

## Ex-vivo Bond Strength Of Adhesive Precoated Brackets And Moisture Insensitive Primer

### Abstract

**Objective :** The present study aimed at assessing the efficacy of moisture insensitive primer in moist environment and the effect of precoating the bracket base with the adhesive and modifying adhesive composition on the shear bond of the material. The study compared the shear bond strength of Transbond XT bonded with MIP in moist environment(Group II) with adhesive precoated APC plus brackets bonded with MIP in moist environment(Group III). These bond strengths were compared with Transbond XT with conventional primer on dry enamel(Group I).  
**Materials & Methods:** The study was performed on 30 maxillary human premolar teeth in each group mounted on acrylic blocks and tested on Instron machine at a cross head speed of 1 mm/min. The site of bond failure is determined from modified adhesive remnant index scoring. ANOVA was used to determine if significant differences existed between the groups  $P < 0.05$ . Weibull analysis was used to calculate the probability of failure at given values of applied force.  
**Results & Conclusions :** Results of the testing showed that there is no significant differences in bond strength of control i.e. Transbond XT(mean 12.00sd4.74 MPa) and Transbond XT+MIP in moist environment(mean 10.26sd3.49 ) and APC Plus +MIP in moist environment(mean 10.13sd3.11 MPa). All the groups showed bond strength values higher than that recommended for clinical bonding. Evaluation of site of bond failure showed that Transbond XT with conventional primer had predominantly cohesive failure within the adhesive, while the remaining two groups showed a failure at adhesive – enamel interface.

### Key Words

Adhesive Precoated Brackets, Moisture Insensitive Primer, Bond Strength, Weibull Analysis

### Introduction :

Direct bonding of orthodontic brackets, since its introduction by George V Newman<sup>[4]</sup>, dramatically improved the clinical practice of orthodontics. The pioneering work of Bunocore<sup>[1]</sup>, Bowen<sup>[2]</sup> and Tavas<sup>[3]</sup> yielded an array of materials which have become an integral part of orthodontic bonding. However the process of bonding is technique sensitive and moisture contamination is considered the most common reason for bond failure<sup>[6]</sup>. A reduction in bond strength of resins to etched enamel after moisture and saliva contamination has been reported by several researchers<sup>[7]. [8]. [9]</sup>.

The fifth generation bonding systems were developed to minimize the steps in bonding and increase the reliability of bonding in moist environment<sup>[6]</sup>. This moisture resistant adhesive is available in a primer formulation that replaces the conventional bonding agent applied to the etched enamel and consists of an aqueous solution of methacrylate-functionalized polyalkenoic acid copolymer and hydroxyethyl-methacrylate. This product has originally been used as a hydrophilic primer in dentine bonding

systems marketed by the same manufacturer (3M Unitek)<sup>[7]</sup>.

In an effort to enhance the quality of the adhesive system and to save chair side time and to perform faster and easier bonding procedures, the light-cured, adhesive- precoated brackets (APC, 3M Unitek) were introduced in 1992. Precoating the brackets necessitated modifications in the composition of the adhesive more specifically the viscosity of the adhesive was reduced in order to facilitate the accurate placement of the precoated bracket on the tooth by the clinician, without having to use excessive pressure on the tooth. Third generation precoated brackets with fluoride releasing and better moisture tolerant properties were introduced in 2004<sup>[10]</sup> (APC Plus).

The use of precoated brackets with a moisture insensitive bonding agent will be a definite advantage in clinical practice which will facilitate bonding in moist environment and also reduce steps in bonding, thereby saving chair-side time.

Hence, the present invitro study investigated the bond strength of metallic brackets bonded Transbond XT with MIP and adhesive precoated brackets ( APC

<sup>1</sup> Shailaja Akella

<sup>2</sup> Siddarth Shetty

<sup>3</sup> Subraya Mogra

<sup>4</sup> V. Surendra Shetty

<sup>1</sup> Reader, Department of Orthodontics  
Uttaranchal Dental and Medical Research Institute.

<sup>2</sup> Associate professor

<sup>3</sup> Professor & Head

<sup>4</sup> Dean & Professor,  
Department of Orthodontics & Dentofacial Orthopedics  
Manipal College of Dental Sciences , Mangalore.

