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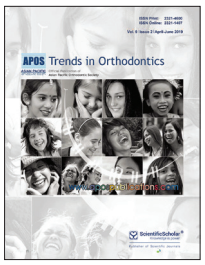

Original Article

Evaluation of shear bond strength of orthodontic adhesives with integrated primer: A comparative study

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ABSTRACT

Objectives: The study aimed to compare the shear bond strength (SBS) of three orthodontic adhesives with integrated primer, with those of self-etching and conventional bonding adhesives.

Material and Methods: One hundred extracted premolars were randomly allocated to five groups. Brackets were bonded using three adhesives with integrated primer (GC Ortho Connect™, Biofix, and Orthocem). A group of brackets was bonded with the conventional bonding procedure (Transbond XT) and one group was bonded with a self-etching primer (Transbond™ Plus). All samples were subjected to thermal aging (5000 cycles: At 5°C and 55°C media). The compressive strength test was performed and the maximum load when the bracket detached was recorded. The amount of residual adhesive (ARI) remaining on the tooth surface was assessed visually.

Results: Significantly higher SBS values were recorded with the conventional technique (14.01 ± 5.79 megapascals [MPa]), compared to other groups except for GC Ortho Connect™ (11.86 ± 3.83 MPa). There was no significant difference between the self-etching group and the groups containing integrated primer samples. However, one of the adhesives with integrated primer presented SBS values near-slightly below the limit considered clinically successful (7.65 ± 3.71 MPa). The ARI scores varied between adhesive groups; GC Ortho Connect™ and the self-etching primer samples showed statistically significantly higher scores compared to the three other groups.

Conclusion: Clinically successful bonding values were achieved with two out of three different adhesives with integrated primer. One of the adhesives with integrated primer provided a similar SBS value to the tri-step conventional bonding procedure. ARI scores varied independently from SBS values.

Keywords: Orthodontic adhesives, Integrated primer, Shear bond strength, Amount of residual adhesive

INTRODUCTION

Correct bonding has a significant impact on the successful progress of the treatment since it is possible to apply orthodontic forces only as long as the attachments remain attached to the teeth. Many *in vivo* and *in vitro* studies evaluated the mechanical and physical properties of the orthodontic bonding systems and researchers are still excited about the development of adhesive systems showing high performance, easy-to-apply, and cheap.[1,2]

The desire of the manufacturers to ease the application of the adhesives and to shorten the bonding procedure to improve patient-physician comfort, lead to the development of a new system eliminating the use of primers. According to the manufacturers, when using these brand-new adhesives with integrated primer, there is no longer a need to apply any primer on the tooth, as it is already incorporated into the paste. A simple etching of the enamel followed by drying is known to be enough

to bond a fixed attachment. However, there are few numbers of studies evaluating the bonding performance of these materials and as far as we know, there is no publication yet evaluating the bond strength of these materials in a comparative study.

Our study aims to compare the bond strength of three different newly developed orthodontic adhesives with integrated primer, with those of self-etch and conventional bonding techniques. The null hypothesis was that there would be no difference in the shear bond strength (SBS) of the adhesives with integrated primer and the conventional two- and three-step bonding procedures.

MATERIAL AND METHODS

The total number of teeth necessary for the study was calculated according to 80% of power, 3 units of difference at a 95% confidence interval. According to our analysis, at least 17 samples for each group were found to be necessary, but the number of teeth for each group was determined as $n = 20$ considering possible technical errors.

Following the approval of the study by the Bezmialem Vakif University Ethics Committee (Project number 04/57), the patients referred for premolar extraction were informed about the study, and the teeth of those who agreed and signed the informed consent form were collected. The extractions were performed for orthodontic, prosthetic, or periodontal reasons from individuals aged between 19 and 33 years old. The teeth collected for the study had no caries, no fluorosis, and no noticeable enamel cracks or restorations. The teeth were kept in distilled water and the water was changed every 2 days. Teeth whose holding period in distilled water was longer than 4 weeks were not involved in the experiment.

