of an apatite-like structure in the presence of an inorganic phosphate solution." Examples of bioactive materials used to provide remineralization through the formation of inorganic mineral-like substances in demineralized dentin include materials such as calcium hydroxide, mineral trioxide aggregate, calcium silicate compounds, and bioactive glasses. These materials are frequently used in vital pulp treatments.[1-3]

Calcium hydroxide is a pulp capping material that has been used in dentistry for many years and accepted as the gold standard.[4] However, it has disadvantages such as poor resistance to pressure, high solubility, and poor adhesion to dentine and resin-based materials.[5] Therefore, it is observed that calcium hydroxide is replaced by a new generation materials such as calcium silicate materials and resin-modified glass ionomers (RMGIC), which can achieve more predictable clinical results.[6]

Glass ionomers (GIC) were introduced as dental filling material by Wilson and Kent in the 1970s. GIC has advantages such as chemical bonding to dental tissue, showing biocompatible properties, and fluoride release. However, it has disadvantages such as showing poor aesthetic properties, long setting time, and low mechanical and bond strength.[7] To improve the physical and chemical properties of traditional GICs, resin-modified glass ionomers (RMGIC) have been introduced.[8]

GIC is used as a base material, especially under composite resin restorations, due to its chemical bonding properties to enamel and dentin.[9] In addition, it is recommended for use as a liner in indirect pulp capping procedures due to its similar clinical efficacy with current pulp capping materials.[10] However, it is thought that the use of bioactive liner/base material under composite resin restorations is reported to be biologically well tolerated by the pulp tissue[11] and has higher remineralization as compared to glass ionomer cements.[12]

Activa BioActive-Base/Liner (Pulpdent, USA) was launched in 2014, claiming that it has strength, aesthetics, and physical properties as good as composite resin and has more calcium, phosphate, and fluoride release and recharging as compared to glass ionomer. The company markets this product as "light-cured resin-modified calcium silicate" (RMCS), which combines the properties of both composite resin and GIC. Although it claims to be biocompatible, the manufacturer recommends using Activa BioActive-Base/Liner only in indirect pulp capping cases.[6] In a study of 3 restorative materials using a resin-based composite (Z100, 3M-ESPE), a resin-modified glass ionomer cement (Vitremer, 3M-ESPE), and a bioactive material (Activa Bioactive-Restorative, Pulpdent), it was stated that the bioactive material takes up fluoride and releases it again, and this can provide inhibition of caries at the restoration margins.[13]

Thanks to the bioactive properties of the Activa BioActive material, it has been suggested that the material reacts to pH cycles and these properties play an active role in the release and recharging of significant amounts of calcium, phosphate, and fluoride. In a study, the Activa material, similar to MTA, Biodentine, and TheraCal LC, has been reported to exhibit the potential to stimulate mineralization and release Ca^{+} and OH^{-} ions (in ionically reinforced conditions).[14] BioActive-Base/Liner can lead to pulp toxicity due to the release of free monomers from incomplete polymerization.[15] However, in a recent *in vivo* study it was concluded that ABA/BL showed better biocompatibility and tissue improvement in rat subcutaneous tissues as compared to calcium silicate-based materials.[16] It is thought that its reparative potential in pulp capping procedures or its effects on vital pulp will emerge as *in vitro* and *in vivo* studies are conducted.

The bonding between the pulp capping material and the restorative material is one of the factors that maintain the vitality and function of the tooth.[17] Today, composite resins are used most frequently as permanent filling material due to its aesthetic properties. The bond between composite resin and pulp capping material is provided by adhesive agents. Generally used adhesive systems are etch-and-rinse and self-etch systems.

The most common method used to evaluate the adhesion properties of restorative materials *in vitro* is bond strength tests, and the most preferred bond strength measurement method is the shear bond test.[18]

In a literature research we found no study that

includes the bond strength of Activa BioActive-Base/Liner, which is marketed as a resin-modified glass ionomer enhanced with bioactive glass to composite resin. Our aim in this study is to investigate that Activa BioActive-Base/Liner shows better bond strength to composite resin, regardless of the use of etch-and-rinse, self-etch, and universal systems.

