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ORIGINAL RESEARCH

Comparison of Shear Bond Strength of Hydrophilic Bonding Materials: An *in vitro* Study

C Nirupama, Sarasa Kavitha, Josy Jacob, K Balaji, B Srinivasan, Ramakrishnan Murugesan, NR Krishnaswamy

ABSTRACT

Aim: The aim of this study was to evaluate and compare the shear bond strength of hydrophilic materials like Transbond MIP with Assure-fluoride releasing light cure sealant paste system (Reliance orthodontic product), Enhance Lc-adhesion booster (Reliance Orthodontics) Prime and Bond NT–one step adhesive with nanotechnology (Dentsply Product India) with Transbond XT as the control group.

Materials and methods: The study was conducted on 180 extracted human premolar teeth which were divided into five main groups. Each group contained 36 teeth, which were further subdivided into three subgroups containing 12 teeth. The teeth were bonded in three different surface environments namely dry, contaminated with artificial saliva and reprimed after contamination with artificial salvia. The brackets were bonded and cured. The shear bond strength was tested using Instron universal testing machine (4501).

Results: The results were subjected to statistical analysis like 3 factorial ANOVA and compared to post-hoc using the Student Newman levels test. The residual resin on the tooth surface after debonding was evaluated with adhesive remnant index.

Clinical significance: The results revealed that in situations in which moisture contamination is critical there is distinct advantage in using hydrophilic primers.

Keywords: Shear bond strength, Hydrophilic primers, Moisture contamination.

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INTRODUCTION

The bonding of orthodontic attachments was a breakthrough in clinical orthodontics. The principle of adhesive dentistry dates back to 1955 when Michael Buonocore[1] using industrial bonding techniques postulated that acids could be used for surface treatment before application of the resins. He subsequently found that etching enamel with phosphoric acid increased the duration of adhesives under water. Later, Bowen developed a restorative material, composite resin (BIS-GMA). Since then bonding systems and materials have undergone continuous improvement overtime.

Traditional composite resin bonding materials and methods mandate completely dry and isolated fields in order to obtain clinically acceptable bond strength. However, maintaining an absolute dry (or) moisture free environment is difficult, if not impossible in certain situations like, bonding on partially erupted teeth, bonding on the lingual surface of lower arch, bonding on the second molar and during surgical exposure of teeth.

To overcome this short coming of moisture sensitivity, glass ionomer was introduced. Although they could tolerate some amount of moisture contamination, their bond strength was inadequate for clinical purposes. To overcome these shortcomings the manufactures have strived hard to bring about a formulation that can be used even in moisture contaminated or wet environments. Two such recent introductions are 'Transbond MIP and Assure'.

Manufacturers of these new materials, claim that these ethanol-based primers promote bonding in a wet environment, without adversely influencing the bond strength.

To our knowledge, very few studies have been performed to evaluate the bond strength and clinical usefulness of ethanol-based primers. The specific objectives of this study is to evaluate the bond strength of orthodontic bonding materials by substituting MIP for conventional primer. Further, the new material is compared with conventional bonding enhancers like, Assure (Reliance Orthodontics Products), which is a fluoride-releasing light cure sealant

paste system used in wet and dry environments, Enhance LC (Reliance Orthodontics)-adhesion booster and Prime and Bond NT, (Dentsply Product, India), which is a one step adhesive with nanotechnology.

MATERIALS AND METHODS

The aim of this study is to evaluate and compare the shear bond strength of Transbond light cure composite used with MIP under three different surface treatments namely, (i) dry, (ii) wet-contaminated with artificial saliva, (iii) applied one layer of primer after saliva contamination. This was compared with other similar types of moisture insensitive materials. The control group employed was Transbond XT with Transbond XT primer.

Transbond MIP is an ethanol-based hydrophilic primer and manufacturers recommend this primer in areas where it is difficult to control the moisture like in the posterior region or on partially erupting teeth, and on impacted teeth, etc.

Assure is a polyacid-modified composite resin more or less similar to resin-modified glass ionomer cements but will not autoset by the acid-based reaction seen with glass ionomer cements.[2] They behave primarily like resins.

