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Shear Bond Strength and Adhesive Remnant Index of ACTIVA a Bioactive Material: An *in-Vitro* Comparative Study

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ABSTRACT

Background & Objective: An optimal connection between enamel surfaces and brackets is necessary for successful fixed orthodontic treatment. The purpose of this study was to assess shear bond strength (SBS) and adhesive remnant index (ARI) of brackets bonded with (ACTIVA) and compare it with other orthodontic adhesives.

Materials & Methods: 64 human upper premolar teeth were randomly divided into four equal groups (16 teeth in each group) according to the type of adhesive used. Then each group was subdivided into two subgroups (8 teeth each), one preserved in deionized water at 37°C for 30 days and one subgroup of each adhesive type was subjected to acid media. Adhesive systems used in the study: Transbond XT group (non-fluoride releasing adhesive), Light Bond group (fluoride releasing adhesive), GC Fuji Ortho LC group (powder and liquid glass ionomer adhesive, and ACTIVA bioactive restorative material group. Using a universal testing machine (1 mm/min), SBS was assessed after 30 days. The ARI was evaluated by using a stereomicroscope with a 10X magnification.

Results: A significant difference ($p<0.05$) between the four tested adhesive systems in SBS in both groups. The mean SBS value in both groups was highest in the Transbond XT group, followed by the ACTIVA group, the Light Bond group, and the GC Fuji Ortho group. Only Fuji Ortho group showed a highly significant difference between the acid challenge and water storage. Regarding the ARI test, ACTIVA has score II in water and score III in acid which means much material is left on the tooth surface.

Conclusion: ACTIVA had a SBS that was higher than Light Bond and GC Fuji ortho LC, however, lower than that of Transbond XT, but still above the clinically acceptable level.

Keywords: Fluoride Releasing Adhesive, Acidic Media, Shear Bond Strength, Bioactive Material, Adhesive Remnant Index, ACTIVA

1. Introduction

An optimal connection between enamel surfaces and brackets is necessary for successful fixed orthodontic treatment (1). Bond failure may be caused by patients applying substantial force to the attachments or by using improper bonding techniques (2). In addition to composite resin, resin-modified glass-ionomer cement (RMGIC) is another material that can be used to bond orthodontic brackets. Some orthodontists favor RMGIC over composite due to its advantages. Fluoride is released and white spot lesions on the enamel's surface are reduced by RMGIC. It is also hydrophilic and has a reasonable range of applications in wet conditions (3).

Despite advances in materials and treatment mechanics, fixed orthodontic appliances still carry a substantial risk of developing white spot lesions (WSLs). The WSL prevalence rang during orthodontic treatment is 13% to 75% (4).

Bioactive materials with properties resembling those of composites and glass ionomer cements have been introduced thanks to ongoing scientific advancements in dental materials. These materials can be beneficial in conservative and pediatric restorative dentistry. Activa Bioactive-Restorative (Pulpdent Corp., Watertown, MA, USA), a new bioactive restorative material, was developed and launched. This substance aims to mimic the physical and chemical properties of natural teeth by combining the strength and aesthetics of composites with the advantages of glass ionomers. The rubberized resin is strong and long-lasting, and it contains reactive glass ionomer fillers that, in addition to their high fluoride content, have high rates of release and recharge of calcium (Ca2+) and phosphate (PO43) ions (5). This material was used as an orthodontic adhesive and can reduce demineralization around brackets (6).

The objective of this study was to evaluate the shear bond strength and adhesive remnant index of this bioactive material and compare it with other orthodontic adhesives.

2. Materials and Methods

2.1 Study design

Comparative in vitro study. This study aimed to evaluate ACTIVA as an orthodontic adhesive in both neutral and acidic environments to mimic oral conditions and compare it with other commonly used adhesives because of its composition of resin and glass ionomer cement

64 healthy permanent upper first premolars with an undamaged buccal surface, no cavities, restorations, cracks, or fluorosis, and no history of chemical treatments. They were kept in a 0.1% (weight/volume) thymol solution for a week, after which they were kept in deionized water until the bonding operations were carried out (7).

