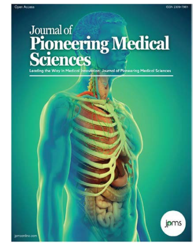

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Effect of Bioactive Adhesives on Shear Bond Strength at the Enamel-Orthodontic Bracket Interface

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Abstract **Objectives:** This study aimed to assess the bonding effectiveness of bioactive restorative materials when used for orthodontic bracket placement. Specifically, it evaluated whether these materials could provide shear bond strength (SBS) comparable to that of conventional resin-based adhesives, while also minimizing residual adhesive and preserving enamel integrity after bracket debonding. **Methods:** Forty-five extracted, caries-free premolars were randomly assigned into three main groups (n=15) based on the restorative material used: ACTIVA Bioactive-Restorative (AB), Beautifil II (BF), and Transbond XT (TB XT) — with TB XT serving as the control. Each group was further subdivided into three adhesive primer subgroups (n=5): FL-Bond II, BeautiBond Xtreme, and Transbond XT Primer. After standardized enamel cleaning and etching procedures, orthodontic metal brackets were bonded to the tooth surfaces and light-cured as per manufacturer guidelines. Specimens were stored in distilled water at 37°C for 24 hours before being subjected to shear bond strength testing using a universal testing machine. Following debonding, residual adhesive was evaluated under a stereomicroscope and scored using the modified Adhesive Remnant Index (ARI). **Results:** Two-way ANOVA revealed a statistically significant interaction between the type of adhesive and restorative material on SBS values ($p<0.001$). Among all combinations, the group using FL-Bond II with Transbond XT exhibited the highest mean SBS (19.44±3.90 MPa), while the Beautifil II group paired with BeautiBond Xtreme showed the lowest bond strength. ARI score analysis demonstrated significant differences across groups, with FL-Bond II generally resulting in minimal adhesive remnants on the enamel surface. **Conclusion:** The combination of FL-Bond II adhesive with conventional Transbond XT composite resin exhibited superior shear bond strength and favorable ARI scores, making it an optimal choice for orthodontic bracket bonding. ACTIVA Bioactive, when used with suitable primers, demonstrated promising results in terms of bond strength and enamel preservation, indicating its potential as an alternative to conventional systems. However, further in vivo and long-term clinical studies are warranted to validate these findings under dynamic oral conditions.

Key Words Bioactive materials, shear bond strength, orthodontic adhesives, enamel integrity, adhesive remnant index, ACTIVA, giomer, FL-Bond II

INTRODUCTION

Orthodontic treatments involving fixed brackets frequently result in white spot lesions (WSLs) in approximately 50% of patients. This occurrence is primarily attributed to difficulties in maintaining oral hygiene and plaque accumulation around the brackets. To mitigate the risk of enamel demineralization, orthodontists commonly implement preventive strategies. These strategies include the application of topical fluoride in various forms such as varnish, paste, gel, or rinse coupled with patient education on effective brushing techniques [1]. The presence of fermentable carbohydrates facilitates the proliferation of bacteria, such as *Lactobacillus* and *Streptococcus mutans*, within the plaque. These bacteria produce acids that lower the pH of the tooth surface, creating a more acidic environment that accelerates plaque formation and encourages the colonization of additional aciduric bacteria [2]. Moreover, utilizing fluoride-releasing bonding agents can significantly reduce the reliance on patient compliance with oral hygiene practices [3].

The introduction of composite resin as a bonding agent for orthodontic brackets in the 1970s marked a transformative advancement in the field. This development not only enhanced the overall effectiveness of orthodontic treatments but also allowed for more precise placement of brackets [4]. Although conventional adhesives are accessible the market requires materials which integrate mechanical retention together with enamel-protective characteristics and fluoride release properties. In recent decades, the bonding of orthodontic attachments using specialized adhesives has become a critical step in clinical orthodontic therapy. This advancement has opened new avenues for the development of innovative adhesive materials and improved bonding techniques.

