

Scanning electron microscopic study of tensile bond strength with acidulated phosphate fluoride applied before and after acid etching: *In vitro*

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Abstract

Introduction: In the field of orthodontics for more than 60 years is the acid etch technique introduced by Buonocore in 1955 that has been used in the direct bonding of orthodontic brackets many studies have proven that topical fluoride is effective in increasing resistance to dental caries or enamel decalcification. These include pretreatment with topical fluoride before etching incorporation of fluoride in the etching solution, and topical application of fluoride in the etched surface before bonding. The mechanism by which fluoride reduces decalcification and caries has also been shown to increase the resistance of enamel to acids This study aims to evaluate the bond strength with acidulated phosphate fluoride before and after acid etching. Detect the changes with the scanning electron microscope on the enamel surface after debonding with the fluoride application before and after acid etching.

Materials and Methods: Sixty human premolar teeth were extracted were washed and immersed in a closed plastic box with physiologic saline solution to keep them free from fungal and bacterial contamination for period of 3 months. The teeth were randomly divided into three groups of 20teeth each for testing tensile strength The specimens were mounted on an acrylic block of dimension (4x2x1cm) to make it compatible to the jigs of the Hounsfield universal testing machine The specimens of Group 1 (Control Group) the etchant solution was applied on the buccal surface of each crown for 15 seconds then rinsed with an abundant spray of water for 10 seconds and dried with an air spray The etched surface became chalky white in appearance. In the group 2 the teeth were immersed in APF gel (Vishal Pharma Ahmedabad) for 4 minutes. The treated teeth were removed from the APF gel and left untouched for 30 minutes. Subsequently the gel was washed off with a 1 minute water spray. The enamel of each tooth was etched for 15 seconds with acid solution for bonding.

In the third group the etchant was applied for 15 seconds, rinsed and dried as in the control group. However the crowns of the teeth were immersed in APF gel for 4 minutes after etching. The buccal surfaces of the teeth were abundantly washed with a water spray for 10 seconds then dried again etched enamel and bracket base were coated with a sealant and the composite resins (3M Unitek Co, USA) immediately applied to the bracket base. The brackets were accurately pressed to the demarcated etched buccal enamel with a placement scalar. To test the tensile strength the acrylic blocks with Hounsfield Universal Testing Machine specimen tooth was mounted the force at which the bond failed was recorded as breaking load and the bond strength The debonded interface in each group were observed with an SEM. The samples were coated all over to a thickness of about 20pm with gold as the conducting material.

Results: The mean tensile strength of control group (Group 1) 7.15 MPa
Group II (APF application before acid etching 7.015 MPa and
Group III. APF application after acid etching 5.26 MPa

Conclusion: The results showed that there is no statistically significant difference in bond strength with the APF application before acid etching but there is significant decrease in bond strength with APF application after acid etching compared to the control group.

In the SEM APF gel produced surface coating appeared to consisting of densely packed small spherical globular structure present in before and after acid etching.

Keywords: scanning electron microscopic study, tensile bond, acidulated phosphate fluoride, after acid etching

Introduction

One of the most significant development in the field of orthodontics for more than 60 years is the acid etch technique introduced by Buonocore in 1955 ^[20] that has been used in the direct bonding of orthodontic brackets. However in patients with poor oral hygiene decalcification and decay have been observed around the bonded brackets. Many studies have proven that topical fluoride is effective in increasing resistance to dental caries or enamel decalcification. These include pretreatment with topical fluoride before etching ^[19], incorporation of fluoride in the etching solution, and topical application of fluoride in the etched surface before bonding. Acidulated phosphate

fluoride increased the uptake of fluoride by the enamel at rates higher than either stannous fluoride or neutral sodium fluoride the mechanism by which fluoride reduces decalcification and caries has also been shown to increase the resistance of enamel to acids, increase the maturation rate of enamel and interfere with the metabolism of micro-organisms. The fluoride deposits in hydroxyapatite to form fluorapatite. However the fluorapatite may affect the bond strength or debonded interface.

Hence This Study Aim To

1. Evaluate the bond strength with acidulated phosphate fluoride before and after acid etching.

- Detect the changes with the electron microscope on the enamel surface after debonding with the fluoride application before and after acid etching.

Materials and Methods

Teeth

Sixty human premolar teeth were extracted from teenagers (9 to 16 years of age). After extraction the teeth were washed and immersed in a closed plastic box with physiologic saline solution to keep them free from fungal and bacterial contamination for period of 3 months.

Brackets

Stainless steel mini-mesh curved Beggs bracket with the bracket base measurement of 3mm X 3mm (256 – 650C) manufactured by TP orthodontic Inc (USA) were used in this study.

Method

The teeth were randomly divided into three groups of 20teeth each for testing tensile strength

Mounting of specimens

The specimens were mounted on an acrylic block of dimension (4x2x1cm) to make it compatible to the jigs of the Hounsfield universal testing machine.