The teeth surfaces were cleaned with pumice containing no fluoride and water. The teeth were rinsed with pressured water and dried. The enamel surface was etched with GC Ortho Etching Gel (37% phosphoric acid, GC EUROPE N.V., Belgium.) for 30 s, rinsed, and dried except for the self-etching primer group. Upper first premolar metal brackets were bonded using different adhesives (Primer integrated adhesives: GC Ortho Connect™ [GC EUROPE N.V., Belgium], Biofix [Biodinamica Dental Products LDA, Portugal], Orthocem [FGM Produtos Odontológicos LDA, Brasil], Self-etching primer + Adhesive: Transbond XT Light Cure Adhesive [3M Unitek, USA], Acid etching + primer + Adhesive system: Transbond XT Light Cure Adhesive [3M Unitek, USA], [Table 1]) to 100 premolars (0.018-inch slot, Roth prescription, Mini Master Series, American Orthodontics, USA). The bracket base was coated with adhesive, then the brackets were gently pressed towards the teeth and the excess adhesive was removed using an explorer. Each bracket was irradiated from the right and left sides for 3 s each with a high-performance LED curing light unit (VALO Cordless in Extra Power mode [irradiance of 3200 mW/cm^2), Ultradent, USA) (wavelength 385–515 nm). The light intensity was controlled before use for each group to ensure consistency of energy output with a radiometer (Demetron LED Radiometer, Kerr Corp., USA). All samples were kept in a dark container filled with distilled water for 24 h for complete polymerization.

Bracket-bonded teeth were subjected to a thermal cycle (5000 cycles; SD Mechatronik Thermocycler, SD MECHATRONIK GMBH, Germany) to mimic the oral environment. The samples were held at 5°C and 55°C distilled water media for 30 s. The transfer time between baths was set to be 10 s. After thermal aging, the teeth were embedded in acrylic blocks and the samples were subjected to an SBS test in Shimadzu Universal Tester (Shimadzu Co., Japan) in the occlusogingival direction with the head speed set at 0.5 mm/min.

The maximum load at the moment when the bracket detached from the tooth surface was recorded. The values obtained in Newton were converted into megapascal unit (Force [Newton]/bracket surface area [10.8 mm^2] = megapascals [MPa] [amount of force per unit area]).

The amount of residual adhesive (ARI) remaining on the tooth surface was assessed visually by a single researcher (Z.B.Y.) according to Artun and Bergland classification;[3]

- *0: There is no adhesive on the tooth surface
- 1: Less than50% of the adhesive remained on the tooth surface
- 2: More than 50% of the adhesive remained on the tooth surface
- 3: All adhesives remained on the tooth surface.

Statistical analysis

The statistical analysis was performed using SPSS (SPSS PC Version 22.0; SPSS Inc., USA) with a 95% confidence level. The distribution of the data was evaluated with the Shapiro–Wilk test.

The normally distributed data were evaluated with a one-way ANOVA test ($P < 0.05$). The difference between groups was analyzed with the Bonferroni test ($P < 0.05$). Fisher's exact test was used to compare the ARI scores between adhesive groups ($P < 0.05$).

RESULTS

The bond strength obtained with the adhesives used in the study differed between groups ($P < 0.001$) [Table 2]. Significant differences were observed between groups according to the results of the Bonferroni test [Table 3].

Significantly higher shear bond values were recorded in Group 5, which includes adhesive samples used in conventional bonding technique, compared to all other

Table 1: The study groups and the tested adhesives; (1), (2), (3) the groups with integrated primer, (4) the group with self-etching adhesive, and (5) the conventional 3-step-bonding group.

Group	Acid	Primer	Adhesive Name	Composition		
				Resin	Filler	Additional contents
1 (*n*=20)	GC Ortho Etching Gel (37%) (GC EUROPE N.V., Belgium)	-	GC Ortho Connect™ (GC EUROPE N.V., Belgium)	Bis MEPP Dimethacrylate Phosphoric ester monomer	Ba Glass filler Silicon dioxide Silica tine particle (38%)	Photoinitiator Fluorescent agent
2 (*n*=20)	GC Ortho Etching Gel (37%) (GC EUROPE N.V., Belgium)	-	Biofix (Biodinamica Dental Products LDA, Portugal)	Biphenyl A glicidilmethecrylate (34,78%), dimethacrylate zurethane ethylene	Inorganic filler (41, 52%)	Titanium dioxide, sodium fluoride, and catalyst
3 (*n*=20)	GC Ortho Etching Gel (37%) (GC EUROPE N.V., Belgium)	-	Orthocem (FGM Produtos Odontológicos LDA, Brasil)	Bisphenol A diglycidyl ether methacrylate (Bis-GMA) (25–35 wt%) Triethylene glicol dimethacrylate (TEGDMA) (10–15 wt%) Methacrylated phosphate monomer (>2 wt%)	Silane treated silicon dioxide (45–60 wt%)	Camphorquinone (<1 wt%) Sodium fluoride (>1 wt%)
4 (*n*=20)	-	Transbond™ Plus (3M Unitek, USA)	Transbond XT Light Cure Adhesive (3M Unitek, USA)	Bisphenol A diglycidyl ether dimethacrylate (10–20 wt%) Bisphenol A bis (2-hydroxyethyl ether) dimethacrylate (5–10 wt%)	Silane-treated quartz (70–80 wt%)	Dichlorodimethylsilane reaction product with silica (<2 wt%)
5 (*n*=20)	GC Ortho Etching Gel (37%) (GC EUROPE N.V., Belgium)	Transbond XT Primer (3M Unitek, USA)	Transbond XT Light Cure Adhesive (3M Unitek, USA)	Bisphenol A diglycidyl ether dimethacrylate (10–20 wt%) Bisphenol A bis (2-hydroxyethyl ether) dimethacrylate (5–10 wt%)	Silane-treated quartz (70–80 wt%)	Dichlorodimethylsilane reaction product with silica (<2 wt%)

