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ORIGINAL ARTICLE

VOLUMETRIC ASSESSMENT OF INTERNAL VOIDS AND SHEAR BOND STRENGTH OF PRIMER BASED AND NON-PRIMER BASED ORTHODONTIC ADHESIVES - AN *IN VITRO* STUDY

Evaluación volumétrica de las superficies internas y la resistencia adhesiva al cizallamiento de adhesivos de ortodoncia con o sin primer: Un estudio *in vitro*

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ABSTRACT

Aim: The aim of this study was volumetric evaluation and comparison of internal voids and shear bond strength (SBS) between primer-based adhesives (PB) and non-primer-based adhesives (NPB).

Material and Methods: Extracted 40 human maxillary premolar teeth were bonded with four different adhesives - group 1: Ormco enlight, group 2: Transbond XT, group 3: Aqualine LC, group 4: Orthofix SPA, followed by three-dimensional microscopic tomographic valuation of the adhesive - tooth bracket interface. The images were reconstructed and 3D volumetric visualisation for mean void volume was performed. Shear bond strength (SBS) assessment was also performed.After shear mode testing, each tooth's enamel surface was examined to determine the Adhesive Remnant Index (ARI), which assesses the amount of adhesive remaining after debonding. Data was tabulated and SPSS software was used for statistical analysis with level of significance set at 0.05.

Results: A statistically significant difference (*p*-value-0.000) in mean void volume and void percentage was observed. SBS showed a statistically significant difference between the groups. (*p*-value-0.000). ARI scores with the Kruskal-Wallis test revealed statistically significant differences (*p*-value= 0.000)

Conclusions: Teeth bonded with NPB adhesive (Aqualine LC) had the highest void volume. Teeth bonded with PB adhesive (Transbond XT) had the highest SBS. Minimal adhesive remnants on enamel were noted for teeth bonded with PB adhesives (Transbond XT & Ormco enlight). Ormco Enlight and Transbond XT left little to no adhesive (Scores 0 and 1), while Aqualine LC and Orthofix SPA had higher adhesive retention (Scores 2 and 3).

Keywords: *Dental Cements; Adhesives; Shear strength; Materials testing; X-Ray Microtomography; Orthodontics*

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RESUMEN

Objetivo: El objetivo de este estudio fue la evaluación volumétrica y la comparación de los huecos internos y la resistencia adhesiva al cizallamiento (SBS) entre adhesivos con (PB) y sin (NPB) primer.

Material y métodos: Se adhirieron 40 premolares maxilares humanos extraídos con cuatro adhesivos diferentes: grupo 1: Ormco Enlight, grupo 2: Transbond XT, grupo 3: Aqualine LC, grupo 4: Orthofix SPA. Posteriormente, se realizó una evaluación tomográfica tridimensional de la interfaz entre el adhesivo y el bracket. Se reconstruyeron las imágenes y se realizó una visualización volumétrica 3D del volumen medio de huecos. También se evaluó la resistencia de adhesión al cizallamiento. Tras la prueba de cizallamiento, se examinó la superficie del esmalte de cada diente para determinar el Índice de Remanente de Adhesivo (IRA), que evalúa la cantidad de adhesivo restante tras la desadhesión. Los datos se tabularon y se utilizó el programa SPSS para el análisis estadístico con un nivel de significancia de 0,05.

Resultados: Se observó una diferencia estadísticamente significativa (p= 0,000) en el volumen medio de vacíos y el porcentaje de vacíos. El SBS mostró una diferencia estadísticamente significativa entre los grupos (p= 0,000). Las puntuaciones ARI con la prueba de Kruskal-Wallis revelaron diferencias estadísticamente significativas (p= 0,000).