Bonding Procedure

The buccal surfaces of the crown were cleaned with pumice using polishing brush for 10 seconds then washed with water and dried with an air spray.

The specimens of Group 1 (Control Group) the etchant solution was applied on the buccal surface of each crown for 15 seconds then rinsed with an abundant spray of water for 10 seconds and dried with an air spray. The etched surface became chalky white in appearance.

In the group 2 the teeth were immersed in APF gel (Vishal Pharma Ahmedabad) for 4 minutes. The treated teetch were removed from the APF gel and left untouched for 30 minutes. Subsequently the gel was washed off with a 1 minute water spray. The enamel of each tooth was etched for 15 seconds with acid solution for bonding. In the third group the etchant was applied for 15 seconds, rinsed and dried as in the control group. However the crowns of the teeth were immersed in APF gel for 4 minutes after etching. The buccal surfaces of the teeth were abundantly washed with a water spray for 10 seconds then dried again etched enamel and bracket base were coated with a sealant and the composite resins (3M Unitek Co, USA) immediately applied to the bracket base. The brackets were accurately pressed to the demarcated etched buccal enamel with a placement scalar.

De-bonding procedure

To test the tensile strength the acrylic blocks with Hounsfield Universal Testing Machine specimen tooth was mounted such that the bracket slot were parallel to the tooth surface. Stainless steel ligature wire (.009) was threaded through the bracket and passed and passed upward to the movable cross head and twisted tightly. So that the load would be applied directly over the centre of the base. The cross head speed of 2mm/minute was used. The force at which the bond failed was recorded as breaking load and the bond strength. The values were recorded and statistically

evaluated and analyzed as per student ‘t’ test.

Scanning Electron Microscopic Study

The debonded interface in each group were observed with an SEM. The samples were coated all over to a thickness of about 20pm with gold as the conducting material. The specimens were observed on the screen at various magnification and the photographs were taken for evaluation

Results

Sixty specimens were equally divided into here groups.

Group I - Control (Normal bonding)

Group II - II APF application before acid etching group.

Group III - APF application after acid etching group.

Twenty specimens of each group were used for testing tensile strength and. The Hounsfield universal testing machine was used for this study.

Breaking load at which bond failure occurred was recorded and the strength values were calculated for each specimen. The mean and standard deviation were found. Statistical analysis of the finding was done by applying the student ‘t’ test.

Table I shows the tensile strength of control and experimental specimens. The tensile strength was calculated using the following equation.

$$\text{Bond Strength} = \frac{\text{Breaking load}}{\text{Nominal area of bonding}}$$

The nominal area of bonding base was found to be 0.09 sq.cm

The mean tensile strength of control group (Group 1) 7.15 MPa

Group II (APF application before acid etching 7.015 MPa and

Group III. APF application after acid etching 5.26 MPa

Table 2 shows Paired t –Test for tensile strength

Table 1: Tensile Bond Strength (In Mpa)

S. No.	Group I	Group II	Group III
1.	7.2	7.3	5.6
2.	6.8	7.1	4.9
3.	7.5	6.8	6.1
4.	7.2	6.5	5.8
5.	7	7	5.4
6.	6.8	7.2	5.2
7.	7.3	6.2	6
8.	6.9	7.1	4.8
9.	7.4	6.6	5.2
10.	6.6	7	5.7
11.	7.6	7	4.6
12.	6.6	7.5	4.9
13.	7.5	7.3	4.7
14.	6.8	6.7	4.8
15.	7	6.5	5.8
16.	7.2	6.9	5.8
17.	7.4	7.5	4.8
18.	7	7.4	5
19.	7.5	7.1	4.9
20.	7.7	7.6	5.2
Total	143.0	140.3	105.2
Mean	7.15	7.015	5.26

Table 2: Paired ‘T’ Test for Tensile Strength

	Mean	SD	Df	SE	T - stat	P -Value	‘T’ TEST
Group I & Group II	7.15 7.015	0.334821 0.377352	19	0.074868 0.084379	1.197993	0.122825	Statistically not significant
Group I & Group III	7.15 5.26	0.334821 0.476169	19	0.074868 0.106475	14.54299	4.74 10 ⁻¹²	Statistically Significant
Group II & Group III	7.015 5.26	0.377352 0.476169	19	0.084379 0.106475	10.36728	1.4 10 ⁻⁹	Statistically Significant



Fig 1: Scanning Electron Microscopic Picture of Normal Bonding



Fig 2: Scanning Electron Microscopic Picture of Acidulated Phosphate Fluoride Application After Acid Etching

