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An In Vitro Evaluation Of Shear Bond Strength Of Two Self Etching Primers At Two Different Time Intervals.

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Abstract

Objective : To evaluate the clinical efficacy of a new restorative material Clearfil S3 bond, SEP for orthodontic bonding in comparison with Transbond plus SEP at two different time intervals.

Methodology : The present study was planned to evaluate the clinical efficacy of a new restorative material Clearfil S3 bond, in comparison with TPSEP at two different time intervals (30 minutes and 24 hours).Stainless steel brackets (Gemini 3M) were bonded on extracted premolar teeth using the above two mentioned SEP's, Transbond XT (3M UNITEK), a BISGMA based composite was used as a common adhesive. Shear bond strength and debonding characters of the two materials were compared.

Results : It can be seen that the mean SBS of Clearfil S3 bond is marginally higher both at 30 minutes (4.8 ±0.63 MPa) and 24 hours (8.5 ±0.59 MPa) time of debonding compared to that of TPSEP (4.43±0.46 MPa; 7.0 ± 0.42 MPa). The difference noted was statistically significant (p= 0.05%).

Key Words
Clearfil S3 bond, Transbond plus SEP, Self Etching Primers, Shear Bond Strength.

Introduction

Over the last half decade, the bonding of various adhesives to enamel and dentine has made a niche in nearly all areas of dentistry including orthodontics. The increase in contact area between the two surfaces, results in more interlocks and, thus greater adhesive forces.[1] The clinical significance of using these microscopic interlocks for bonding followed the introduction of the acid etch technique by Bunocore in 1955 and revolutionizing modern adhesive dentistry.

Although these newly innovated Self Etching Primers (SEP) were initially developed for use on dentine, it has been reported that adhesive system combining conditioning and priming (sixth generation bonding system) can be used successfully to bond orthodontic brackets on to the enamel surface[2],[3],[4]. The main ingredient of SEPconsists of methacrylated phosphoric acid ester (etchant and primer). Their composition also includes initiators, stabilizers, fluoride complexes and water.

As a later innovation, in the SEP group, a new 'no mix' SEP was introduced. This reduced technique sensitivity by eliminating the chance of improper mixing and the resulting inadequate SEP activation further reducing the chair side time. The main ingredient of this SEP is 10Methacryloyloxydecyl dihydrogen phosphate (MDP) and other constituents of this SEP are comprised of Bisphenol A diglycidylmethacrylate (BisGMA), 2-Hydroxyethyl methacrylate (HEMA) (hydrophilic), Ethyl alcohol, Water and Silanated colloidal silica[5].

Clearfil S3, used for restorative purpose, has recently been considered as one step SEP in orthodontics. Clearfil S3was evaluated in comparison with Transbond plus SEP, whose Shear bond strength (SBS) was determined at two different time intervals, half an hour after bonding when the initial arch wires are placed and 24 hours from the time of bonding when the adhesive would achieve most of its bond strength.

Material And Method

Our study comprised of 120 freshly extracted upper premolar teeth, which were stored in 0.2% wt/vol thymol solution to prevent bacterial contamination and dehydration.

Teeth included were anatomically and morphologically well defined, non-carious with intact buccal enamel, which were extracted for orthodontic purpose, Further the teeth were subdivided into four groups which were color coded and bonded with different material at different time as follows

- Group A1White (30teeth) bonded with transbond plus for 30 minutes
- Group A2 Blue (30teeth) bonded with transbond plus for 24 hours
- Group B1Green (30teeth) bonded with Clearfil S3bond for 30 minutes
- Group B2 Pink (30teeth) bonded with Clearfil S3bond for 24 hours

The teeth were mounted on, color coded acrylic blocks, and were then bonded with metal brackets using two different SEP's according to their respective groups as mentioned above. The buccal surface of the teeth was polished with pumice slurry using rubber cup and washed with distilled water and dried.