Currently, orthodontics employs three primary categories of bonding materials: self-adhesive cements, resin-modified glass ionomer cements (RMGICs), and composite resins with various priming systems. Composite resins are the most frequently utilized bonding materials, owing to their user-friendly nature and satisfactory bonding strength, which yields positive clinical outcomes. However, they are not without limitations. These include the absence of fluoride release and the potential for WSLs to form around brackets after treatment completion, as well as challenges related to bonding in specific clinical scenarios.

Studies investigating the initial properties of giomers have demonstrated that their mechanical characteristics are comparable to those of resin composites. Giomers are characterized as "intelligent particles," developed to integrate the aesthetic and mechanical strength of resin composites with the fluoride-releasing capabilities of glass ionomers.

This material utilizes glass ionomer technology in conjunction with a pre-activated S-PRG surface. When exposed to polyacrylic acid, the fluoraminosilicate particles undergo a chemical reaction, facilitating their incorporation into the resin matrix, which enables a continuous release of fluoride upon interaction with saliva in the oral environment. Furthermore, these ions not only inhibit bacterial adhesion to dental surfaces but also serve as a buffering system, neutralizing the acids produced by these microorganisms [5-7].

The primary objective of fixed orthodontic treatment is to achieve sufficient bond strength between brackets and tooth surfaces. The bond must be strong enough to withstand the various stresses encountered during treatment while also being easy to remove without damaging the enamel [8, 9]. The advent of bioactive dental adhesives represents a significant advancement in dentistry. These adhesives are designed to interact positively with biological tissues, providing therapeutic benefits that extend beyond simple adhesion [10]. Bioactive materials are formulated with various bioactive components, including calcium phosphate, fluoride, and antimicrobials. These components promote remineralization, support dentin regeneration, and help reduce the risk of secondary caries [10-12]. Fluoride-releasing orthodontic bonding materials, such as GICs and RMGICs, were developed with the specific aim of preventing WSLs. While these bonding agents have demonstrated fluoride release in vivo, the literature reports conflicting findings regarding their anticariogenic properties. Additionally, the bond strengths of GICs and RMGICs tend to be significantly lower than those of conventional resins [13-15].

Numerous factors influence orthodontic bond strength, including the choice of bonding material, enamel etching procedures, bracket design, and type of adhesive agent employed. Additionally, variables related to the teeth such as tooth type and fluorosis are documented, along with environmental factors that include blood, saliva, and moisture contamination [8]. While many researchers endorse the use of RMGICs for bonding orthodontic brackets, the findings in the literature remain inconsistent. Clinical investigations have indicated a higher incidence of debonding with RMGICs compared to combinations of primer and composite resin, underscoring the complexity of bonding outcomes in orthodontics [16-18].

Research has shown that bond failures are significantly more prevalent with RMGICs compared to resin-based composite bonding systems. ACTIVA BioACTIVE-RESTORATIVE, a recently introduced enhanced RMGIC from Pulpdent Corporation (Watertown, MA, USA), integrates the properties of traditional RMGICs with a modified resin matrix, resulting in improved resilience and enhanced physical properties [19]. While there are currently no studies specifically examining the bracket bond strength of ACTIVA, the material has exhibited flexural, compressive, and tensile strengths that are comparable to those of composite materials and markedly superior to those of glass ionomer cements and RMGICs [20, 21]. The research addresses an essential knowledge gap through performance evaluation of new bioactive adhesive materials (such as ACTIVA) which lack sufficient orthodontic application data. Apart from antibacterial action the ion release function contributes to dental remineralization.

The exploration of novel adhesive materials and methodologies is crucial as orthodontics continues to evolve. This study aimed to evaluate the shear bond strength (SBS) of a bioactive material used for bonding orthodontic brackets, comparing its performance to that of traditional resin-based adhesives.

Null Hypothesis:

- Bioactive restorative materials do not significantly affect shear bond strength (SBS) when compared with conventional resin-based adhesives
- The type of adhesive system used does not significantly influence SBS across different restorative materials. Additionally, the rationale for enamel preservation expectations has been expanded in the Introduction

MATERIALS AND METHOD

Materials used in this study and application steps were described in Table 1.