Enhance LC[3,4] is an adhesion booster used along with a light-cured sealant. It contains ethanol and water and according to the manufacturer, it can increase adhesion of composite to any enamel (saliva contaminated enamel, fluorosed, hypocalcified or deciduous enamel).

Prime and Bond NT[4] is an acetone-based primer used with Esthet X composite material.

One hundred and eighty extracted human premolar teeth were stored in distilled water at room temperature. All the teeth were mounted on color coded acrylic blocks in such a way that they were embedded in the acrylic up to the cervical margin with long axis of tooth kept parallel to that of the central axis of the acrylic block. This is to simulate the natural position of the teeth in oral cavity. These sample blocks were segregated into 15 different colors for proper identification (Fig. 1).

Thirty-six teeth were randomly assigned to five different adhesive groups and were further divided into three subgroups of 12 teeth each and the teeth were subjected to three different types of surface treatments. All the teeth were first cleaned and polished for 5 seconds with slurry of non-fluoridated pumice and a rubber prophylactic cup for 5 seconds. Each tooth was then rinsed, dried and etched for 60 seconds with 37% orthophosphoric acid. The artificial saliva used in this study was from the ICPA company and comprised of glycerine and sodium carboxy methyl cellulose. The brackets used in this study were mesh backed, preadjusted edgewise maxillary/mandibular biscupid brackets [3M Unitek, Gemini brackets], with 0.022 or 0.018 slot size and the average surface area for the bracket base was 10.611 mm^2 (Fig. 2).

The different types of surface treatments were as follows:

Treatment 1

1. Etch
2. Rinse/dry
3. Applied a layer of primer
4. Applied a layer of composite
5. Light-cured

Treatment 2

Same as treatment 1, but a layer of artificial saliva (Fig. 3) was applied.

Treatment 3

Same as treatment 1, but after contamination it was reprimed. The materials were divided into five groups and were evaluated under three environments:

Groups IA, IB and IC were used as control groups and groups IIA to VC were used as experimental groups.

After all the teeth in the five groups were subjected to pumice prophylaxis and acid etching procedures the teeth were bounded as follows (Fig. 4):

Fig. 1: Samples prepared for testing debonding

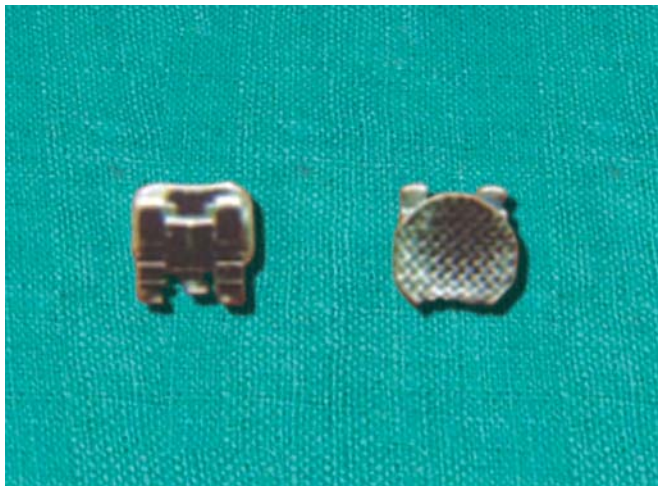


Fig. 2: PAE—Stainless steel brackets from 3M Unitek (Gemini)

Fig. 3: Artificial saliva

Fig. 4: Group I: Bonding procedure

Group IA—Transbond XT with a Conventional Primer in Dry Environment

After pumice prophylaxis, and acid etching procedures, a small amount of Transbond XT primer was applied on to the tooth surface. Using a syringe a small amount of Transbond XT adhesive paste was applied on to the bonding surface of the bracket.* Immediately after applying the adhesive, the brackets were placed lightly on the tooth surface and pressed firmly to seat the bracket. The excess adhesive was gently removed using a sealer from around the bracket base. The adhesive was light cured.**

Group IB—Transbond XT with a Conventional Primer in Artificial Saliva Contaminated Environment

After pumice prophylaxis, and acid etching a layer of artificial saliva was applied using a small applicator brush and care was taken to see that enough saliva was applied to allow complete hydration of the surface and it was allowed to stand on the surface for about 10 seconds. Then a layer of Transbond XT primer was applied and later Transbond XT adhesive paste was used to bond the bracket on to the tooth surface and brackets were placed lightly on to the tooth surface and pressed firmly to seat them in position. Gently, the excess adhesive was removed using a sealer from around the bracket base and cured for 20 seconds by placing the light source approximately 2 mm from the incisal edge and 10 seconds for both the mesial and distal sides of the brackets.