The teeth were randomly divided into four equal groups (16 teeth in each group) according to the type of adhesive used. Then each group was subdivided into two subgroups (8 teeth each) and one preserved in deionized water at 37°C for 30 days and one subgroup of each adhesive type was subjected to acid media as seen in Figure 1. Before bonding, the teeth were placed on auto-polymerized acrylic blocks. 64 stainless-steel Discovery® Smart MBT brackets for upper first premolars were utilized (Dentaurum), which have a 10.56 mm2 bonding surface area and 0.022-inch slot size. The buccal tooth surface was polished with non-fluoride-containing pumice for 10 seconds, cleaned and dried for 10 seconds (8).

2.2 Bonding materials and procedures

Bonding materials (Figure 2) include:

- Transbond XT group: adhesive that doesn't release fluoride (3M Unitek, Monrovia, California, USA).
- Light Bond group: (Reliance Orthodontic Products, Itasca, Illinois, USA) is a fluoride-releasing adhesive.
- GC Fuji Ortho LC group: powder and liquid glass ionomer orthodontic adhesive, (GC Company, Tokyo, Japan).
- ACTIVA restorative material group: (Pulpdent Corporation, watertown, USA).

Bonding procedures performed according to the manufacturer's instructions for each type of adhesive as follows:

The enamel surfaces were washed, dried and etched for 30 seconds with 37% phosphoric acid. The etched enamel surfaces were coated with a thin layer of adhesive primer (except GC Fuji Ortho group which does not have primer), which was then polymerized for 30 seconds by an LED light curing device.

The bracket base was coated with adhesive and positioned in the middle of the buccal tooth surface during bonding. Equal pressure was applied during each bracket bonding procedure (200 grams), to obtain a homogeneous adhesive thickness (8). The extra adhesive was removed and the LED light curing unit was used for 40 seconds (10 seconds from each side of the bracket), at a curing intensity of 1500 mw/cm2 (4).

After the bonding processes were completed, the first group of bonded teeth was kept in deionized water in the incubator at 37°C for 30 days with daily refreshment of deionized water to prevent cumulative effects (9, 10). For the acid challenge group, the bonded teeth were kept in deionized water at 37°C for 24 hours. A 500 ml solution of the acidic solution (pH=2.5) was made by gradually adding HCl [1M] to deionized water (Figure 3), by submerging the samples in the acidic solution using a regimen of three sessions per day lasting five minutes each, with equal breaks of two hours between sessions, for 30 days. To simulate the wet oral environment and acidic exposure, the samples were kept in deionized water at 37°C for the remainder of the day. Each storage medium was periodically replaced after each session (10).

2.3 Shear bond strength test

Using a 5 KN load cell and a crosshead speed of 1 mm/min, the Tinius-Olsen universal testing machine was utilized to perform the shear bond strength test after aging operations for 30 days (11). The pressure was applied vertically in the occluso-gingival direction at the enamel-bracket interface from a knife-edge rod (attached inside the upper arm of the universal testing machine) until adhesive failure was detected. The debonding force was measured in Newtons and converted to megapascals (MPa) by dividing it by the surface area of the bracket base (12).

2.4 Estimation of adhesive remnant index

The enamel surface of each tooth and the debonded bracket were examined under a stereomicroscope (Hamilton, Italy) with a 10 X magnification, to determine the primary site of bond failure. Artun and Bergland assigned a score to the location of the bond failure (13) as follows:

0 = No adhesive remained on the tooth surface.

I = Less than 50% adhesive remained on the tooth surface.

II = More than 50% adhesive remained on the tooth surface.

III = All the adhesive has remained on the tooth surface.

2.5 Statistical analysis

SPSS version 19 was used for data collection and analysis. The following statistics were utilized:

2.6 Descriptive statistics

Means, standard deviations, and statistical tables as shown in Table 1.