Materials and Methods

Forty-two cube-shaped acrylic blocks of 2×2 cm in dimension were obtained. Cylindrical cavities of 4 mm in diameter and 2 mm in depth were formed in the middle of the acrylic blocks, and Activa BioActive-Base/Liner material was applied using a syringe to completely cover the cavities according to the manufacturer's instructions, and polymerized for 20 seconds with an LED light device (Woodpecker Led-G, China). The prepared specimens were divided into 6 subgroups. The adhesive agent was applied to each subgroup according to the manufacturer's instructions (Table I). The

Table I *Materials Used in the Study*

Material	Contents	Lot no.	Application stages
ACTIVA BioACTIVE-base/liner (Pulpdent)	Diurethane dimethacrylate. Bis (2-(methacryloyloxy) ethyl) phosphate, barium glass, ionomer glass, polyacrylic acid/maleic acid copolymer, dual-cure chemistry, sodium fluoride, colorants	190418	Two-paste system dispensed directly from an automix syringe. 3 setting mechanisms: - self-cure - light-cure (20 s) - acid-base reaction
Gluma 2 Bond (Heraeus Kulzer)	Acid: 35% orthophosphoric acid. Adhesive: methacrylate, ethanol, fillers, polymerization initiators, glutaraldehyde	K010553	Two-step ER adhesive technique 1. Apply 35% phosphoric acid etchant 15 s 2. Rinse for 15 s, dry for 10 s 3. Apply adhesive for 15 s, dry with air for 5 s, light-polymerize 20s
Gluma Self Etch (Heraeus Kulzer)	2-HEMA, 4-META, acetone, polymerization initiators	010912	One-step self-etch adhesive technique 1. Apply adhesive for 20 s 2. Gentle air stream 5–10 s 3. Light-polymerize 20 s
Gluma Bond Universal (Heraeus Kulzer)	4-META, methacrylate monomer, acetone, methacryloyloxidecyl dihydrogen phosphate (MDP)	K010034	Universal adhesive system (SE technique) 1. Apply adhesive for 20 s 2. Gentle air stream 3. Light-polymerize 10 s
Clearfil SE Protect Bond (Kuraray Noritake Dental)	Primer: 10-MDP, 12-MDPB, 2-HEMA, hydrophilic dimethacrylate, water. Adhesive: 10-MDP, Bis-GMA, 2-HEMA, hydrophobic dimethacrylate, dl camphorquinone, N, N-diethanol-p-tolidine, silanized colloidal silica, surface treated sodium fluoride	6D0081	Two-step SE adhesive technique 1. Apply primer for 20 s 2. Dry with mild air flow 3. Apply bond and mild air for 5 s 4. Light cure for 10 s
Clearfil S^3 Bond Plus (Kuraray Dental)	10-MDP, HEMA Bis-GMA, colloidal silica, camphoroquinone hydrophilic aliphatic dimethacrylate, hydrophobic aliphatic dimethacrylate sodium fluoride, accelerators, initiators, ethanol, water	6L0082	One-step self-etch adhesive technique 1. Apply adhesive for 20 s 2. Gentle air stream 5 s 3. Light-polymerize 10 s
Clearfil S^3 Bond Universal (Kuraray Dental)	Bis-GMA, HEMA, ethanol, 10-MDP, hydrophilic aliphatic dimethacrylate, colloidal silica, camphoroquinone, silane coating agent, accelerators, initiators, water	3D0040	Universal adhesive system (SE technique) 1. Apply adhesive for 10 s 2. Gentle air stream 5 s 3. Light-polymerize 10 s
Filtek Ultimate Universal Restorative (3M ESPE)	Silica and zirconium particles in free form as filler. Bis-GMA% 1–10, UDMA, TEGDMA% <1 PEGDMA% <5 BIS-EMA	N873246	Apply 2 mm by incremental technique and light-polymerize 20 s

polymerization of the adhesives was carried out with an LED light device.