### Address For Correspondence:

Dr. Shailaja Akella, House No: 96, Engineer's Enclave,  
Phase I, Gms Road, Dehradun - 248001

Mobile no: 09897972666

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plus) brackets bonded with MIP under moist conditions and compared them to conventional composite bonding material- Transbond XT with Transbond XT primer bonded under dry condition.

The present study would also evaluate if change in adhesive composition would significantly affect the bond strength as the adhesive used to precoat the metal brackets in APC Plus brackets essentially differ in the filler content.

### Materials and methods:

Fox et al<sup>[11]</sup>(1994) presented critical review of 66 publications on orthodontic bond strength testing and suggested a detailed protocol for future bond strength testing in orthodontics. Their criteria were taken into account when the protocol for the present in-vitro study was developed.

For the purpose of the study, 90 maxillary premolars extracted as a part of orthodontic treatment were collected and the teeth were non carious, unrestored and with no enamel cracks and not previously chemically treated and were debrided and stored in normal saline solution at room temperature. The time elapsed between the extraction and the bonding experiment was less than 6

months.

The teeth were randomly divided into three groups of thirty each, to be tested using different adhesive primer combination.

The teeth were sectioned using a straight fissure bur on aerotor hand piece at two thirds the root length (to facilitate mounting) and were mounted in cold cure acrylic blocks in three different colors – clear, red, and green, of diameter 23mm using alginate impression of metal ring, such that the buccal surface was exposed parallel and slightly above the rim of the impression. The mounted teeth were stored in normal saline till bond strength testing was done.

The brackets used for testing were Gemini series maxillary premolar brackets – Roth prescription and 022 slot (3M Unitek) and adhesive precoated Victory series low profile maxillary premolar brackets - MBT prescription and 022 slot brackets- APC Plus brackets (3M Unitek). All the brackets have similar bracket base (mesh base) design.

For all groups, the teeth were rinsed under tap water and cleaned with oil free, non fluoridated pumice using micromotor and rinsed and dried with chip-blower. Etching was done with 37% orthophosphoric acid gel etchant (Viscous Etch, Orthosource) for 30 seconds. The teeth were then rinsed and dried using absolute alcohol + acetone solution (dehydrating agent) until the buccal surfaces of the etched teeth appeared to be chalky white in color. Brackets were bonded on the buccal surfaces according to the instructions supplied by the manufacturer of each product.

#### **Bonding procedure:**

**Group I :** A thin coat of Transbond XT primer was applied using an applicator tip and light cured for 5 seconds. Brackets were placed onto the centre of the tooth, parallel to the long axis, with Transbond XT on the base of the bracket. After the bracket was properly positioned on the tooth, each bracket was subjected to 300 grams of force using a force gauge (Dontrix gauge) for 10 seconds and excess bonding resin was removed using a small scaler. The adhesive was light cured using SmartLite PS (DENSPLY, Germany) with a 5 W light emitting diode with average light intensity of 950mW/sqcm and wavelength of 450 – 490 nm (intensity maximum at 460nm),

for 20 seconds according to manufacturer's instructions. All the brackets are mesh based and similar in bracket base design.

**Group II:** MIP : After pumicing and etching like in group I, the bonding surface was wet with two coats of distilled water and then MIP primer was applied and then bonding (with Transbond XT ) and curing was done similar to that of control group.

**Group III :** APC Plus : After pumicing and etching and wetting the bonding surface like in group II, Adhesive Precoated Brackets (APC plus) were positioned and force of 300 gms for 10 seconds was applied using force gauge (Dontrix gauge) and excess bonding resin was removed using a small scaler. The adhesive was light cured using SmartLite PS (DENSPLY, Germany).

After bonding, all the specimens were stored in distilled water for one hour, at room temperature, before the shear bond strength testing was done.

#### **Bracket Surface Area Measurement:**

The bracket surface area is measured by micrometric analysis using an eye piece reticle and objective lens of 2.5 X magnification, on a light microscope (Leitz, Germany) and calculated to be 9.71 sqmm for APC Plus brackets and 10.80 sqmm for Gemini series brackets. The different bracket base areas do not affect the comparisons as the effective bond strength in MPa is being compared which includes the bracket surface area.