Study Setting and Sample

This study involved 45 caries-free, extracted premolar teeth. The teeth were ethically obtained from oral surgery and orthodontic clinics, following approval from the ethics committee of King Abdulaziz University and adherence to international ethical guidelines. Teeth with cavities, developmental defects, fillings, or cracks were excluded from the study. Extracted teeth were stored in 0.1% thymol solution at 4°C and used within 3 months of extraction. The sample size calculation was conducted using the G*Power 3.1.9.7 software. Effect size was determined based on the means and standard deviations of the groups. The significance level (α) was set at 0.05, while the desired power of the study was established at 0.95. Based on these parameters, the calculated power of the study was found to be 95%, indicating that a total sample size of 45 samples was necessary.

Specimen Preparation

The specimens were categorized into three groups (n = 15) based on the materials used: ACTIVA Bioactive-Restorative (AB) (Pulpdent, USA), Beautifil II (BF) (Shofu, Japan), and Transbond XT (TB XT) (3M Unitek, USA), the latter serving as the control group. Each group was further divided into three subgroups (n=5) according to the adhesive systems employed: BeautiBond Xtreme (Shofu, Japan), FL-Bond II (Shofu, Japan), and Transbond XT Primer (3M Unitek, USA) as the control. The enamel surfaces were cleaned using a rotary instrument with a rubber cup and oil-free pumice.

Subsequently, the enamel was etched with 37% phosphoric acid (N-Etch, Ivoclar Vivadent) for 30 seconds, followed by rinsing and drying for 10 seconds. Prime and adhesive were applied with a micro-brush, and light curing was performed using an LED curing unit (Elipar FreeLight 2, 3M/ESPE, St. Paul, MN, USA) in accordance with the manufacturer's instructions. The dental orthodontic metal bracket (Victory Series, 3M Unitek Co., Monrovia, CA, USA) was securely placed on the midsection of the buccal surface of the teeth using a bracket-holding tweezer to ensure precise alignment. Any excess bonding agent was meticulously removed prior to light curing. The bracket base was measured by a digital caliper and the surface area was calculated and found to be 12 mm^2.

All bonding procedures were conducted by the same operator, and the specimens were subsequently stored in distilled water at 37°C for 24 hours to prevent the tooth enamel from drying out until the test process. Bracket positioning was standardized using a positioning jig to ensure uniformity across all specimens. Figure 1 summarizes specimen grouping, and Table 1 shows the composition and application of each material.

Shear Bond Strength Test

Following 24 hrs and prior to the debonding test, the crowns of the specimens were separated from their roots using a slow-speed saw under coolant. The specimens were then embedded in acrylic resin (Meditray, Promedica Dental Material GmbH, Germany) within a phenolic ring, ensuring that the labial surfaces of each crown were oriented perpendicular to the base of the mold using a mounting jig. To assess the SBS, the specimens were fixed to a universal testing device (Instron 5944, Instron Corporation, Canton, MA, USA) with their facial surfaces positioned parallel to the applied force. A shear force was exerted at the bracket-tooth interface through an occluso-gingival load. Each test was recorded via a computer connected to the apparatus, at a crosshead speed of 0.5 mm/min applied to the bracket-enamel interface until fracture occurred. The recorded failure load (N) was then converted to megapascals by dividing the force (N) by the surface area of the bracket base, which is 12 mm². Post-debonding, both brackets and teeth were examined under a stereomicroscope (25 ×; Olympus, Tokyo, Japan), at ×10 magnification to evaluate any residual adhesive. Following debonding, the amount of resin material