2.7 Inferential statistics

One-way ANOVA test: to test any statistically significant difference of the SBS among groups. Tukey's HSD test and Games-Howell were used after ANOVA if gave significant differences. A level of significance of $p < 0.05$ was considered for statistical analysis.

Table 1. Descriptive statistics and ANOVA test of the shear bond strength (MPa) of different groups.

Media	Groups	N	Mean (MPa)	SD.	Min. (MPa)	Max. (MPa)	ANOVA	
							F-test	p-value
Water	Transbond XT	8	43.865	8.807	33.08	55.91	36.245	0
	Light Bond	8	25.977	4.208	20.83	34.09		
	GC Fuji Ortho	8	14.041	3.4	10.41	18.93		
	ACTIVA	8	31.11	5.29	23.67	38.35		
Acid	Transbond XT	8	37.156	3.519	32.61	44.5	6.18	0.002
	Light bond	8	30.06	3.73	25.56	35.98		
	GC Fuji Ortho	8	27.616	6.09	19.88	36.93		
	ACTIVA	8	31.931	4.64	24.14	37.27		

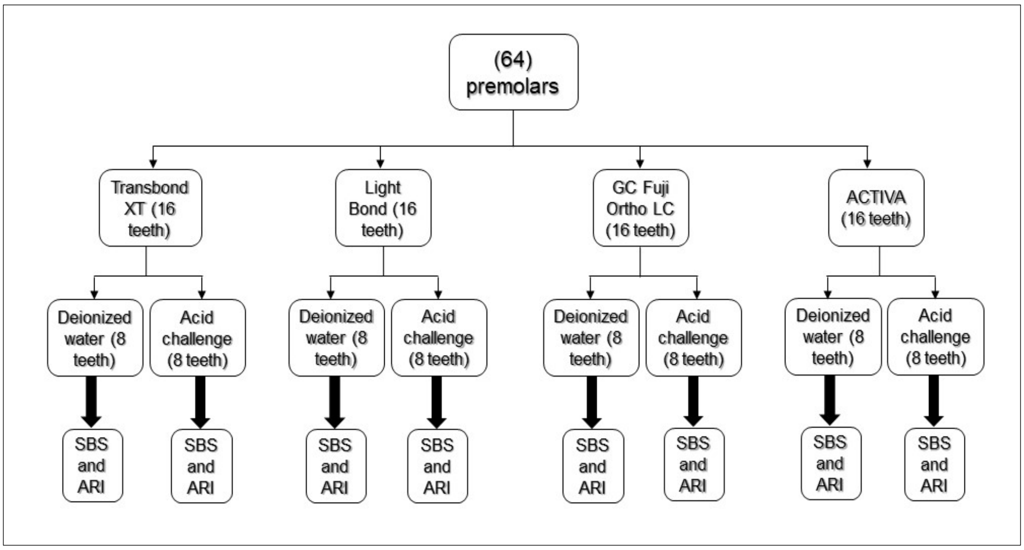

Figure 1. Sample distribution (**Prepared by Authors, 2025**).

(Transbond XT group)

(Light Bond group)

(GC Fuji Ortho LC group)

(ACTIVA group)

Figure 2. Adhesive systems used (**Prepared by Authors, 2025**).

Figure 3. Acid challenge (**Prepared by Authors, 2025**).

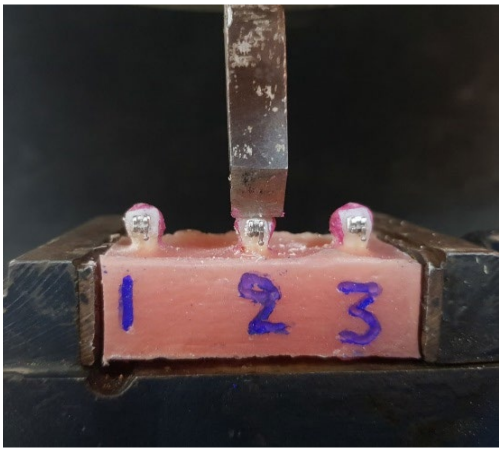

Figure 4. Shear bond strength test (**Prepared by Authors, 2025**).