#### **Bond Strength Testing:**

Shear bond strength testing was done with the INSTRON Universal Testing machine (Model 4206). The machine was set and calibrated according to manufacturer's instructions. The acrylic block with embedded tooth and bonded bracket were positioned in the jig to ensure that the bracket edge corresponding to occlusal edge was always the site where load was applied. The steel plunger was used to apply load at the bracket adhesive interface. The brackets were shear tested for failure using a load cell of 500 N and a cross head speed of 1 mm/min.

The debonding force was recorded in kilograms by the computer and it was converted into bond strength (force per unit area) by the following formula:

**Bond strength =**

Debonding force (kgs) X 9.81  
Bracket surface area

#### **Site Of Bond Failure :**

The site of bond failure is determined from the adhesive remnant index scoring. Each of the tested specimens was observed under a stereomicroscope (Leitz, Germany) with magnification of 10 X and scoring was given according to the modified adhesive remnant index<sup>[12]</sup>.

Score 5 - no composite remaining on the enamel

Score 4 - less than 10% of composite remaining on the tooth surface

Score 3 - more than 10% but less than 90% of composite remaining on the tooth

Score 2 - more than 90% of composite remaining on the enamel

Score 1- all composite remaining on the tooth, along with the impression of the bracket base.

All the values recorded were tabulated and statistically tested.

#### **Statistical Analysis :**

The test statistics was performed using statistical package for social sciences (SPSS VERSION 13). Descriptive statistics were calculated for each group of teeth tested. Analysis of Variance was used to determine if significant differences existed between the various groups compared. The Pearson chi-square test was used to identify any significant difference in the ARI score amongst the groups. Significance for all statistical tests was predetermined at  $P < 0.05$ .

As indicated by Fox et al<sup>[11]</sup>, mean and S.D. may not be the best indicator for the performance of the material as when considering bonding materials, the weaker values (the tail of the distribution) are of more importance and hence a survival analysis - Weibull analysis is performed to analyze the characteristic bond strength and the probability of failure for each of the material at given values of applied force. The Weibull distribution<sup>[13],[14]</sup> is a parametric test. It takes into account the tail values of a distribution. It is characterized by the Weibull modulus, which expresses the spread of the data, and the normalizing parameter or characteristic level, in this case the characteristic bond strength, which more or less corresponds with the mean bond strength for a Gaussian distribution. It can be characterized by the following equation which relates probability of failure (Pf) to applied

stress ( ). The equation is:  
 $Pf = 1 - e^{-\left(\frac{\sigma - u}{o}\right)^m}$

u and o are constants. u is the lowest level of stress at which Pf approaches zero and is normally assumed to be equal to zero. o is the normalizing parameter or characteristic level and is described by Fox et al as the characteristic strength. The higher the Weibull modulus, lesser reliable is the material and as the characteristic strength increases, the reliability increases.

**Results :**

In Group I , during the bond strength testing, two brackets deformed extensively on debonding, and with two specimens reading could not be obtained ( debonded at 0 newtons ) due to technical snag (improper circuit with the computer reading the force values) in the testing machine. hence these specimens were not included in the study. The mean and S.D. were computed for 26 specimens in group I (Table 1).

In the control group (group 1) the mean bond strength (MPa) was found to be 12.00 sd 4.74 MPa with a range of 4.24 MPa to 20.53 MPa (confidence interval of 10.09 to 13.92)

In the moisture insensitive group bonded with Transbond XT (group II) the mean bond strength observed was 10.26 sd 3.49 and the range is 4.04 MPa to 20.32 MPa. (Confidence interval of 8.95 to 11.56)

In the adhesive precoated groups, group III – bonded with APC plus, the mean bond strengths observed were 10.13 sd 3.11 MPa and ranged from 5.26 MPa - 16.48 MPa (confidence interval of 8.97 to 11.29).

The ANOVA test was done to find if significant differences existed between the groups and it showed no statistical significant difference between the groups (p=0.023).

Table II shows the site of failure and the percentage of specimens having adhesive or cohesive failure and it shows that the bond failure was predominantly cohesive (within the adhesive material) in group I with 61.5% specimens showing an ARI score if 3, while groups II and III showed failure at enamel adhesive interface with 56.7% showing score of 4 in group II and 53.5 % in groups III.