Conclusiones: Los dientes unidos con adhesivo NPB (Aqualine LC) presentaron el mayor volumen de vacíos. Los dientes unidos con adhesivo PB (Transbond XT) presentaron el SBS más alto. Se observaron mínimos restos de adhesivo en el esmalte en los dientes unidos con adhesivos PB (Transbond XT y Ormco Enlight). Ormco Enlight y Transbond XT dejaron poco o ningún adhesivo (puntuaciones 0 y 1), mientras que Aqualine LC y Orthofix SPA presentaron una mayor retención adhesiva (puntuaciones 2 y 3).

Palabras clave: *Cementos dentales; Adhesivos; Resistencia al corte; Ensayos de materiales; Microtomografía por Rayos X; Ortodoncia*

INTRODUCTION

Bonding is a term conventionally used to describe the attachment of the bracket using bonding resins to the enamel surface.[1,2]

Orthodontic treatment should aim for both effectiveness and efficiency, considering factors such as the total treatment duration and the number of required appointments. The basic principles for successful bonding include cleaning the adhesive surfaces, good wetting, ensuring intimate adaptation, applying appropriate bonding force, and performing complete curing (polymerization).[3,4] Failure of bracket bonding to the enamel has a prevalence ranging from 3.5% to 10%.[5]

Adhesion failures are most common within the first 90 to 180 days of bonding the accessories.[6] These failures can lead to an extension of the treatment duration, resulting in both direct and indirect extra expenses and causing dissatisfaction among patients.[7] Debonding of brackets can occur due to many reasons and the most important among them include the composition and consistency of the adhesive being used, curing parameters, the tooth surface preparation, and voids in the adhesive and tooth interface after curing of the adhesive.[3]

Polymerisation shrinkage during the curing process creates a space between the enamel surface and adhesive, allowing bacteria, ions, fluids, and toxic substances from the oral cavity thereby resulting in enamel decalcification, enamel discoloration, corrosion, and decreased bond strength.[8]

Microleakage caused by polymerization shrin-kage can be detected at the interface of the adhesive material and enamel surfaces, or the contact of the adhesive material and brackets.[9] It is important to keep the amount of bracket movement during placement to a minimum to prevent disruption of the polymerization process which may weaken the bond.[10] Bond strength depends on adhesive contact between the bracket and the tooth surface and the lesser the internal voids the greater the bond strength.[3] Voids in the bonded interface result in reduced bonding surface area that leads to premature failure.[11] Also, voids within the bulk of the adhesive can lead to fracture of the material due to increased stress.[4] As a result, debonding can occur at the bond interface (tooth-adhesive or bracket-adhesive) or a bulk fracture of the adhesive can occur.

Microleakage in the tooth adhesive complex has been reported for different types of brackets bonded with different adhesive systems.[12] The distinction between primer-based adhesives (PB) and non primer based (NPB) adhesives lies in the application of a bonding agent (primer) to the tooth surface before attaching brackets or other appliances.[13]

Primer is a low-viscosity unfilled resin containing triethylene glycol dimethacrylate (TEGDMA) and bisphenol A-glycidyl methacrylate (BIS-GMA).[14] Its purpose is to penetrate the etched enamel, enhance adhesion, and protect against demineralization, which increases bond strength and reduces marginal leakage.[15]

However, skipping the primer application has been found to lower the risk of occupational exposure to unpolymerized resin and shortens bonding time by removing a step, thereby reducing the chance of mois-ture contamination.[16]

Non-primer, single-component adhesives, requiring only an etching phase, contain phosphoric ester monomers that provide a stable bond without the need for a separate primer.[17] There is no literature on the evaluation of the internal void distribution in NPB adhesives, and a comparison of PB and NPB adhesives for internal voids has never been published in the past. This study aimed to evaluate the effect of adhesives without primer and adhesives with primer on internal void volume and shear bond strength in four different orthodontic adhesives.

MATERIALS AND METHODS

This *in vitro* study was conducted at the White lab, Saveetha Dental College and Hospital from the period of March to June 2023. The present study was approved by the Institutional Systematic Review Board (SRB Reference No-SRB/SDC/ORTHO- 2203/ 23/056).