Composite Resin Application

Following the polymerization of the adhesives, composite resin (Filtek Ultimate Universal Restorative (3M ESPE, St. Paul, Minnesota, USA) was applied with the help of a cylindrical plastic tube with a 2 mm inner diameter and 2 mm height on the pulp capping materials and polymerized for 20 seconds with an LED light device. The composite resin was polymerized for another 20 seconds after the plastic tube was cut and removed with a scalpel tip.

Finally, the specimens were kept ready for the shear bond test by remaining for 24 hours in an oven (37°C, 100% humid environment).

Shear Bond Test

A universal test device (Instron, Lloyd Instruments, UK) was used for shear bond strength measurements, and separating force was applied at a speed of 1 mm per minute. The force value of the composite specimen at the moment of separation from the surface of the capping material was obtained in Newton and converted to mega pascal (Mpa) by dividing the surface area in the rupture area.

Examination of Fracture Surfaces

All specimens were examined under ×10 magnification stereomicroscope (Leica MZ 12; Leica Microsystems GmbH, Wetzlar, Germany) to determine the fracture type, and fracture types were grouped as adhesive, cohesive, and mixed. Fracture at the interface of the composite/pulp capping material was classified as adhesive fracture, cohesive fracture in the composite layer, or capping material, and mixed fractures with adhesive and cohesive fractures. In addition, images of the specimens were taken in scanning electron microscope (SEM) (Zeiss Gemini 500) under vacuum at ×200, ×500, ×1000, and ×2000 magnifications.

Statistical Analysis

The data obtained in the study were analyzed with IBM SPSS Statistics Version 21 package program. Shapiro-Wilk test was used because of the unit numbers while investigating the status of variables coming from the normal distribution. 0.05 was used as the significance level while interpreting the results. It was stated that there is a significant difference when $p<0.05$, and there is no significant difference if $p>0.05$. Two-way ANOVA was used to evaluate the effects of independent variables on the dependent variable. While examining the differences between groups, the Kruskal-Wallis H test was used because the variables did not come from the normal distribution.

Results

The analysis results on the differences between the adhesive agent groups in terms of the bond strength values of Activa BioActive-Base/Liner are shown in Table II. There was no statistically significant difference between the adhesive agent groups in terms of the bond strength values of Activa BioActive-Base/Liner ($p>0.05$). The highest bond strength was seen in the Clearfil SE Protect group, and the lowest value was seen in the Clearfil S^3 Bond Universal group.

All fracture types were detected in the Activa BioActive-Base/Liner material except for adhesive fracture. Cohesive fractures which occurred in the composite were observed mostly (Table III) (Figures 1 and 2).

Discussion

The aim of this study was to examine the bond

Table II *Bond Strength Values Between Activa BioActive-Base/Liner and Adhesive Agent Groups*

		Shear bond strength value (MPa)					Kruskal-Wallis H test		
Adhesive agent group	**N**	**Mean**	**Median**	**Min**	**Max**	**ss**	**Mean rank**	**H**	**p Value**
Gluma 2 Bond	7	12.54	10.07	2.95	24.39	7.90	19.29	6.889	0.229
Gluma Self Etch	7	19.30	12.98	9.30	41.85	12.20	27.14		
Gluma Universal	7	12.45	13.54	5.96	20.08	5.89	20.29		
Clearfil SE Protect	7	**19.83**	13.48	9.27	42.95	12.70	28.14		
Clearfil S^3 Plus	7	13.93	10.45	3.98	25.57	8.85	20.71		
Clearfil S^3 Universal	7	**9.44**	7.57	1.12	26.01	7.97	13.43		
Total	42	14.58	11.80	1.12	42.95	9.74			

Table III *Fracture Types After the Shear Bond Test of Activa BioActive-Base/Liner*