Table 2: The intergroup comparison of the shear bond strength in Mpa.

	Mean (Mpa)	Standard Deviation	*P*-value
Group 1	11.86	3.83	<0.001***
Group 2	10.06	3.44	
Group 3	7.65	3.71	
Group 4	10.13	3.77	
Group 5	14.01	5.79	

One-way ANOVA, P<0.05*, <0.01**, <0.001***. Mpa: Megapascal

Table 3: The comparison of the shear bond strength between adhesive groups.

	Group 2	Group 3	Group 4	Group 5
Group 1	NS	0.01**	NS	NS
Group 2		NS	NS	0.03*
Group 3			NS	<0.001***
Group 4				0.03*

Bonferroni test (P<0.05*, <0.01**, <0.001***, NS: Non-significant)

groups except Group 1 ($P < 0.03$, $P < 0.03$, $P < 0.001$). Among the groups containing integrated primer, the highest shear bond values were recorded with Group 1. The difference between Group 1 and Group 3, which both consist of adhesive samples with integrated primers, was statistically significant ($P < 0.01$). There was no significant difference in the comparison between the self-etching group and the groups containing integrated primer samples.

The ARI scores are presented in [Figure 1]. The comparison of the ARI scores between adhesive groups is presented

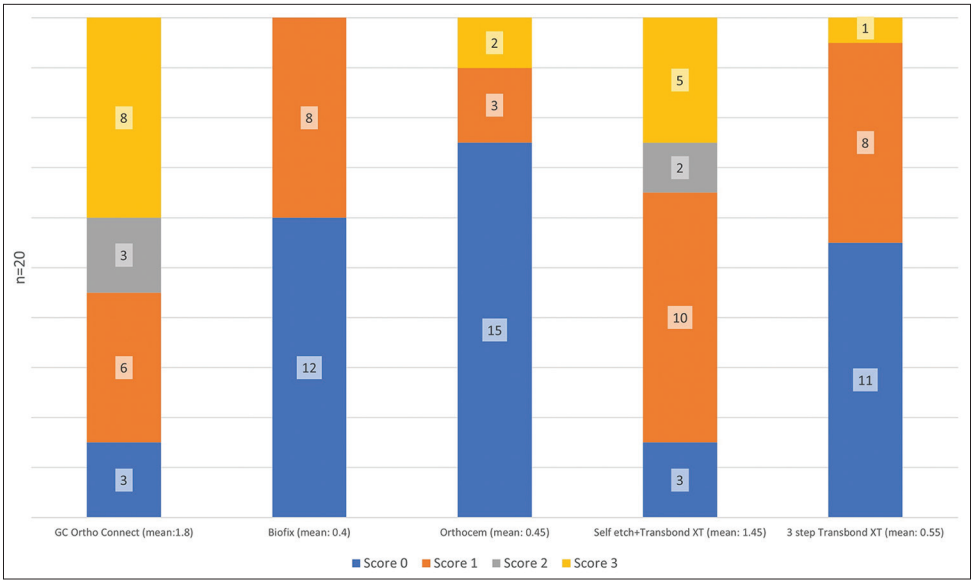

Figure 1: The distribution of the adhesive remnant index scores.

in [Table 4]. Group 1 and Group 4 showed statistically significantly higher scores compared to the other 3 groups. No significant difference was noted between the ARI scores of Groups 2, 3, and 5.

Table 4: The comparison of the ARI scores between adhesive groups.