Table 1: Materials used in this study and its applications

Materials	Composition	Manufacturer	Application
Adhesives			
Beauti-Bond xtreme	Acetone, water, Bis-GMA, carboxylic acid monomer, TEGDMA, organophosphate monomer, acid resistant silane coupling agent.	Shofu, Kyoto, Japan	Apply on tooth surface. Gentle air blows for 3 seconds then strong air blow. Light cure 5 s for LED.
FL-BOND II	Bottle1 (Primer): methacrylic adhesive monomer,ethanol, water. Bottle 2 (Bonding Agent): urethane dimethacrylate, 2-hydroxyethyl methacrylate, alumino-fluoro- borosilicate glass (S-PRGS), triethylene glycol dimethacrylate.	Shofu, Kyoto, Japan	Apply Primer for 10 s. Then air dry 5s. Apply Bonding agent. light cure the for 10 s for LED.
Transbond XT Primer	37% Phosphoric acid Bis-GMA, TEGDMA.	3M Unitek, USA	Air dry tooth surface, apply thin uniform coat of Primer to be bonded.
Restorative Materials			
ACTIVA Bioactive (reinforced compomer)	Mixture of other methacrylates and diurethane with amorphous silica, modified polyacrylic acid, and sodium fluoride.	Pulpdent, Watertown, MA, USA	Bulk fill, allow to self-cure for 2 min, and light cure for 20 s.
Beautifil II (Giomer)	BISGMA, TEGDMA, aluminum oxide, silica, aluminofluoro-borosilicate glass filler, pre-reacted glass-ionomer filler, camphoroquinone	Shofu, Kyoto, Japan	place a thin layer of 1mm thickness, and light cure for 10 s.
Transbond XT (Resin)	Silane-treated quartz, Bis-GMA, dichlorodimethyl silane, silane-treated silica, diphenyliodonium hexafluorophosphate.	3M Unitek, USA	place a thin layer of the adhesive, and light cure for 10 s.

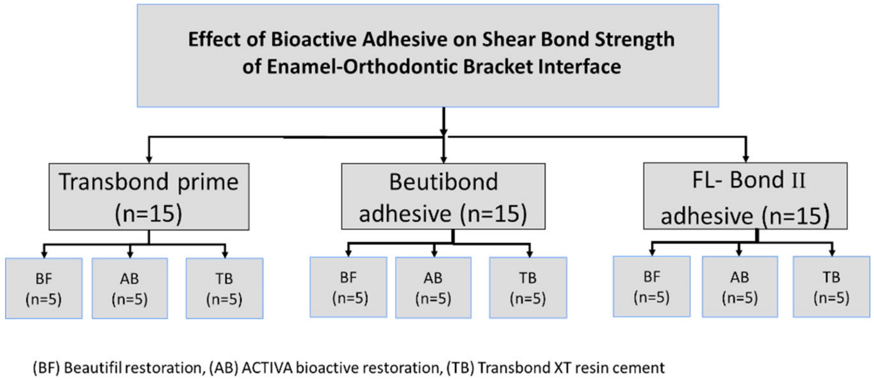

Figure 1: Flow chart of study design.

Table 2: Adhesive remnant index (ARI) score and criterion.

ARI Score	Criterion
0	No adhesive left on the tooth
1	Less than half of the adhesive left on the tooth
2	More than half of the adhesive left on the tooth
3	All adhesive left on the tooth, with a distinct impression on the bracket mesh
4	Enamel fracture

Table 3: Two-way ANOVA

Source	Sum of squares (II)	Df	Mean square	f-value	p-value
Restorative material	362.91	2	181.46	37.21	<0.001*
Adhesive	152.53	2	76.27	15.64	<0.001*
Material * adhesive	314.71	4	78.68	16.13	<0.001*

df degree of freedom, *Significant (p<0.05)

adhering to the enamel surface was assessed and scored using the modified Adhesive Remnant Index (ARI), according to the original description of Artun and Bergland [22] (Table 2).

Statistical Analysis

Ordinal data were presented as frequency and percentage values and were analysed using Kruskal-Wallis's test, followed by Dunn's post hoc test. Numerical data were presented as mean and standard deviation (SD) values. They were tested for normality and variance homogeneity by viewing distribution and using Shapiro-Wilk's and Levene's tests, respectively. The data were normally distributed with homogenous variances across different variables. They were analyzed using a two-way ANOVA test. The comparisons of simple effects were made using the error term of the two-way model. P-values were adjusted for multiple comparisons using the False Discovery Rate (FDR) method. The significance level was set at p<0.05 within all tests. Statistical analysis was performed with R statistical analysis software version 4.4.1 for Windows[1].