	Adhesive agent	Adhesive	Cohesive-capping material	Cohesive-composite	Mixed	Total
Bioactive Base/Liner	Gluma 2 Bond	0	0	**5**	2	7
	Gluma Self Etch	0	2	1	**4**	7
	Gluma Universal	0	0	**5**	2	7
	Clearfil SE Protect	0	0	**4**	3	7
	Clearfil S^3 Plus	0	0	3	**4**	7
	Clearfil S^3 Universal	0	0	**6**	1	7

strength of resin-modified glass ionomer (Activa BioActive-Base/Liner) enhanced with bioactive glass to composite resin with different dental adhesive systems. Good adhesion of the materials used as the pulp capping/base material to the restorative materials is an important factor for preserving the vitality of the tooth.[17] Although the Activa BioActive-Base/Liner material, which has been developed in recent years, attracts attention with its biological and physical properties, there are few studies on the material. The manufacturer recommends the use of a Bioactive liner/base material under the indirect pulp capping of Class I, II, III, and V restorations without pulp involvement and all composite resin and amalgam restorations.

In many studies it is recommended to be used instead of traditional glass ionomer cements in the sandwich technique because the resin content in RMGIC increases the bond strength to the composite resin.[19] The bond strength of RMGIC to composite resin varies depending on the adhesive technique used, and it has been suggested that self-etch systems show better bonding than etch-and-rinse systems.[20] In our study, two-step and one-step self-etch adhesive systems showed higher bond strength than two-step etch-and-rinse and the universal systems we applied with the self-etch technique.

Alzraikat et al[21] evaluated the bond strength of ProRoot MTA, TheraCal LC, and Fuji IX, a traditional capsule glass ionomer, using different adhesive systems (etch-and-rinse and one-step self-etch) in their study. They reported that Fuji IX material showed lower bond strength to composite resin than did TheraCal LC and statistically higher than ProRoot MTA. In addition, groups using one-step self-etch technique in Fuji IX showed higher bonding values than groups using the etch-and-rinse technique. It is thought that material-dependent factors such as viscosity affect the adaptation of GICs to the tooth surface and bond strength. It has been suggested that low adaptability may lead to partial defects in the restoration margins and small gaps that may affect the bond strength values of the composite.[22]

Deepa et al[23] investigated the bond strength of TheraCal LC, Biodentine, and resin-modified glass ionomers to composite resin using the universal adhesive system. The highest bond strength was seen in RMGIC, and TheraCal LC showed a bond strength value close to RMGIC. In Biodentine groups, a statistically significantly lower bond strength value was found as compared to the other

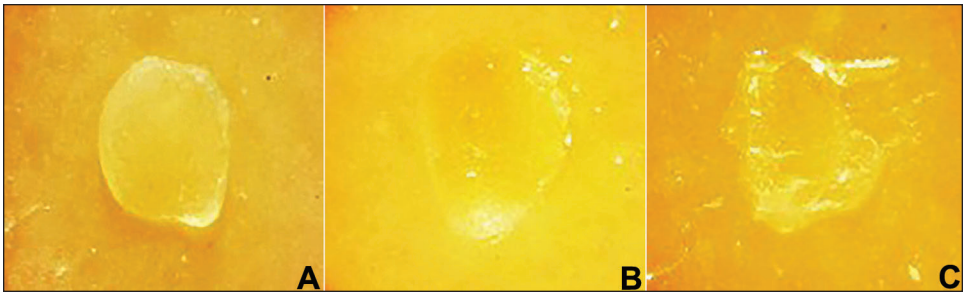


Figure 1
(A) Cohesive fracture in composite resin, (B) cohesive fracture in capping material, and (C) mixed fracture.