	Group 2	Group 3	Group 4	Group 5
Group 1	<0.001*	0.001*	NS	0.003*
Group 2		NS	0.003*	NS
Group 3			0.001*	NS
Group 4				NS

Fisher's Exact test (P<0.05*, <0.01**, <0.001***, NS: Non-significant).
ARI: Amount of residual adhesive

DISCUSSION

The bonding procedure is closely related to the successful performance of orthodontic treatment because forces are transmitted with the brackets attached to the enamel. Besides performing a successful bonding, it is also important to reduce the duration of this long procedure in terms of patient-doctor comfort. Although adhesives with integrated primer, specially developed for this purpose, have been available on the market, for now, less than a decade, there are still few comparative studies in the literature evaluating the bonding performance of these adhesives. Our study aimed to contribute to the literature and the clinical practice by evaluating the SBS of three different adhesives with integrated primer compared to the conventional bonding protocol and to the self-etching system *in vitro*. In the present study, the material selection was based not on their composition but on their usage indication, which is bonding brackets without primer application. The samples in the conventional protocol consisting of the tri-step procedure, involving etching, priming, and bonding, were bonded using Transbond XT, which is accepted as the gold standard. Transbond XT has proven to be a successful agent in previous studies, and this group of samples has a control group characteristic that also checks the setup of our *in vitro* study.[4-6] The self-etching adhesives are known to, provide lower but acceptable shear bond values compared to the primer application following acid conditioning protocol.[7] A group of samples was bonded using Transbond™ Plus to be able to compare the SBS of self-etching primers to those of adhesives with integrated primer. In our study, significantly higher SBS values were obtained in the acid and rinse group compared to the self-etching primer group in harmony with the literature. This difference can be explained by the fact that the removal of the hybrid layer formed on the enamel prisms with etching by washing and drying provides a better infiltration surface of the resin micro tags enabling better micro retention.

In the literature, the debonding force of the brackets is often recorded with a shear force applied with a steel tip with standardized crosshead speed until breakage.[8-12] In harmony with other studies in the literature, we placed the labial surfaces of the brackets parallel to the blade-type shearing wedge of the testing machine and ensuring visually that the tip touched only the bracket while applying the breaking force vertically. A bracket with known dimensions having a rectangular base was used to ease the conversion of the data provided in newtons into megapascals which is the unit of stress between two surfaces.[8]

O'Brien *et al*.[13] reported that most of the bond failures occur in the first 6 months of the treatment and they suggested three possible explanations for this; any clinical technical sensitivity would become evident within the initial stages of the force application, the patients start trying the restricted foods and unbalanced occlusal forces. This finding has been confirmed by subsequent studies in the literature.[14,15] On the other hand, many *in vitro* studies evaluated the bracket failure rates after thermal aging with 5000 cycles, which would correspond to approximately 4.16 years of clinical service.[16,17] Elekdag-Turk *et al*.[2] reported no significant difference in SBS values for 2000 and 5000 thermal cycles; however, these number of cycles both presented a significant difference from samples that were not subjected to thermal aging.[2] In the present study, aging with 5000 cycles was applied since 4.16 years of clinical service is covering the duration of an ordinary orthodontic treatment.

Clinically successful bracket adhesion values were determined in the range of 5.9–7.8 MPa to sustain normal oral and orthodontic forces.[18,19] In our study, the highest shear bond values were obtained with the Transbond XT group, followed by the GC Ortho Connect™ group containing integrated primer that provided also successful bonding. On the other hand, the Orthocem group, which is one of the adhesives with integrated primer, showed SBS values near-slightly below the threshold values stated in the literature. In a study by Scribante *et al*.,[20] the bonding performance of Orthocem and that of another adhesive, which similarly does not require a primer application, were compared with the conventional bonding protocol using two different bracket bases.[20] Similarly, significantly lower SBS values were obtained with Orthocem compared to other groups. It has been suggested that this difference may depend on the particle size and its distribution in the materials. It was also emphasized that Orthocem contains less filler (45–65 wt% vs. 70–80 wt%) and more resin (35–50 wt% vs. 15–30 wt%) compared to Transbond XT, and it was postulated that the bonding performance of the materials depends on the type of resin they contain. Considering the various filler-resin percentages in the formulation of the materials presented in [Table 1], we also believe that the chemical formulation has an important impact on the SBS values. Furthermore, different values were recorded depending on the design of the bracket base. In our study, the same bracket type was used in each group to eliminate the differences regarding the bracket base design.

The SBS and the resin tag penetration of brackets bonded with a single component bonding system (Biofix), a chemical-cured bonding system (Unite), and the conventional light-cured bonding system (Transbond XT) were previously compared under laboratory conditions.[21] Higher mean SBS values were achieved with brackets bonded with Transbond XT compared to those obtained with Biofix and Unite. The authors explained this difference with the fact that acid etching creates a morphologically porous layer by demineralization and the surface free energy of the modified enamel layer is increased providing better micro invasion by capillary attraction.[22] This explanation was supported by the resin penetration being highest for Transbond XT, when compared to Biofix and Unite.