Group IC—One Layer of Transbond XT Conventional Primer Applied after Artificial Saliva Contamination

After pumice prophylaxis, and acid etching procedures, a layer of Transbond XT primer was applied and the surface was contaminated by applying a layer of artificial saliva on to the tooth surface to be bonded and later reprimed with another layer of Transbond XT primer, before bonding the bracket and cured as mentioned above.

The same steps were followed with the other groups also.

After all the teeth were bonded, acrylic blocks were allowed to stand in air for 10 minutes before they were put into a water bath at 37°C, and were stored for about 48 hours. After 48 hours of storage, the shear bond strength of the brackets was tested using an Instron universal testing machine 4501 (Fig. 5).***

The shear bond strength was then calculated and expressed as MPa according to the following formula,

$$\frac{\text{Debonding force in Kg} \times 9.81}{\text{Surface area of the bracket base in mm}^2} = \text{MPa}$$

STATISTICAL ANALYSIS

The results were subjected to ANOVA 3 factorial analysis, and compared post-hoc using the Student-Newman-Keuls

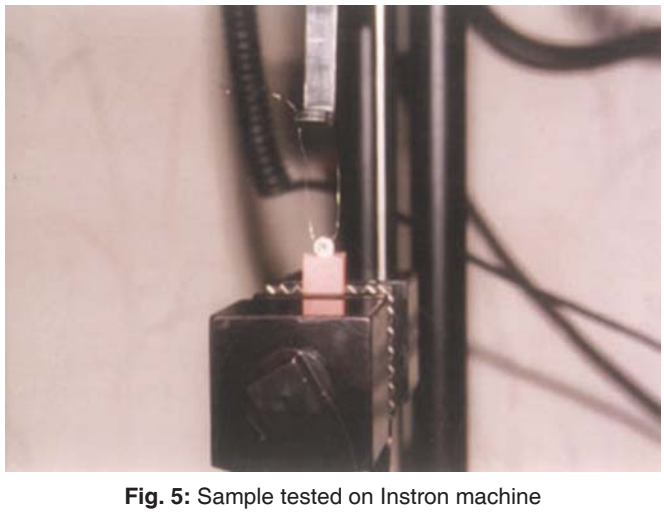


Fig. 5: Sample tested on Instron machine

* Gemini brackets (3M, Unitek), ** Acta de Satelac, *** Instron Universal Testing Machine

(SNK) test. The variation in the bond strength in between the five groups were observed.

BOND SITE FAILURE

Adhesive Remnant Index

Adhesive remnant index,[5] developed by Artun and Bergland in 1984 is a 4 point scale which is used to assess the amount of the composite remaining on the tooth surface on debonding. This was used in this study to assess the amount of composite remaining on the tooth surface after debonding and was examined under ×10 magnification.

RESULTS

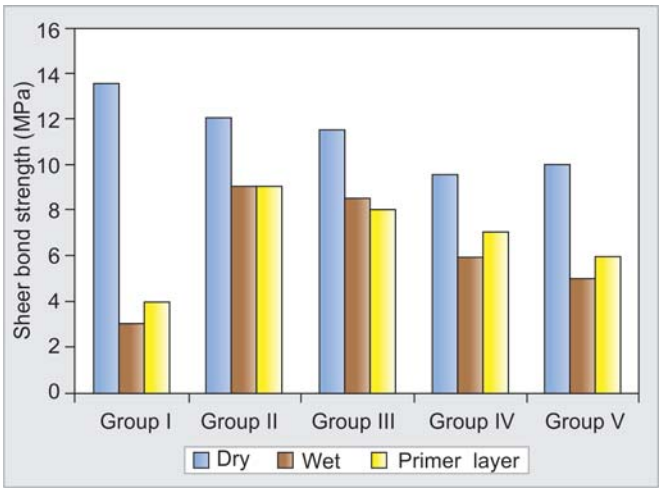
This study was performed on 180 premolars extracted for orthodontic purpose and free of caries, without enamel damage. They were divided into five equal groups and each group was further subdivided into three groups, and color coded Instron universal testing machine set at a speed of 0.5 mm/minute was used to evaluate shear bond strength and the readings were expressed in Megapascals.