Table III shows the Weibull modulus of the materials tested in the four groups. Group III has the highest Weibull modulus of 3.79 and group I shows the lowest Weibull modulus of 2.66. The characteristic strength observed is

Table I: Descriptive Statistics Of The Three Groups.

Group	N	Minimum	Maximum	Median	Mean	Std. Deviation	95% C.I
Group 1	26	4.247	20.530	12.641	12.004	4.74	10.089 to 13.918
Group 2	30	4.045	20.327	10.214	10.258	3.49	8.9548 to 11.561
Group 3	30	5.259	16.484	9.304	10.127	3.11	8.9657 to 11.288

Bond Strength –mpa

Table II: Adhesive Remanant Index Scoring In The Three Groups

ARI SCORE	Group		
	Group 1	Group 2	Group 3
2	0 0%	1 3.3%	0 0%
3	16 61.5%	8 26.7%	12 40%
4	8 30.8%	17 56.7%	16 53.3%
5	2 7.7%	4 13.3%	2 6.7%
Total	26 100%	30 100%	30 100%

$\chi^2 = 15.441, p = 0.017, sig$

Table III: Weibull Analysis

	Mean Bond Strength (Mpa)	Weibull Modulus (B)	Standard Error Of Modulus	Normalising Parameter (Characteristic Strength) (A) (Mpa)	Correlation Coefficient Of Linearised Least Square Plot
Group I	12	2.66	0.096	13.55	0.985
Group II	10.26	3.31	0.109	11.44	0.984
Group III	10.13	3.79	0.2	11.21	0.963

Table IV: Force Required For Various Probabilities Of Failure

	Force For 99% Chance Of Failure (Mpa)	Force For 95% Chance Of Failure (Mpa)	Force For 30% Chance Of Failure (Mpa)	Force For 5% Chance Of Failure (Mpa)	Force For 1% Chance Of Failure (Mpa)
Group I	24.06	20.47	9.19	4.44	2.4
Group II	18.15	15.94	8.37	4.66	2.85
Group III	16.77	14.97	8.54	5.11	3.51

highest for group I (13.55 MPa) and lowest for group III (11.21 MPa), and groups II shows characteristic strength values close to group III ( 11.44 MPa and 11.21 MPa respectively.) The correlation coefficient obtained is very high for all the groups (>0.95)

Table IV relates the probability of bond failure to applied stress. It can be seen that force for 99% chance of failure is highest for group 1 (24.06 MPa) and lowest for group III (16.77 MPa) – it means that group 1 can withstand higher force when compared to all other groups. It can also be seen that at 1 – 5 % chance of failure, there is little difference between the groups. It can be seen graphically on fig.1 by the fact that the curves cross over each other in this region.

The Graph – representing the plot of Weibull curves on common axes (Fig 1)