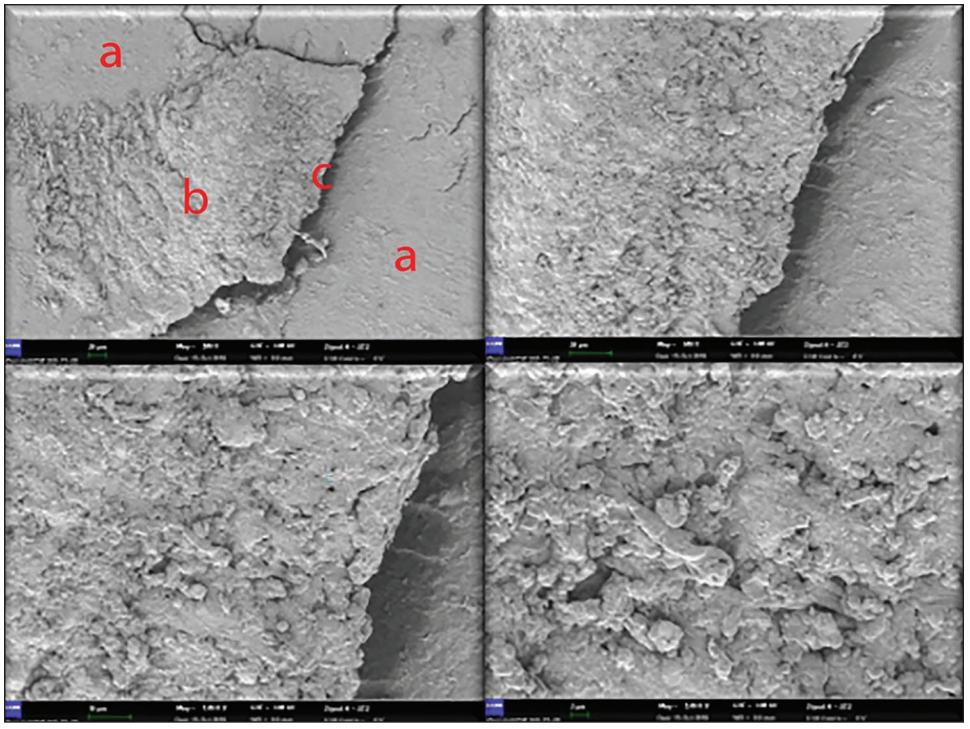


Figure 2
Mixed fracture SEM image (×200, ×500, ×1000, and ×2000 magnifications). a = Activa BioActive-Base/Liner, b = composite resin, c = fracture line.

groups. It has been suggested that TheraCal LC and RMGIC materials may have shown higher bond strength to the composite resin due to their resin content.

Karadas and Atıcı investigated the bond strength of pulp capping materials (Biodentine, TheraCal LC, Ultra-Blend plus, Calcimol LC, ApaCal ART, EQUIA Forte, and Ionoseal) to dentin. EQUIA Forte (traditional hybrid glass ionomer) was stated to have a statistically higher bonding strength than the other materials, and it was concluded that Ionoseal material (RMGIC) had the lowest bonding strength value to dentine.[24]

Assuming that Activa BioActive products have been developed as an RMGIC, it is expected to be self-adhesive to dental tissues.[25] Related to this issue, it has been reported that the initial failure rate was high in the 1-year clinical follow-up of posterior restorations performed with ACTIVA BioACTIVE Restorative in adults.[26] The main reasons for the failure described in that randomized study were loss of restoration, postoperative sensitivity, and incidence of secondary caries.

BioActive Restorative was placed on the tooth tissue without any surface treatment, by etching with acid and applying self-etch adhesive, and the bond strength was investigated. Separation of the material from the tooth was observed even before the specimens were subjected to the shear test. It was found that the bond strength of the specimens applied with self-etch adhesive increased.[25]

In our study, the most common cohesive fractures (formed within the composite resin) and mixed fractures were observed, respectively. Many studies have reported that when a high rate of cohesive and mixed fractures is observed, it can be concluded that a good bond is obtained between the two materials.[27]

Conclusion

It was concluded that if composite resin is planned to be placed on Activa BioActive-Base/Liner as a permanent restoration, the two-stage self-etch technique may be preferred.

Informed Consent

Written informed consent was obtained from the patients who participated in this study.

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