STATISTICAL INFERENCE

There is highly statistically significant difference in the mean values of MPa (Graph 1) among and within the groups ($p < 0.001$). The ANOVA results obtained for, within the groups were also found to be statistically significant.

DISCUSSION

Direct bonding of brackets has revolutionized orthodontic treatment but bonding such attachments using regular primers in moisture contaminated areas, like when bonding partially erupted teeth, lingual bonding, bonding impacted teeth, etc. in routine orthodontic bonding procedures is still difficult. Complete isolation cannot be obtained due to the presence of moisture. With the advent of hydrophilic bonding materials, successful orthodontic bonding on a moisture contaminated enamel surface is made easy. Initially hydrophilic primers were used for dentin bonding in restorative dentistry, but now hydrophilic enamel primers have been introduced in orthodontic bonding to display moisture from the enamel surface, isolated for bonding.



Graph 1: The bond strength in MPa by the five groups

To our knowledge, very few studies[6-10,33] have been conducted with bonding agents that are suitable for orthodontic bonding, in moisture or saliva contaminated areas.

The aim of this study was to evaluate and compare the shear bond strength of newly introduced hydrophilic materials. In the present study, 180 extracted human upper and lower premolar teeth were taken and were divided into five main groups. Each group contained 36 teeth, which were further subdivided into three subgroups containing 12 teeth each in order to generate a meaningful data.[11,12] Various storage media have been used to store teeth like water, saline, artificial saliva, etc. and in this study, all teeth were stored in distilled water at 37°C as suggested by Nigela A Fox,[27,33] as the storage media does not alter the bond strength.

In the present study rubber cup prophylaxis with pumice was done on all the teeth.[13]

Various solutions have been proposed for acid etching–Buonocore[1] first introduced phosphoric acid in 1955 to increase the adhesion of acrylic filling material to the tooth surface. Polyacrylic acid,[14,15] nitric acid,[16] maleic acid[17] and sulfuric acid[5] have all been tried for etching and it has been found that the depth of etchant at all concentrations were significantly less than that of phosphoric acid despite producing surface regularities. Laser etching was also compared to chemical etching and was considered inferior to etching done with orthophosphoric acid as suggested by Akhildo et al.[18] As orthophosphoric acid is the standardized acid used, the same has been used in this study to etch the enamel surface.

After the introduction of acid etching technique introduced by Buonocore[1] in 1955 it was GV Newman[19] who first bonded orthodontic brackets to teeth by employing acid etch technique.

Several studies[20,21] have been done to evaluate the etch time. The etch time ranges from 15 to 120 seconds. Several authors observed that there was no significant difference in the bond strength when phosphoric acid was employed for a time period of 15 or 60 seconds.[20-23,12] When 120 and 60 seconds of etch time was compared, it was found that with 120 seconds etch time there was increased enamel loss and decreased bond strength.

In the present study, 37% orthophosphoric acid gel was used to etch the enamel surfaces of the teeth for about 60 seconds, as it provided better control than the liquid as recommended by Beech and Jalay.[24] Later after rinsing, the teeth were bonded using five different hydrophilic bonding materials.

According to the manufacturers,[4] MIP literally means 'not physically or chemically sensitive to small quantities of moisture'. Clinically it means, 'after rinsing and thoroughly removing the etchant, drying a tooth surface before priming is not required'. It is made up of HEMA, to attain moisture insensitivity and BIS-GMA for bond strength. It is unfilled to help penetrate the etched surface of tooth. Therefore, it was used in this study to bond on contaminated enamel surfaces of the teeth. The other materials which have been advocated for bonding in moist environment are Assure primer, Enhance LC and Prime and Bond NT which are supposed to possess more or less the similar properties like MIP according to the product literature available on MIP,[4] and hence were selected for comparison with MIP. In this study, two different types of primers were used namely, ethanol-based and acetone-based primers. Ethanol has the property to diffuse very easily into water acetone-based primers do not easily get diffused in water as far as enamel bonding is concerned and thus produce lesser bond strengths. But, when used on dentin they produce higher bond strength values due to more moisture content in dentin.[25] MIP, being an ethanol-based primer, gets diluted more easily when used on a wet enamel surface unlike when compared to acetone-based primer like Prime and Bond NT.