RESULTS

The two-way ANOVA results presented in Table 3 showed that there was a significant interaction between both tested variables (p<0.001). The simple effects comparisons presented in Table 4 showed that for all types of adhesives, there was a significant difference between different restorative materials (p<0.001). For the Transbond prime, pairwise comparisons showed TB XT and AB to have significantly higher values than BF (p<0.001). For Beutibond, all pairwise comparisons were statistically significant, with TB XT having the highest value followed by AB and BF having the lowest (p<0.001). For FL-Bond II, they were also all statistically significant, with TB XT having the highest value, but BF had a higher value than AB (p<0.001).

Within all restorative materials, there was a significant difference between adhesive types (p<0.001). For BF and TB XT, pairwise comparisons showed FL- Bond II to have significantly higher values than other adhesives (p<0.001). In contrast, for AB, they showed Transbond prime to have significantly higher values than other adhesives (p<0.001). Summary statistics for shear bond strength values are presented in Figure 2 and 3.

The results of the failure modes distribution are presented in Table 3 and Figure 4. For Transbond prime, there was no significant difference in ARI scores measured in different restorative materials (p = 0.062). The difference was statistically significant for other adhesives, with BF and TB XT having significantly higher scores than AB (p = 0.004). There was a significant difference in ARI scores measured in different adhesives within different materials. For BF, Beutibond, and FL- Bond II had significantly higher scores than Transbond prime. For AB, Transbond prime had significantly higher scores than other adhesives. For TB, Transbond prime and Beutibond had significantly higher scores than FL- Bond II. The effect size for interaction between adhesive and restorative material was large ($\eta^2 = 0.64$), indicating a substantial impact on SBS values. Representative samples were presented to show the difference in ARI scores in Figure 5.

Values with different upper and lowercase superscripts within the same horizontal row and vertical column, respectively, are significantly different, * significant (p<0.05). where (BF) represents Beautifil restoration, (AB) ACTIVA bioactive restoration, and TB Transbond XT resin cement.

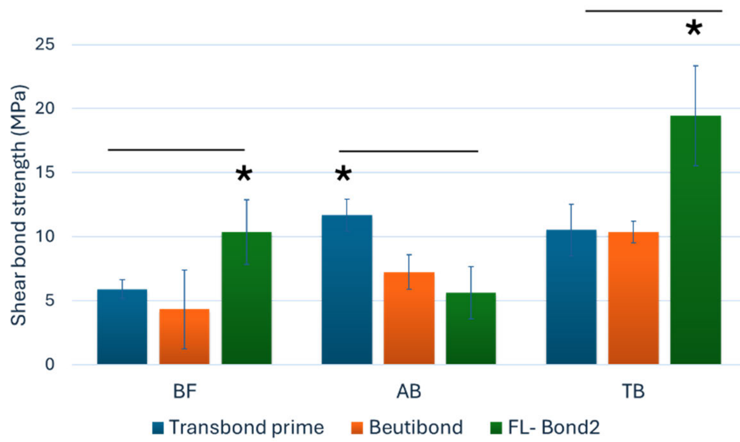

Figure 2: Bar chart showing mean and standard deviation values (error bars) of shear bond strength (MPa) of all restorative groups; where (BF) represents Beautifil restoration, (AB) ACTIVA bioactive restoration, and TB Transbond XT resin cement. * Indicate significance, line indicates the adhesive groups per each restorative material