Bonding procedure using Transbond plus SEP and Transbond XT (Adhesive): Group A1 and A2 -

Transbond Plus SEP is a dental adhesive system developed for orthodontic bonding dispensed in the form of a single use, foil package (L-Pop system) and comprises of three compartments **(Fig 1)**. The first compartment contains methacrylated phosphoric acid ester which is the main ingredient, initiators and stabilizers; the second compartment

Fig 1 : Transbond Plus Sep

contains water, fluoride complex and stabilizers. For activation the contents of the two compartments are folded squeezed into the third compartment and the resultant mix is directly applied to the enamel surface and then rubbed with the applicator provided. This is followed by one to two seconds of gentle air burst. No rinsing was required after application, then the bracket was bonded. The adhesive was cured for 10 seconds

Bonding procedure using Clearfil S3 SEP and Transbond XT (Adhesive): Group B1 and B2-

It was directly applied from the bottle to the enamel surface with the help of an applicator brush **(Fig 2)**. The SEP was left in place for 20 seconds, followed by drying with high pressure air for 5 seconds and then cured for 10 seconds. The adhesive was then applied to the base of the metal bracket directly from the

Fig 2 : Clearfil S3 Bond

syringe and cured. All the bonded teeth were kept in distilled water at room temperature after which Group A1 (n = 30) to be debonded after 30 minutes, and bonded teeth of Group A2 (n = 30), were debonded after 24 hours respectively for Groups B1and B2

Evaluation of Bond Strength:

SBS was tested with a UNIVERSAL INSTRON Testing Machine and was evaluated according to the following procedure for all the four groups. The machine has two vertically placed jaws. **(Fig. 3)**.

- The acrylic block with the tooth embedded was placed in the lower jaw (Fixed head)
- A custom made metal chisel (blade) of 10 cm length, 1cm width and 1mm thickness at the tip of the chisel used to debond the bracket was fitted to the upper jaw of the machine (Movable head)

An occluso-gingival force was applied to each bracket producing a shear force at the bracket/tooth interface at a crosshead speed of 1mm/min.

The INSTRON unit was attached to an electronic console that displayed the debonding forces acting on the bracket tooth interface. Thus, the exact force at which the bracket debonded was noted from the console. This force was expressed in Newton's.

To evaluate the SBS in MPa, from the force value, the following formula was used,

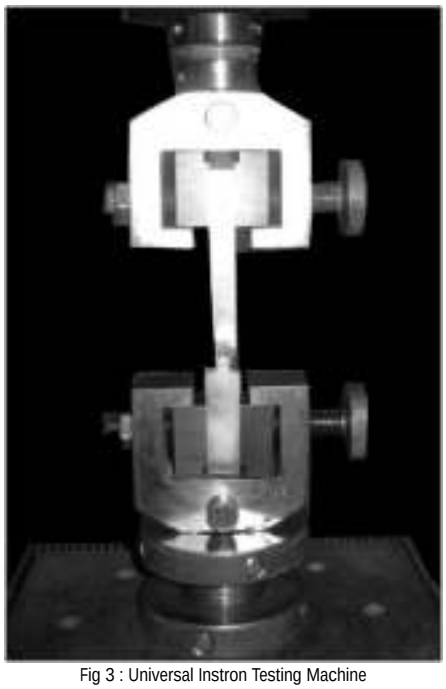

Fig 3 : Universal Instron Testing Machine

$$SBS = \frac{\text{Force in Newton's}}{\text{Area of the bracket base (sq. mm.)}} \text{ (Mpa)}$$

The area of the bracket base was measured by using Digital Vernier Calipers (12.654mm2). **(Fig3a)**

Results

SBS

IndividuallyTPSEP and Clearfil S3 exhibited lower mean SBS at 30 minutes time interval compared to mean SBS at the end of 24 hours.

The Mean, Standard deviation and Range of SBSof Clearfil S3 is marginally higher both at 30 minutes and 24 hour time of debonding compared to TPSEP {Clearfil S3 4.89±0.63MPa (30 minutes); 8.56±0.59MPa (24 hours)} {TPSEP 4.43±0.46MPa (30 minutes); 7.0 ± 0.42 MPa (24 hours); The difference noted was statistically significant (p= 0.05%). **(Table I)**

Table I: Mean Shear bond strength of Transbond Plus SEP (Group A) and Clearfil S3 (Group B) at 30 minutes and 24 hrs

Time	Shear Bond strength						Significance
	TP SEP (Group A)			CLEARFIL S3 (Group B)			
	Mean (MPa)	S.D	Range	Mean (MPa)	S.D.	Range	
30 Min	4.43	0.46	3.1-5.2	4.89	0.63	3.9-6.1	p= 0.05* (significant)
24 Hrs	7.0	0.42	6.0-7.8	8.56	0.59	7.4-9.8	p= 0.05* (significant)