The teeth were bonded in three different surface environments namely dry, contaminated with artificial saliva and reprimed after contamination with artificial saliva as suggested by Mark J Webster.[8] The brackets were bonded to the tooth surface and cured. A visible light cure unit (Acta de satelac) was used in the present study, which emitted light at a wave length of 468 nm[26] and the specimens were cured advocated by Mark J Webster.[8] After bonding, the teeth were stored in distilled water at 37°C for 48 hours.

Various debonding methods can be used to debond a bracket from a tooth using opening pliers, Perry 1980,[3] chateltton model DTC Universal Tester Newman,[19] and the MTS testing machine NigeIa-1980[27,28] which are similar to the Instron machine. In the present study Instron universal testing machine (4501) was used to assess the shear bond strength.[11] Bond strengths were accurately calculated and expressed as MPa, after the assessment of the bond surface area, which was found to be 10.611 mm^2. Clinically acceptable bond strengths have been reported to range from 6 to 8 MPa.[29] These bond strengths are considered to be able to withstand masticatory and orthodontic forces. Bonding in presence of moisture, also in this study, produced values which were well above the clinically acceptable bond strength values indicating the use of these hydrophilic bonding materials in contaminated environments as depicted in various studies.[8-10,9,34,35]

The bond strength of Transbond XT with MIP showed greater bond strength when compared to the rest of the materials. Assure also showed bond strength which was less than that of MIP and the difference was found to be statistically insignificant. There is a wide variation in the MPa in the literature. Although most of the studies were done *in vitro*, the reason for the variation may be due to the fact that in Mark J Webster's study, the study was conducted on bovine enamel. However, the low MPa could be due to the fact that the teeth which were used for the study were contaminated with fresh human saliva, where as in this study human premolar teeth were selected which were contaminated with artificial saliva, this could be the reason for the variations found in the bond strengths of the materials used in this study. Thus the different bond strengths obtained in these studies could have been due to the differences in the fields of bonding, testing and handling the materials by the operator.

In this study, when the primers were bonded to a surface that was contaminated before placing the adhesive, showed a mean bond strength value which was lesser than that of the groups where in the contaminated surfaces were reprimed before bonding. Slightly higher bond strength values were obtained for the groups in which contaminated surfaces were reprimed before the bracket placement. This indicated that, the reapplication of primer provided an acceptable bond strength and similar observation has been reported by Mark J Webster and Manville G.[8] The bond strength obtained with all the five types of primers in this study, in dry conditions were found to be higher than the other two surface treatments. The values obtained for MIP in this study compare well with that of the previous studies.[9,10,30,36]

The results obtained from this study were subjected to statistical analysis. Ideally the bond between the bracket and enamel is unique as it is temporary because it should be intact till the completion of the orthodontic treatment and hence must be able to withstand the heavy masticatory forces and forces of occlusion but it must also be debonded with minimal trauma to the enamel and with less clean-up procedures.

The residual resin on the tooth surface after debonding was evaluated with adhesive remnant index (ARI), developed by Artun and Bergland.[18]

Various studies have shown that the ARI score depends on:

- The type of adhesive used
- Position of the tooth within the arch
- Method of bracket removal
- Bracket base material.

When the ARI score was done for the present study, it was observed that, among all the 5 groups, the teeth which were bonded in dry environment had the highest ARI scores whereas, the teeth that were bonded under the surface treatment 2, that is the teeth which were contaminated with artificial saliva[31] had the lowest ARI score and the teeth which were bonded under the surface treatment 3 (after repriming) had the next lowest ARI score. The ARI scores obtained in this study are similar to that of the ARI scores obtained by Mark J Webster[8] and Ross S Hobson.[10] Therefore highest ARI scores were obtained for the teeth under the surface treatment l, the next lower scores were for the teeth under the surface treatment 3 and the lowest scores were for the teeth under the surface treatment 2.