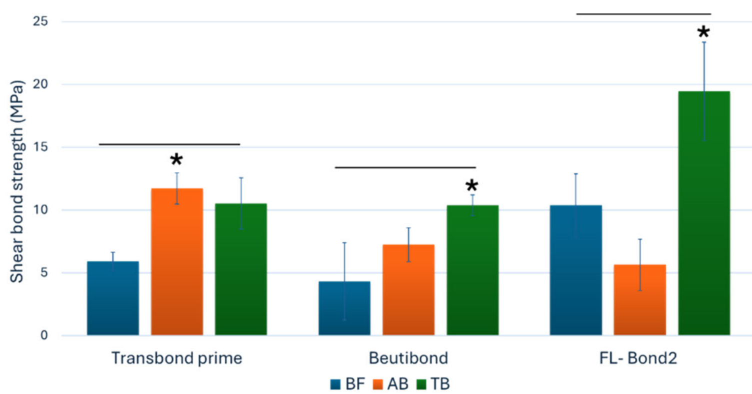

Figure 3: Bar chart showing mean and standard deviation values (error bars) of shear bond strength (MPa) of all adhesive groups; where (BF) represents Beautifil restoration, (AB) ACTIVA bioactive restoration, and TB Transbond XT resin cement. * Indicate significance, line indicates the restoration groups per each adhesive material

Figure 4: Stacked bar chart showing Adhesive remanent index (ARI) distribution, whereas; (BF) represents Beautifil restoration, (AB) ACTIVA bioactive restoration, and TB XT Transbond resin cement

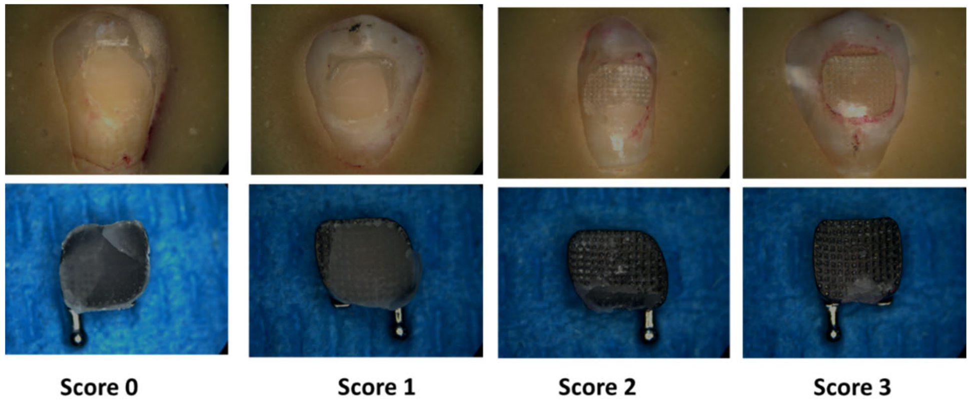

Figure 5: Representative samples displaying various ARI scores were examined using a stereomicroscope (25×; Olympus, Tokyo, Japan) at ×10 magnification.

Table 4: Simple effects comparisons

Adhesive	Shear bond strength (MPa) (Mean±SD)			f-value	p-value
	BF	AB	TB XT		
Transbond prime	5.91±0.74Bb	11.71±3.08Aa	10.52±2.52Ab	9.63	<0.001*
Beutibond	4.32±1.24Cb	7.24±1.35Bb	10.36±2.03Ab	9.36	<0.001*
FL- Bond II	10.36±2.03Ba	5.63±0.83Cb	19.44±3.90Aa	50.48	<0.001*
f-value	10.06	10.18	27.67		
p-value	<0.001*	<0.001*	<0.001*		

DISCUSSION

The introduction of enamel bonding for orthodontic applications in 1965 marked a significant advancement in orthodontic treatment. As highlighted by Owens and Miller, the direct bonding of orthodontic brackets to enamel was made feasible through the pioneering work of Buonocore, Bowen, and Tavas and Watts [23-26]. These researchers played a crucial role in developing the procedures and materials that have established contemporary standards for orthodontic adhesives.

Shear bond strength is influenced by several factors, including the adhesive properties of the bonding materials, the interactions at various interphases such as between the tooth and composite bonding material [27]. Numerous adhesive agents have been developed for bonding orthodontic brackets, with foundational research playing a crucial role in establishing the procedures and materials that define current standards in orthodontic adhesives. Ongoing developments continue to introduce new materials aimed at enhancing the quality of bonds between brackets and both natural teeth and artificial surfaces [28]. In this study, the bond strength of bioactive materials was measursed when bonded with fluoride releasing adhesives compared to conventional adhesive used for orthodontic bracket bonding.