Sample size calculation was done based on the study done by Ozturk *et al.*,[9] reporting on microleakage in different orthodontic adhesives using the G Power 3.1.9.4 (Universitat Keil, Germany) with alpha error set at 0.05 and power set at 95 %, yielding a sample size of 40 with 10 in each group.

Material selection and preparation

Forty premolar teeth extracted for orthodontic purposes without any congenital anomalies, surface flaws, cracks, decay, or any restorations were collected from the Department of Oral & Maxillofacial Surgery. Following extraction, teeth were mechanically cleaned using pumice and rubber prophylactic cups to remove any debris and the teeth were then placed in a solution containing an aqueous solution of hydrogen peroxide.

The teeth were left in this solution for one week and after immersing in distilled water, they were stored in saline at room temperature. A high-speed air turbine hand-piece sectioned the teeth under water irrigation to remove the root. The teeth were then randomly included in four groups, with ten teeth per group which are as follows:

Group 1: Enlight (Ormco, United States);

Group 2: Transbond XT (3M Unitek, United States);

Group 3: Aqualine LC (Tomy International Inc, Japan);

Group 4: Orthofix SPA (Orthofix, Verona, Italy).

The enamel of the teeth was prepped for bonding with a water/pumice slurry in brushes for 15 seconds before rinsing and drying with an air stream. The buccal surface of the teeth was treated for 15 seconds with 37% phosphoric acid gel and then rinsed for 10 seconds with water and dried for 10 seconds with an oil- and moisture-free air spray.

The buccal surface of all etched teeth showed a chalky white appearance that indicated etched enamel. Four different types of orthodontic adhesives were used for bonding the brackets onto the extracted premolars. All teeth were bonded using 0.022''x 0.028'' MBT Gemini ceramic (3M Unitek; Monrovia, CA, USA) brackets.

For PB adhesives, a thin layer of primer was applied to the exposed enamel surface followed by light curing for 3 seconds, and the brackets of these groups were loaded with Ormco enlight and Transbond XT (Group 1 and Group 2).

The brackets were positioned with firm pressure followed by adequate flash removal around them followed by light curing for 3 seconds from the mesial and distal surfaces (Woodpecker Light Cure Lux E Plus -3 Second curing). Whereas for NPB adhesives, brackets were loaded with Aqualine LC and Orthofix SPA after etching and cured for 3 seconds (Woodpecker Light Cure Lux E Plus -3 Second curing). The bonded tooth samples were then immediately subjected to a Micro CT scan. Figure 1 shows a picture of sectioned tooth samples bonded with a ceramic bracket.

MicroCT scanning

Using a high-resolution micro-CT scanner (Version 1.16.1.0, SKYSCAN2214) with an X-ray source current of 60 mA, and voltage of 130 kV. The x-ray source had a pixel size of 12.04 mm, a 1-mm-thick Al filter, a rotation step of 0.3, and an exposure time of 500 ms. All specimens were scanned to assess the amount of voids in the interfaces. The tooth samples with the bonded brackets were mounted on the rotating stage in the Micro CT scanner in such a way that the X-rays were directed to the occlusal surface of each sample to assess the internal voids in the tooth-adhesive and adhesive-bracket interfaces.

Then, reconstructed the original micro-CT images using the software program -NRecon; 3D volumetric visualization and analysis were done with CT Vox and measured the volume between the adhesive and the bracket as well as the voids inside the adhesive. All the procedures were done by the same skilled operator.

Bond Strength Test

A mechanical testing device (Model 4411; Instron, Canton, MA, USA) was used to perform the Shear Bond Strength (SBS) test. The tooth-bracket interface was positioned parallel to the testing instrument using a mounting jig. The bracket-tooth contact was subjected to occlusal-gingival stress, generating a shear force at a crosshead speed of 1.0 mm/min until the bond failed.