3. Result

The mean SBS values of the tested materials were higher than the optimal limits suggested by Reynolds (14) which is 5.9 to 7.8 MPa, and thus, sufficient for clinical use. In both groups (water storage and acid challenge) the Transbond XT group had the greatest mean SBS value, followed by ACTIVA group, Light Bond group, and Fuji Ortho group, as shown in Table 1.

One-way analysis of variance (ANOVA) was used to compare the mean SBS differences between the two groups (water storage and acid challenge). Games-Howell and post-hoc Tukey tests were used to compare SBS between groups (Table 2).

The impact of the aging medium (water storage and acid challenge) on the SBS of the four test adhesive systems was determined (Table 3).

Adhesive remnant index (ARI)

The amount of adhesive left on each tooth surface after debonding was measured using the ARI developed by Artun and Bergland (13). The ARI in both groups was compared as shown in Table 4.

Table 5 shows how the ageing media (water storage and acid challenge) affect the distribution of ARI scores for the four test adhesive systems.

Table 2. Comparison of shear bond strength test in both groups by the Post-hoc Games-Howell test and Tukey HSD.

Media	Post hoc test	Groups		Mean Difference	p-value
Water	Games-Howell	Transbond XT	Light bond	17.888	0.002
			GC Fuji Ortho	29.824	0.000
			ACTIVA	12.755	0.021
		Light bond	GC Fuji Ortho	11.936	0.000
			ACTIVA	-5.132	0.188
		GC Fuji Ortho	ACTIVA	-17.068	0.000
Acid	Tukey HSD	Transbond XT	Light bond	7.096	0.022
			GC Fuji Ortho	9.540	0.002
			ACTIVA	5.225	0.130
		Light bond	GC Fuji Ortho	2.443	0.716
			ACTIVA	-1.871	0.848
		GC Fuji Ortho	ACTIVA	-4.315	0.262

Table 3. Independent t-test are used to compare the effects of ageing media on the mean shear bond strengths of the four test adhesive systems.

Groups	Comparison		
	mean difference	t-test	p-value
Transbond XT	6.709	2.001	0.065
Light bond	4.083	-2.054	0.059
GC Fuji Ortho	13.575	-5.505	0.000
ACTIVA	0.821	-0.330	0.746

Table 4. Descriptive statistics and Kruskal Wallis Test of water storage and acid challenge groups.

Media	Groups	Descriptive statistics					Group difference Kruskal Wallis Test		
		N	Median	Mean Rank	Min.	Max.	X2	df	p-value
Water	Transbond XT	7	1	13.64	1	3	12.849	3	0.005
	Light bond	7	1	7.86	0	2			
	GC Fuji Ortho	7	2	14.93	1	3			
	ACTIVA	8	3	22.50	2	3			
Acid	Transbond XT	8	1	10.69	0	2	14.167	3	0.003
	Light bond	8	1	9.63	0	2			
	GC Fuji Ortho	7	2	20.86	1	3			
	ACTIVA	7	2	22.36	1	3			

Table 5. Distribution of ARI scores in both aging media.

Groups	Media	Descriptive statistics			Comparison	
		N	Median	Mean Rank	MWU test	p-value
Transbond XT	Water	7	1	9.93	14.5	0.077
	Acid	8	1	6.31		
Light bond	Water	7	1	8.86	22	0.428
	Acid	8	1	7.25		
GC Fuji Ortho	Water	7	2	7.14	22	0.735
	Acid	7	2	7.86		
ACTIVA	Water	8	3	9.75	14	0.068
	Acid	7	2	6.00		

4. Discussions

The purpose of this study was to assess the bioactive material (ACTIVA) if used as an orthodontic adhesive, its SBS and ARI when compared with other orthodontic adhesive systems, after subjecting them to two different ageing media (the water storage and the acid challenge) for 30 days, which can simulate the oral environment. Because of ACTIVA's composition of resin and glass ionomer cement we compare it with other adhesives like Transbond XT which is non-fluoride releasing adhesive resin and is the standard adhesive in orthodontics and Light Bond adhesive and GC ortho Fuji(RMGIC) which are fluoride-releasing adhesives.