According to the literature, a minimum bond strength of 6-8 MPa is necessary to endure typical orthodontic forces [4]. All materials demonstrated satisfactory SBS except BF group. The findings indicate that the conventional TB XT composite resin bonded with FL-Bond II exhibited the highest bond strength among the tested groups which reject the null hypothesis. This outcome underscores the efficacy of conventional resin-based adhesives, particularly in orthodontic applications where strong, reliable bonding is essential. The superior performance of Transbond XT can be attributed to its formulation with methacrylate-based resins that provide strong adhesion to dental substrates. FL-Bond II achieves its high SBS value from S-PRG fillers that perform dual functions in resistance improvement and fluoride-release alongside acid-buffering ability to minimize WSL risk. When paired with FL-Bond II, which enhances the bonding process, the combination benefits from superior chemical interactions that promote chemical bonding to enamel. FL-Bond II incorporates fluoride containing S-PRG fillers (Surface Pre-Reacted Glass-ionomer) which offer a permanently available protection against secondary caries by an optimised remineralisation of the adjacent hard tooth structure [29]. The ARI results indicated that 80% of the score 1 ratings were associated with this group (Figure 4), correlating well with the SBS data and suggesting that this combination leaves minimal adhesive on the enamel.

The findings of our study are consistent with previous research. Katırcıoğlu and Büyükbayraktar conducted a study comparing the shear bond strengths of Transbond XT, Light Bond, BracePaste®, Nova Compo SF (Imicryl, Konya, Turkey), and Rely A Bond (Reliance, Itasca, USA). They found that the Transbond XT group exhibited a significantly higher bond strength compared to the Nova Compo SF group. However, when Transbond XT was compared to the other groups, no significant differences were observed [30]. Shams et al. assessed three different adhesive agents for shear bond strength (SBS) over two time periods in their study. The brackets were bonded using Transbond XT, BracePaste®, and GoTo (Reliance Orthodontic Products, Itasca, IL, USA) adhesives with Transbond XT primer. After 24 hours of testing, no statistical difference was observed between the GoTo and Transbond XT groups, while the BracePaste® group exhibited a significantly lower bond strength. Additionally, the study found no significant difference in the number of shear strokes between Group 1 and Group 2 [31]. Clinical tests showed that using Transbond Primer with ACTIVA BioACTIVE achieved suitable shear bond strength results. The bioactive components in ACTIVA disperse fluoride and calcium ions through mechanical adhesion which provides dual protective effects to the enamel. The research outcomes hold specific relevance when treating patients with substantial demineralization risks.

Furthermore, our study indicated that using Transbond XT with AB after enamel etching produced significantly greater bond strength compared to the Beautifil composite group. There is a scarcity of data on the shear bond strength of brackets bonded with ACTIVA BioACTIVE-Cement. These results suggest that employing AB alongside conventional Transbond Prime maintains bond strength and may also help in preventing white spot lesions, as previously reported in Saunders, K.'s research. [19]. The ARI index results showed an equal distribution between scores 2 and 3 (40% each), which aligns well with the SBS results.

This investigation took place in an in vitro setting which fails to duplicate the oral condition along with elements like masticatory forces along with saliva and temperature modifications. The behavior of bracket debonding could differ while present inside the human body. Future investigations should conduct clinical tests followed by thermocycling and extended service period testing to validate these laboratory findings in actual patient settings.

CONCLUSIONS

Research shows the use of FL-Bond II with Transbond XT resin composite leads to strong shear bond strength while creating negligible adhesive residue thus qualifying it for routine dental applications. By combining ACTIVA Bioactive with Transbond Primer the product achieved suitable SBS levels and may help protect the enamel material structure.

Clinical trials need to evaluate the oral performance of bioactive materials that combine adhesive properties with fluoride release. Clinicians should choose bonding agents that maintain adequate mechanical strength while protecting the enamel structure according to recommended clinical practices.

Conflicts of Interest

The authors declare no conflicts of interest.

Ethical Statement

All procedures involving human teeth were approved by the Institutional Review Board of King Abdulaziz University (IRB #123-DENT-2024). Informed consent was obtained from all donors prior to tooth extraction in accordance with ethical guidelines.

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