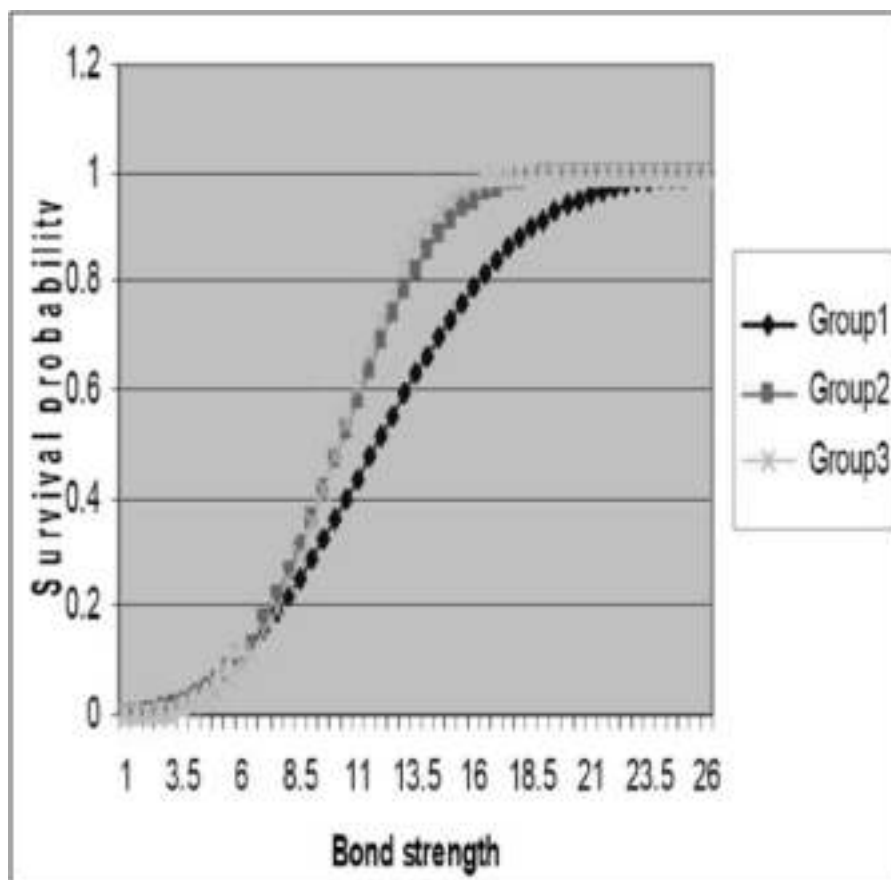


Fig 1 :Weibull Graph

shows the curve by group I being displaced extremely to the right indicating that for these brackets, there would be a lower probability of debonding during treatment, and the curve by group II and group III overlap considerably.

#### Discussion:

The results show that the bond strength values decrease in the order of group 1 > group 2 > group 3 with mean bond strength values of 12.00sd4.74 MPa, 10.26sd3.49, and 10.13sd3.11 respectively.

According to Reynolds<sup>[4]</sup>, bond strengths of 5.9 to 7.8 MPa suffice for most clinical orthodontic needs, although it is unclear whether this refers to shear or tensile bond strength. The bond strength values obtained are higher than the values prescribed by Reynolds et al and hence suggest that these material combinations can be used successfully clinically.

The bond strength values of the conventional adhesive under dry condition is in accordance with the values obtained by Grandhi et al<sup>[16]</sup> (11.06 MPa), Schanwdevdt et al<sup>[18]</sup> (14.82 MPa), and Arndt Klocke et al (15.07 MPa). However, the values are lower than that obtained by Webster et al<sup>[17]</sup> (26.1) and Hobson<sup>[15]</sup> (15.69 MPa) and higher than those obtained by Rajagopal et al<sup>[19]</sup> (9.57 MPa).

The bond strength values obtained for conventional adhesive with MIP in wet conditions is similar to those obtained by Hobson et al<sup>[15]</sup> (12.89 MPa) and Grandhi et al<sup>[16]</sup> (9.69 MPa) (after wetting the etched enamel surface with water), and Rajagopal et al<sup>[19]</sup> (9.07 MPa) (after wetting the etched enamel surface with saliva). Slightly higher bond strength values were seen with Schanwdevdt et al<sup>[18]</sup> (14.02 MPa) (after wetting the etched enamel surface with water) and Arndt Klocke et al (14.91 MPa) and Webster et al<sup>[17]</sup> (21.9 MPa) (after wetting the etched enamel surface with saliva).

The adhesive precoated brackets APC Plus - demonstrated significant bond strengths in the present study.

By applying a layer of Transbond MIP to acid conditioned enamel, in addition to micromechanical retention, a reversible hydrolytic bond mechanism can be established by breaking and reforming of carboxylate salt complexes formed between the ionized carboxyl groups of the methacrylate functionalized-

polyalkenoic acid copolymer and residual enamel calcium. This might enhance the bonding onto water-contaminated or saliva contaminated enamel surfaces.

The Weibull analysis also shows that uncoated brackets bonded with Transbond XT and Transbond primer in dry environment (control group) would perform better clinically with lesser bond failures than the other groups and the performance of the other twogroups is comparable and the reliability decreasing in the order of group II > group III.

It can be seen from **Table IV** that force for 99% chance of failure is highest for group 1 (24.06 MPa) and lowest for group III (14.76 MPa) – it means that group I can withstand higher force when compared to all other groups.

It can also be seen that at 1 – 5 % chance of failure, there is little difference between the groups. It can be seen graphically on Fig.1 by the fact that the curves cross over each other in this region.