Due to the limited study period, this study was done on maxillary and mandibular human premolar teeth and it was not tested on other teeth like, incisors and molars where the chance of moisture contamination is very high. Only the buccal surfaces of the premolar teeth have been taken into consideration and the study must also be performed on lingual surfaces to evaluate the bond strength when attachments other than brackets are used for bonding, especially with wires that are used as fixed lingual retainers. The forces like masticatory and occlusal stress could not be simulated as present in the clinical situations, therefore, further studies should be done under *in vivo* conditions to assess the bond strength of this material. The teeth collected were not from a particular age group, hence the age of the patient was not taken into consideration in this *in vitro* study. Studies should be performed in young permanent teeth, partially erupted teeth to determine the bond strength on such teeth because of the presence of a prismless layer on these teeth, which will reduce the retention, according to studies[32] by Der Hong, Sheen and Wei Nan. Moisture contamination is very common when bonding attachments to partially erupted young permanent or surgically exposed teeth.

The bond strength in older permanent teeth is greater than that of the younger teeth because recently erupted teeth are completely covered with pronounced perikymata and rod-ends. With age, the perikymata and rod-ends may wear away. As a result of age changes in the organic portion of enamel, presumably near the surface, teeth may become harder and thereby reinforce the bond strength. Therefore, this material should be tested on young permanent and erupting teeth, as these teeth are covered by a prismless layer thus indicating reduced retention when compared to the permanent teeth.

In vitro studies provide very important data concerning the physical and mechanical properties of a material, but the final evaluation can only be provided when you assess these materials under clinical conditions. Due to the limited study period, the bond strength of MIP could not be tested clinically, and hence further clinical studies should be performed in order to evaluate the performance of this material.

CONCLUSION

In the present study, bond strength of enamel was tested using, five different hydrophilic bonding systems, under three different surface treatments.

From the present study, it can be concluded that:

- Noncontaminated enamel surfaces had the highest bond strengths for both the hydrophilic and hydrophobic materials.[37]
- When the contamination occurred after the primer had been placed and cured as in treatment 3 (repriming after artificial saliva contamination) then a simple drying and reapplication of primer rendered adequate bond strengths.
- The different hydrophilic bonding systems used in this study, showed improved bond strengths, with reapplication of primer after saliva contamination.

Clinically acceptable bond strengths have been reported to range from 6 to 8 MPa. These bond strengths are considered apt to withstand masticatory and orthodontic forces. In this study, mean bond strengths were well above this minimal requirement, and all combinations of bonding adhesives, with or without saliva contamination, resulted in sufficient bond strengths except for Transbond XT in contaminated conditions. However, there were variations in bond strengths with each adhesive and surface treatment used in this study.

The results of this study reveal that in situations in which moisture contamination is critical there is a distinct advantage in using a primer that enhances the bond strength. Both the ethanol-based primers evaluated in the study viz MIP with Transbond XT and Assure offered sufficient bond strength. However, the acetone-based primer and adhesion booster did not offer sufficient bond strength under contaminated conditions for clinical use.

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ABOUT THE AUTHORS

C Nirupama (Corresponding Author)

Reader, Department of Orthodontics and Dentofacial Orthopedics Karpaga Vinayaga College of Dental Sciences, Kodambakkam Chennai, Tamil Nadu, India, e-mail: niruortho6@yahoo.com

Sarasa Kavitha

Lecturer, Department of Pedodontics, Karpaga Vinayaga College of Dental Sciences, Chennai, Tamil Nadu, India

Josy Jacob

Reader, Department of Pedodontics, Karpaga Vinayaga College of Dental Sciences, Chennai, Tamil Nadu, India

K Balaji

Professor, Department of Orthodontics, Tagore Dental College Chennai, Tamil Nadu, India

B Srinivasan

Reader, Department of Orthodontics, Karpaga Vinayaga College of Dental Sciences, Chennai, Tamil Nadu, India

Ramakrishnan Murugesan

Reader, Department of Orthodontics, Kalinga Institute of Dental Science, Bhubaneshwar, Odisha, India

NR Krishnaswamy

Professor and Head, Department of Orthodontics and Dentofacial Orthopedics, Ragas Dental College and Hospital, Chennai Tamil Nadu, India