Figure 1.
A sectioned tooth sample bonded with a ceramic bracket

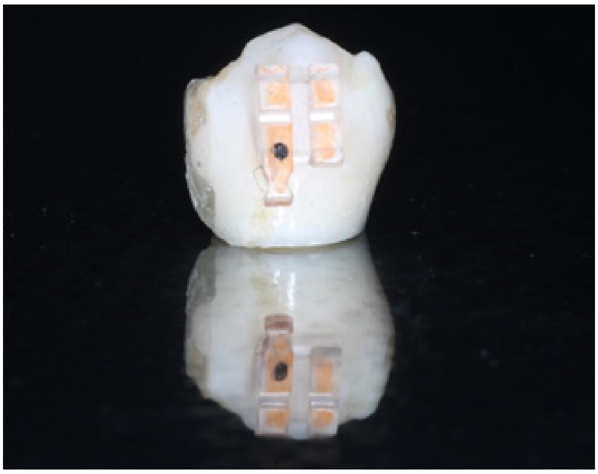
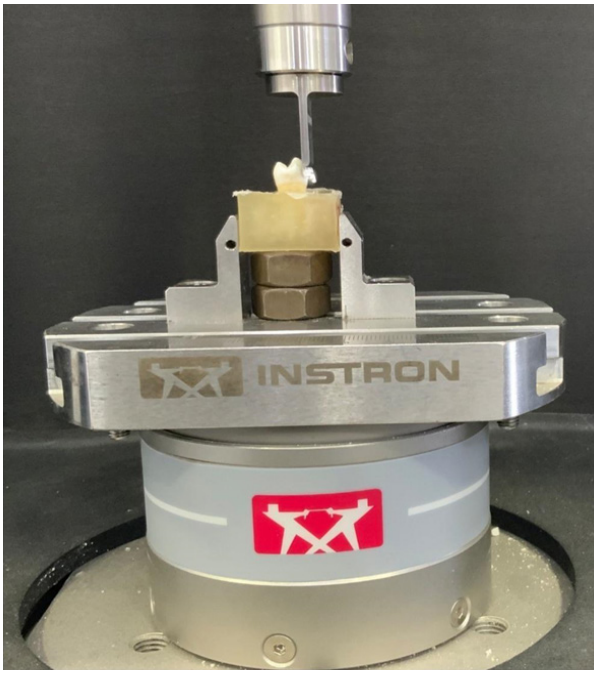


Figure 2.
Instron universal testing machine for shear bond strength (SBS) evaluation



This was done with the flattened end of a steel rod and attaching it to the crosshead of a Universal Testing Machine. The load applied at the time of fracture was recorded in Newtons. Figure 2 shows a bonded tooth subjected to SBS evaluation.[5]

Adhesive Remnant Index (ARI)

Following shear mode testing, the enamel surfaces of the teeth were individually examined. An optical microscope, specifically a Stereomicroscope with a 10X magnification, was employed to assess the Adhesive Remnant Index (ARI) score. The ARI score evaluates the amount of adhesive left on the tooth after debonding:

Score 0: No adhesive left on the tooth surface.

Score 1: Less than 50% of adhesive remaining.

Score 2: More than 50% of adhesive remaining.

Score 3: All adhesive left on the tooth surface, with an impression of the bracket base.

Statistical analysis

Statistical analysis was performed using SPSS Version 23.00 (SPSS Inc, Chicago, Illinois, USA). The data was checked for normality using the Shapiro-Wilk test with the level of significance set at $p<0.05$. The Shapiro-Wilk normality test indicates that the dataset conforms to a normal distribution. The inter-group analyses of void volume, percentage of void volume, and bond strength were done using one-way ANOVA.

RESULTS

The data was normally distributed (p-value >0.05) and one way ANOVA was performed to compare the mean values of internal void volume, void percentage, and SBS among the adhesive groups.