If a bond failure rate of 5% is considered clinically acceptable, and if forces of around 5 MPa are experienced clinically, then group III – APC plus with MIP would be better in a clinical situation despite group I having higher mean bond strength. However the problem is that the stress applied in a clinical situation is very difficult to measure and varies throughout the mouth.

The ANOVA test was done to find if significant differences existed between the groups and it showed no statistical significant difference between the groups (p=0.023).

In this study, MIP under moist conditions produced acceptable bond strength compared to conventional primer, as was reported by Ross Hobson<sup>[15]</sup>, Grandhi et al<sup>[16]</sup>, Schanwdevdt et al<sup>[18]</sup>, Arndt et al, Rajagopal et al<sup>[19]</sup>. The values obtained for group II are in agreement with previous studies that evaluated the shear bond strength of conventional and hydrophilic primers on dry and contaminated (with distilled water and saliva) enamel by Arndt Klocke and Grandhi<sup>[16]</sup> and Rajagopal et al<sup>[19]</sup> (contamination with artificial saliva).

However this is not in agreement with Littlewood<sup>[19]</sup>, Rix et al<sup>[22]</sup>, Webster et al<sup>[17]</sup>, Zeppieri<sup>[20]</sup> who showed that bond strength of moisture insensitive primer in wet conditions is significantly lower than conventional primer in dry environment.

Bishara et al<sup>[24]</sup> (1997) found that precoated brackets have significantly lower bond strength than those obtained by Transbond XT on uncoated brackets. But Bishara et al<sup>[25]</sup> in 2002 studied the effects of modifying the adhesive composition of APC and APC II brackets on bond strength and found that it did not affect the shear bond strength.

The range of ARI scores clearly demonstrated that Transbond XT used in dry field showed a significantly greater frequency of bracket failure sites within the adhesive itself, but when used with MIP in moist environment, it debonded more frequently at the enamel- adhesive interface. This agreed with the results of Caccifesta et al<sup>[26]</sup>, Webster et al<sup>[17]</sup>, and Rajagopal et al<sup>[19]</sup>, the findings are probably due to the hydrophobic properties of the adhesive composite.

Regarding the percentage of area of adhesive remaining on tooth, MIP/Transbond XT and MIP/APC Plus left significantly less adhesive than did conventional primer/Transbond XT. This implies a reduction in chair-side time spent on adhesive removal after debonding

**Limitations :** The bond strength values in all three groups compared favorably with Reynolds<sup>[4]</sup> minimal bond strength values. However, clinical conditions may significantly differ from an in vitro setting. It needs to be emphasized that this is an in vitro study and the test conditions have not been subjected to the rigors of the oral environment. Heat and humidity conditions of the oral cavity are highly variable. Because of the probable differences in in vivo and in vitro conditions, a direct comparison cannot be made with the findings of the present studies.

It must also be noted, that the use of the terms 'moist' or 'wet' implies the presence of water, whilst saliva or crevicular fluid are considered 'contaminants'. Although the presence of water can be prevented by adopting moisture- control precautions during bonding procedures, the orthodontist is often faced with the problem of bonding in an environment with increased contamination risk from saliva.

Also, a continually increasing tensile or shear load applied to bonded brackets in the laboratory is not representative for the force applications that occur clinically. The type of debonding force in machines is not the same as the force applied in clinical debonding. Hence a direct

comparison of the clinical performance of these materials is not suggested to be made from the results of this study and to obtain better clinical information, clinical trials of the materials is recommended.

### Conclusions:

Following conclusions can be drawn from the present study:

1. Moisture insensitive primer with Transbond XT produced bond strength comparable with conventional adhesive Transbond XT and hence MIP can be effectively used to bond to enamel when isolation is a problem.
2. Adhesive precoated brackets – APC Plus produced bond strength comparable with Transbond XT and hence it can be used in combination with MIP in moist environment.
3. The change in the composition of the adhesive in APC Plus brackets did not significantly affect the bond strength of the material.
4. Transbond XT with conventional primer had predominantly cohesive failure within the adhesive, while the remaining three groups showed a failure at adhesive–enamel interface.
5. All the adhesives tested showed bond strength values higher than those recommended for clinical bonding.

The present study showed that MIP and APC plus brackets could be used successfully clinically under moisture sensitive conditions .

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