*Statistical significance tested using Independent 't' test

Statistical Analysis

Following statistical analysis were used

- Mean, Standard deviation and Range
- Student's Independent t-test

Discussion

Clinically, etching the enamel surface, with 37% phosphoric acid for 15 seconds, creates micro porosities within the enamel and reduces surface tension that allows the resin to penetrate and polymerize within the etched enamel rods. Some by products like Calcium monophosphate and Calcium sulphate are formed which need to be washed away with copious water rinse.

Orthodontic bonding is a technique sensitive procedure, and moisture is cited as the most common cause for bond failure. Contamination causes plugging of porosities caused by acid etching and a reduction on surface energy. Thus the penetration of the resin is impaired and the micromechanical retention is compromised.[6]

SEP's were introduced in an effort to minimize enamel loss. In these SEP both the first and the second step of conventional bonding technique were combined together and the etchant and the primer were dispensed into a single acidic primer solution.

These SEP offer a distinct clinical advantage with reduced chair side time and reduced cross contamination. The reactive components are esters from bivalent alcohols with methacrylic and phosphoric acid and its derivatives. The phosphate residue is to etch the enamel, whereas the methacrylate component of the molecule is available for copolymerization with the bonding agent and the composite resin. There is no need to rinse off the reaction products or residual phosphoric acid esters because both are subsequently polymerized in to the bonding layer[2],[3].

The main ingredient of the self-etching primers is methacrylated phosphoric acid ester that dissolves calcium from hydroxyapatite. Rather than being rinsed away, the removed calcium forms a complex and is incorporated into the network when the primer polymerizes. Etching and monomer penetration to the exposed enamel rods are simultaneous and the depth of etch and primer penetration are similar[7].

The contents of the self etching primer are dispensed in the market in a form of a "lolly pop". It contains etchant and the primer in two separate compartments which require mixing prior to use. The mixed contents of the SEP are applied on the enamel surface by rubbing in a circular motion with the help of an applicator brush for approximately 3seconds.

A moisture-free air source is used to deliver a gentle burst of air to the enamel for 1-2 seconds. It was noted that the enamel surface appears uniformly shiny after the gentle air burst step. The bracket is then bonded onto the tooth surface.[5]

Studies have reported that SEP's are more efficient than conventional etching in terms of reduced dissolution of enamel, reduced chair side time coupled with clinically acceptable level of bond strength.

As a later innovation, a new SEP was introduced which offers the advantage of not having to be mixed prior to application. This in turn may reduce technique sensitivity by eliminating the chance of improper mixing and the resulting inadequate SEP activation, in addition to further reducing the chair side time.

Clearfil S3 used for restorative purpose has recently been considered in orthodontics. It is based on a new "Molecular Dispersion Technology"[8] with reported high bonding abilities in restorative dentistry. Thisnew technology combines hydrophobicand hydrophilic components in oneconstantly homogeneous solution. This solution does not crystallize out, and is dispensed in a single bottle system. It contains 10-Methacryloyloxydecyl dihydrogen phosphate (MDP) Bis-phenol A diglycidylmetha- crylate (Bis-GMA) 2-Hydroxyethyl methacrylate (HEMA), Hydrophobic Dimethacrylate Camphorquinone, Ethyl alcohol, Water and Silanated colloidal silica.

One major ingredient of S3Bond is the "MDP" monomer. The special molecular structure of MDP enables the bonding agent to decalcify, penetrate and create a chemical bond with calcium (ion) and hydroxyapatite crystals present in the enamel structure simultaneously.

MDP creates a chemical bond with the hydroxyapatite. The pH of Clearfil S3 is 2.7, yet it results in adequate enamel bond strength. According to the manufacturer, this chemical bond with Clearfil S3 Bond is also known to resist hydrolysis. Therefore, it is likely to be less affected by hydrolysis from saliva in the enamel, thus showing reliable and durable adhesion.[8]

Bond strength should be adequate to withstand masticatory forces and those generated by treatment mechanics, while at the same time permitting bracket removal without damage to enamel surface and facilitate easy cleanup.