Table 1.
Mean, standard deviation and One-way ANOVA test, *p*-values for void volume, void percentage, and shear bond strength of the four adhesive groups.

	Mean±SD of internal void volume (mm^3)	*p*-value (Void volume)	Mean±SD Void volume percentage	*p*-value (Void volume percentage)	SBS Mean±SD (MPa)	*p*-value
Group 1	3.04±0.41	<0.05	21.20±1.16	<0.05	9.39±0.23	<0.05
Group 2	1.91±0.54		13.6±2.68		12.03±1.21	
Group 3	15.44±0.12		40.34±0.14		11.43±0.48	
Group 4	6.66±1.19		32.25±1.42		2.54±0.59	

Table 2.
Adhesive Remnant Index (ARI) scores for four adhesive groups, presented as the number of samples with the corresponding percentage of the total for each ARI score category with *p*-values obtained using Kruskal-Wallis test.

ARI score	Group 1 N (%)	Group 2 N (%)	Group 3 N (%)	Group 4 N (%)	*p*-value
0	4 (40)	3 (30)	0 (0.0)	0 (0.0)	<0.05
1	5 (50)	5 (50)	2(20)	0 (0.0)	
2	1 (10)	2 (20)	6 (60)	7 (70)	
3	0 (0.0)	0 (0.0)	2 (20)	3 (30)	

Void volume and void percentage

The descriptive statistics, one-way ANOVA of void volume (expressed as mm^3), and void percentages are tabulated in Table 1. Figure 3 illustrates the micro-CT scan of brackets bonded to the teeth. A significant difference in the mean void volume and percentage among the groups was noted (*p*-value-0.000) and teeth bonded with Aqualine LC had the highest void volume (15.44±0.12 mm^3) and void percentage (40.34±0.14).

Shear bond strength (SBS)

Table 1 also gives the Mean, Standard Deviation, and SBS of the four adhesive groups. A significant difference was found between the groups (*p*-value -0.000) and teeth bonded with Transbond XT had the highest SBS values (12.03±1.21 MPa)

Adhesive remnant index scores (ARI)

Table 2 summarizes the Adhesive Remnant Index (ARI) scores for four adhesive groups, indicating the distribution of ARI scores (0 to 3) among samples in each group. As the data was not normally distributed (*p*-value <0.05), the Kruskal Wallis test was done to compare the frequency of ARI scores among the groups. It showed statistically significant results (*p*-value - 0.000).

The results suggest that Enlight and Transbond XT had none or less than 50% adhesive remaining on the tooth after debonding

(Score 0 and 1), while Aqualine LC and Orthofix SPA exhibited higher retention of adhesive, especially with Score 2 and Score 3.

DISCUSSION

In the present study, an evaluation of the void volume and void percentage of different adhesives along with SBS and ARI scores was done. The adhesives used were both PB and NPB and void distribution, void volume and shear bond strength assessment was performed. Internal void volume, representing potential gaps or spaces within the adhesive, is a key metric for evaluating the structural integrity of the adhesive, while SBS measures the force required to break the bond between the adhesive and the tooth surface. As NPB adhesives are highly sought after in clinical practice owing to their reduced chair side time, it is important to assess the internal voids and the influence on SBS.

On the evaluation of void volume and percentage with a micro-CT, it was noted that voids were seen at the adhesive/enamel and adhesive/bracket interfaces. When comparing the void volume and void percentage among the studied adhesives, statistically significant ($p<0.05$) differences were noted and maximum voids were observed in teeth bonded with NPB adhesive (Aqualine LC). Similarly, significant differences among the adhesives for SBS were noted ($p<0.05$) and teeth bonded with PB adhesive (Transbond LC) had the highest SBS.

Teeth bonded with PB adhesives (Enlight and Transbond XT) had comparatively lesser internal void volume and higher SBS. Teeth bonded with a NPB adhesive (Aqualine LC) had higher void volume but also good SBS.