The sample used in this study included human upper premolars extracted for orthodontic treatment because they were readily available and to mimic the results of clinical situations **(15)**, as the use of bovine teeth as a substrate for shear bond strength testing is questionable and their enamel structure differs significantly **(16)**.

A higher mean SBS does not always imply improved clinical performance **(17)**. Therefore, rather than obtaining the highest possible value, the most crucial issue regarding SBS in clinical orthodontic practice is achieving a sufficient bond strength that facilitates the safe detachment of fixed appliance components **(18)**. The mean SBS values in this study were greater than the clinically appropriate SBS range (5.9 to 7.8 MPa) by Reynolds **(19)** in all groups (in water storage and acid challenge), indicating that all adhesives can withstand shear stress to a sufficient degree.

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)**.

According to the results of the SBS test, there were statistically significant differences among the tested adhesive systems in both groups. In both challenges, the ACTIVA group was less than the Transbond XT group but more than the Light Bond group and the Fuji Ortho GC group. This may be because of its composition of the Bioactive ionic resin matrix, Shock-absorbing rubberized resin component and reactive ionomer glass fillers **(21)** which means it has features of both composite and glass ionomers. Yet, our study results did not match with Sayed et al **(22)**, who found that ACTIVA SBS was significantly higher than that of the group Transbond XT. This disagreement may be due to using a self-etching adhesive with Transbond XT or the specimens were subjected to thermocycling. Thermocycling was performed on the samples, which involved 500 cycles in distilled water between 5 and 55°C. The teeth were immersed for 20 seconds during each time, with bath intervals ranging from 5 to 10 seconds.

In both ageing groups, Transbond XT group had a highest mean value of SBS, while Fuji Ortho group had the least mean value of SBS among the tested adhesive. Still, above clinically acceptable SBS, this result may be explained by the use of 37% phosphoric acid instead of polyacrylic acid conditioner to condition the enamel surfaces in Fuji Ortho group, which would result in a rougher enamel surface and, as a result, increase the bond strength **(23-25)**. Additionally, our findings support those of Ruwiaee and Alhuwaizi **(26)**, which found a significant difference in SBS between Fuji Ortho adhesive and Transbond XT adhesive, with showing significantly higher values across all Transbond XT of thermal cycle quantities.

The light Bond group had lower mean SBS values than the Transbond XT group, and the difference between these two groups was significant in both ageing media. These findings matched those of Salih and Al-Janabi **(27)**, who looked at the SBS of brackets made of metal and ceramic that had been bonded using various bonding agents. Subsequently it was found that the SBS values were lower for brackets made of metal and ceramic when Light Bond was used as opposed to Transbond XT. However, our results study did not agree with those of Hatf and Al-Khatieeb **(28)**, who found no significant difference in mean SBS between Light Bond and Transbond XT. This discrepancy may be caused by the fact that only 3 hours were spent incubating the specimens before debonding and that no ageing strategy was used in their study.

The result of this study also disagreed with Al-Khatieeb et al **(29)** who found that the highest SBS was obtained with Light Bond followed by Transbond.This may be due to using human third molars teeth, and self-etching primer SEP (Transbond Plus ™) was used with Transbond but the difference is not significant.