In a study by Shapinko *et al*.,[23] the adhesive with an integrated primer that we tested in the first group (GC Ortho Connect™) was compared with the conventional acid-rinse-prime application. The adhesive with integrated primer was used as recommended by the manufacturer and with an extra application of a primer layer. Shapinko *et al*.[23] found that the additional primer layer showed a trend to increase the mean SBS value of the integrated primer adhesive group, but the differences were not statistically significant. Similarly, in our study, no statistically significant difference was found between GC Ortho Connect™ and Transbond XT. In our study, the extra primer application was not performed, because it was not recommended by the manufacturer, and we would not be able to test the advantage of the material without primer application. The mean SBS values reported by Shapinko *et al*.[23] are lower compared to our results even though thermal aging equivalent to approximately 4 years of clinical service was performed in our study. This difference may have resulted from the use of human teeth in our study and bovine teeth in the other study. Moreover, Shapinko *et al*.[23] employed a different method: the wire loop protocol, which consists of a strand of braided stainless steel wire going around the wings of brackets. The wire loop protocol is one of the two most common methods together with the shear blade technique for testing orthodontic SBS and the wire loop technique is proven to produce lower SBS values.[24]

The necessity of primer application before bracket bonding has been tested with various bracket adhesives. In most of these studies, there is no statistically significant difference in terms of SBS.[25-27] Researchers suggest that a resin phase devoid of filler is present in sufficient amounts on the adhesive to fill the micropores in the etched enamel, providing good adhesion. In our study, the products claimed to contain integrated primer were tested, but the conventional system was used as it is described by the manufacturer. The achievement of successful bonding values with these new adhesives can also be explained by the fact that the resin phase penetration to the acid-modified enamel surface was good enough to ensure successful bracket bonding except for the Orthocem samples.

In the studies by Shapinko *et al*.[23] and Scribante *et al*.,[20] there is no mention of any aging process before SBS measurements are performed. However, it has been proven in the literature that there is a difference between the SBS

values of samples with and without thermal aging.[28] In our study, the thermal cycle aging was applied to better simulate the oral environment and to be able to make more accurate clinical projections.

Considering the ARI scores used to evaluate whether the separation during rupture is caused by the bracket-adhesive or tooth-adhesive interfaces, we observed that higher scores were recorded in the first and the fourth groups compared to the other groups. We can conclude that separation occurs evenly between the bracket and enamel surfaces in these groups. In the other groups, the ARI score was lower than 1, meaning that the adhesive remained <50% on the tooth surface. Since there was no statistically significant difference between these groups, we can suggest that SBS values and ARI scores do not show parallelism. Although there are studies in the literature that suggest that SBS values and ARI scores are compatible, our results are similar to those of studies that do not find SBS values in harmony with ARI scores.[29,30] This can be explained by the fact that ARI scores vary according to the resin type independently from SBS values. However, the method difference in these studies and the lack of the aging process are parameters that should be evaluated together with this interpretation of the results. Moreover, the ARI scores in our study were assessed visually with the naked eye since the use of a stereomicroscope was not available within the study. This might be considered a limitation of the study.

The most important limitation of our study is it's *in vitro* nature. Although this limitation is tried to be optimized with the aging procedure, the oral environment has more parameters than *in vitro* conditions. It is necessary to support or revise the results obtained with clinical studies. There is a need for randomized controlled studies evaluating the bracket survival rate *in vivo* using adhesives with integrated primer. Furthermore, it has been suggested that the primer can prevent or reduce the microleakage between the demineralized enamel surface and the adhesive interface.[25] Whether or not the contribution of the adhesives with integrated primers to white spot formation is a parameter that should be investigated in clinical conditions.

CONCLUSION

Based on our findings, the following conclusions can be drawn:

- Clinically successful bonding values have been achieved with 2 (GC Ortho Connect™, and Biofix) out of three different adhesives with integrated primer
- One of the adhesives with integrated primer (GC Ortho Connect™) provided a similar SBS value to Transbond XT, which is considered the gold standard for adhesion
- No significant difference was observed between the SBS values of the self-etching group and the groups containing integrated primer samples
- ARI scores varied independently from SBS values.

Declarations

This study was presented with a poster at the 17th Turkish Orthodontic Society International Virtual Congress, Turkey.

Availability of data and material

Available on request.

Ethics approval

The study was approved and followed by Bezmialem Vakif University Ethics Committee with file number 04/57.

Declaration of patient consent

Patient's consent not required as there are no patients in this study.

Financial support and sponsorship

Nil.

Conflicts of interest

There are no conflicts of interest.

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