Hence it can be assumed that SBS is not affected only by internal voids in an adhesive. Higher adhesive remnants were observed on the enamel for teeth bonded with NPBs. The bond strength of adhesives should be enough to retain the bracket on the tooth surface, also avoid damaging the enamel surface during debonding and enable the removal of the leftover adhesive.[13] The presence of voids in adhesives leads to decreased surface area and increased stress concentration that may affect the bond strength, consequently may lead to premature failure.[3] Also voids within the bulk of the bonding material may lead to fracture within the structure.

In a previous study by Purk *et al.*,[18] it was observed that class II composite restorations were associated with greater bond strength if they had fewer voids, and an increase in the number of voids led to reduced microtensile bond strength.

In the study by Britton,[19] micro-CT was used to assess void characteristics in bonded orthodontic brackets, analyzing three bracket adhesive combinations-conventional, pre coated, and flash-free. In the present study micro CT was used but the study groups were different except for the conventional orthodontic adhesive (Transbond XT) and both SBS, ARI scores were assessed for the different adhesives.

The initial findings of this study suggest a correlation between adhesive voids and bond strength which is not in line with the present study results. Pre-coated brackets demonstrated minimal voids and the highest bond strength, whereas the flash-free group exhibited elevated void levels and lower bond strength.

Within each category, the presence of voids was observed at interfaces (adhesive/enamel and adhesive/bracket), which was similar to the present study.[19] In the present study, the mean void percentage and SBS of conventional adhesive (Transbond XT) were very similar to the study by Britton.[19]

Microleakage around orthodontic brackets has been studied extensively when compared to void volume assessment and occurs irrespective of the bracket type used, adhesive type, or the curing method. Previous studies have reported greater gap formation and microleakage occurring at the adhesive–enamel interface rather than the adhesive and bracket interface.[10,20]
Arhun *et al.*,[12] in their study had reported on the microleakage of metal and ceramic brackets and they concluded that metal brackets had greater microleakage than ceramic brackets,[12] while Arıkan *et al.*,[21] reported that teeth cured with light emitting diode (LED) had the least microleakage.

The results of the study by Alkis *et al.*,[10] on the comparison of microleakage around orthodontic brackets with different adhesives concluded identical amounts of microleakage with the studied adhesives. The enamel–adhesive interface is critical in terms of white spot lesions because the accumulation of bacteria in this location can cause enamel demineralization.

Limitations

Due to the three substantial variances in densities (tooth, adhesive, and bracket), brackets partially filled some of the gaps, especially at the bracket-adhesive contact. As a result, the data was difficult to read.

Clinical significance

By examining internal voids among different orthodontic adhesives, the study seeks to provide valuable insights into optimizing adhesive selection ultimately contributing to the enhancement of orthodontic treatment outcomes. The observed variations in internal void volume between primer based and non primer based adhesives didn't seem to affect shear bond strength.

The implications of present study findings on the clinical decision-making process include selecting orthodontic adhesives and to justify the need for PB adhesives as NPB adhesives reduce the chair side time.

CONCLUSIONS

Teeth bonded with NPB adhesive (Aqualine LC) had the highest void volume and clinically adequate SBS. Internal voids did not affect the SBS of NPB adhesives and teeth bonded with PB adhesive(Transbond XT) had the highest SBS with minimal adhesive remnants on enamel.

CONFLICT OF INTERESTS

The authors declare no conflict of interest.

ETHICS APPROVAL

The present study was approved by the Institutional Systematic Review Board (SRB Reference No-SRB/SDC/ORTHO-2203/23/ 056)

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AUTHORS' CONTRIBUTIONS

Anjusha Divakar: Conceptualization; Data Curation; Formal Analysis; Investigation; Methodology; Writing – Original Draft.
Ravindra Kumar Jain: Project Administration; Resources; Supervision; Validation; Visualisation; Writing – Review & Editing

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