The current study demonstrated that there was a highly significant difference between Light Bond group and Fuji Ortho group in water storage, these findings were in agreement with those of Mohammed and Rafeeq **(30)**, who discovered that the SBS was higher in Light Bond than Fuji Ortho LC but that the difference was not statistically significant. This may be explained by the fact that both adhesive systems released fluoride, that debonding was performed after 24 hours, or that they used bovine mandibular permanent incisors in their study (Bond strengths to human enamel were superior to those to bovine teeth **(31)**.

The present study observed that the difference between Fuji Ortho group and ACTIVA group was highly significant in water storage, this may happen as a result of ACTIVA's unique composition of bioactive ionic resin, patented rubberized resin, and patented bioactive ionomer glass **(5)**.

The current study showed that there were no significant differences in the SBS among the tested adhesive systems in both ageing groups only in the Fuji Ortho group there was a significant difference. The fact that found no significant differences between the experimental and control groups after evaluating the SBS of bonded teeth stored in acidic soft drinks and artificial saliva supports these findings (32). This agreement may be explained by the use of a nearly identical acid challenge protocol. Pulgaonkar and Chitra (33) assess the impact of Coca-Cola®, Sprite®, and Maaza® on adhesive residues and SBS under orthodontic brackets. When exposed to Coca-Cola, orthodontic brackets exhibited a substantial decrease in SBS. This discrepancy could be due to a distinct immersion technique (15 minutes, three times per day, separated by 8 hours intervals; this immersion cycle was repeated for 15 days).

Artun and Bergland (13) developed the ARI method to measure the amount of adhesive remaining on the tooth surface after debonding. With a score range of 0 to 3, it was one of the most widely used indices in orthodontic adhesive testing (34-36). This index was used to calculate the impact of different aging media (water storage and acid challenge) on the amount of adhesive that remained on the enamel surface after 30 days and other types of adhesive systems were employed in this study. Furthermore, the analysis of ARI in the present study showed that the difference between the Transbond XT group and the ACTIVA group was significant in the water storage group and the acid challenge group, while the difference between Light Bond group and the ACTIVA group was highly significant in the water storage group and significant in the acid challenge group.

Pseiner et al (28) discovered that Transbond XT and light bond did not significantly differ from one another as in our investigation. The failure occurred primarily at the bracket adhesive interface, as indicated by ARI ratings of 2 and 3. As a result, the cohesive strength of the adhesive and its bond to enamel were stronger than their respective counterparts' bonds to the bracket base. It would be advantageous if there were less adhesive left over after debonding since it would speed up the cleanup process after treatment and reduce the risk of iatrogenic enamel loss Artun and Bergland (13).

Regarding the effect of aging media, the current study showed no significant differences in the frequency of ARI scores among the tested adhesives in water storage and acid challenge groups. This conclusion is reinforced by (32), who discovered that the distribution of adhesive remains among the studied specimens was unaffected by the acidic beverages. Additionally, Normando and Capelozza Filho (33) demonstrated that there were no differences in ARI scores between experimental groups of soft acidic drinks and the control group (artificial saliva).

5. Conclusion

ACTIVA had a SBS that was higher than Light Bond and GC Fuji Ortho LC), however lower than Transbond XT, but still above the clinically acceptable level, so that ACTIVA can be used as an orthodontic adhesive. According to the result of ARI, more than half of the ACTIVA material remains on the tooth surface and this complicates its removal at the end of treatment.

6. Declarations

6.1 Acknowledgments

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6.2 Ethical Considerations

The study was approved by the College of Dentistry/University of Baghdad's local ethics commission. Project number, 596422, Ref. number: 596.

6.3 Authors' Contributions

All authors contributed in current research and reviewed and approved the final version of the manuscript.

6.4 Conflict of Interest

The authors declare that there is no conflict of interest regarding the publication of this study.

6.5 Fund or Financial Support

This research received no specific grant from any funding agency in the public, commercial, or not-for-profit sectors.

6.6 Using Artificial Intelligence Tools (AI Tools)

No artificial intelligence or AI-assisted tools were used in the preparation of this manuscript.

7. Publisher's Note

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