The present study is done to evaluate the clinical efficiency, of a restorative 'no mix' self etching primer Clearfil S3), recently introduced in the field of orthodontics for bonding. The objective of this study is to evaluate the SBS and debonding characteristics of Clearfil S3 and compare it with a widely used self etching primer (Transbond Plus SEP) in orthodontics at two different time intervals (30 minutes that is, at the time the initial arch wires are ligated and 24 hours from the time of bonding when the adhesive has achieved most of its bond strength).

Transbond XT, a commercially available (3M Unitek); BISGMA based composite is the most commonly used peer reviewed adhesive with clinically acceptable bond strength compatible with different primers. While the manufacturers recommend the use of Transbond XTfor Transbond Plus SEP, the Clearfil S3 can be used with any light cure adhesive combination and thereforein the present study, Transbond XT was used as the adhesive for bonding with both TPSEP and Clearfil S3.

A total of 120 premolar teeth were divided in to two groups consisting of 60 samples each. Group A was bonded using Transbond Plus and Group B will be bonded using Clearfil S3. These two groups were further subdivided into groups A1and A2(TPSEP); B1and B2 (Clearfil S3) debonded at 30 minutes and 24 hours respectively.

In vitro bond strength testing is carried out by applying debonding force through the universal testing machine either in a shear or tensile loading mode.[9]

Various crosshead speeds ranging from 0.5mm to 5.0mm have been reported for applying debonding force. Studies[10] have reported that the results of mean bond strength values can decrease with increased cross head speed. A cross head speed of 1.0mm was used to during debonding in the present study as recommended by Fox.[11]

The mean SBS of all the four groups were statistically analyzed to determine, if there was any significant difference between the materials under study. The result showed that the mean SBS of both

Transbond Plus and Clearfil S3 are distinctly lower when debonded at 30 minutes compared with that of 24 hours and the difference was statistically significant. Further it was also noted that Clearfil S3 had marginally higher mean SBS both at 30 min (Clearfil S3 : 4.89 ± 0.63 MPa; TPSEP 4.43±0.46MPa) and 24 hour (Clearfil S3 8.56±0.59MPa ;TPSEP 7.0 ± 0.42 MPa) time of debonding compared to that of TPSEP which was statistically significant.

The mean SBS noted in the present study for both the SEP's debonded after 30 minutes and 24 hours is within the clinical acceptable range though the values at 30 minutes were low. Reynolds, in a review of orthodontic bonding, proposed that maximum bond strength of60-80kg/G(6-8MPa) would be required for successful clinical bonding, but those adhesives with invitrobond strength of approximately 50 kg/cm35 MPa "could be sufficient. The lower mean SBS values noted after 30 minutes of debonding would imply that lighter forces be applied at the time of initial arch wire placement.

There are only few other studies,[12],[13] which have evaluated Clearfil S3 and TPSEP. They too have reported higher bond strength for Clearfil S3 as reported in this study, though different parameters were taken in to consideration for bond strength evaluation.

Bishara et al[12] studied the effect of modifying the self-etchant bondingprotocol on the SBS of Transbond Plus SEP and Clearfil S3. They suggested that only one light curing application is needed to successfully bond bracket with Clearfil S3. Later in another study by Ostby et al[13] evaluated the effect of pH of SEP's on the SBS. They reported that Clearfil S3 with the highest pH (SBS= 6.5 ± 1.9 MPa; least acidic; pH = 2.7) exhibited a higher bond strength when compared to TPSEP (SBS= 4.2 ± 1.9 MPa) with a pH close to 1.0.

Conclusion

Clearfil S3 had marginally higher mean SBS both at 30 minutes and 24 hour time of debonding compared to TPSEP. Clearfil S3 has an edge over TPSEP in terms of ease of handling since it is premixed and can be directly applied on the enamel surface and eliminates the possibility of improper activation of the SEP. The TPSEP needs mixing of the ingredients and should be rubbed on the enamel for 3 seconds. Thus, Clearfil S3 can be considered as a newer generation of SEP for orthodontic bonding with clinically acceptable SBS with added advantages of ease of handling, reduced chair side time, less dissolution of enamel and longer shelf life.

The conclusions drawn from this study however need to be substantiated through in vivo clinical studies and survival analysis.

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