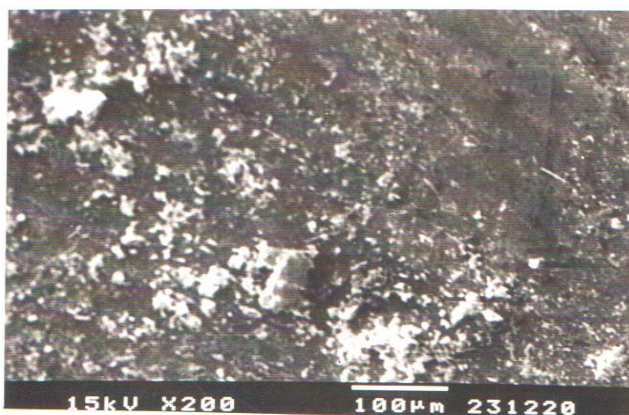


Fig 3: Scanning Electron Microscopic Picture of Acidulated Phosphate Fluoride Application Before Acid Etching

the different orthodontic elements make most of the patient find it difficult to brush properly even with excellent brushing habit.as a result plaque readily accumulates around the brackets and the acidic nature of this material enamel Demineralization and white spot lesion can occur. The dematerialized surface enamel is considered to be precursor or early lesion of enamel caries. The demineralization is not a continuous process. A series of cycles between demineralization and remineralization of enamel adjacent to orthodontic brackets presents in the clinical situation¹. Despite efforts to educate and motivate patients undergoing orthodontic treatment the presence of decalcification remains a problem because the preventive oral hygiene maintenance program rely heavily on patient compliance³. White spot lesion after orthodontic treatment with fixed appliances may present an esthetic problem even 5 years or more after the treatment This persistent problem has speculated a search for a preventive procedure program for the prevention of decalcification and caries one or more of the following factors are responsible for the prevention program⁹ fluoride has a remarkable ability to reduce the incidence of dental caries by increasing the resistance of enamel to acid, increasing the maturation rate of enamel and interfering with the metabolism of microorganism. Recent evidence shows that fluoride may facilitate the demineralization of white spot lesion. Regular use of fluoride tooth paste during fixed appliance therapy was not sufficient to inhibit lesion development adjacent to orthodontic brackets fluoride availability should be independent of patient cooperation and that the fluoride ion should diffuse or dissolve over a prolonged period. Even though different fluorides are used for topical application neutral sodium fluoride, stannous fluoride, acidulated phosphate fluoride are the three agents that are currently in use; In orthodontics topical fluoride are applied before acid etching, incorporation of fluoride in the etching solution and topical application of fluoride to the etched enamel surface before bonding as a preventive measure against decalcification. The acidulated phosphate fluoride which is selected as the material for this study has 1.23% of fluoride as sodium fluoride buffered to Ph of 3-4 in 0.1M phosphoric acids. The main advantage of APF is its ability to deposit fluoride in enamel to a deeper depth than neutral NaF and long term benefits. The short term benefit is that APF acts initially as a potent reservoir of fluoride and the long term effect is the formation of fluorapatite as the retained calcium fluoride layer is dissolved away from the enamel surface. The brackets used in this study were mesh brackets. Mesh based brackets were used as they are better than metal brackets with perforated bases. Reynolds and Fraunhoten have reported that metallic attachments with a coarse mesh are the most suitable type for direct bonding procedure For

Discussion

Proper oral hygiene compliance in orthodontic patients has always been difficult to maintain. The bands, brackets and

the purpose of study sixty teeth were divided into three groups 20 each for the measurement of tensile bond strength. The hours 10 field universal testing was used to record the breaking load. The bracket area was measured as 0.3 cm x 0.3 cm and calculated bracket base area was 0.09 sq.cm.

The tensile stress was calculated by using the formula.

$$\text{Stress} = \frac{\text{Breaking load}}{\text{Area of the bracket base}}$$

In the control (group I) the mean tensile strength calculated by using the above mentioned formula was found to be 7.15 MPa In the experimental group (group 2) APF application before acid etching and mean tensile strength was found to be 7.015 MPa In the experimental group (group 3) APF application after acid etching and mean tensile strength was found to be 5.26 MPa In this study Group II APF application before acid etching no significant difference were noted in the bond strength of treated and untreated group. However in group III APF application after acid etching the bond strength decreased compared to the control group. Group III APF application after acid etching the bond strength is lower than the control group. The topical fluoride fills the inter prismatic spaces occupied by $\text{Ca}_5(\text{PO}_4)_3$ and the formation of CAF_2 after etching capacity of adhesives. This reaction product could be the cause of a reduction in resin bond strength. In the SEM study APF gel produced surface coating appeared to consist of a uniformly thick layer of densely packed small globular particles present before and after acid etching. The recent research^[5, 15], indicate that the onetime preventive procedure at the time of bonding has little beneficial effect in reducing white spot formation but the regular and repeated use of the low concentration of fluoride reduces the incidence and severity of white-spot formation.

Hence the application of acidulated fluoride before acid etching may offer protection from decalcification or a caries attack without affecting the bond strength. But the application APF after acid etching will have an adverse effect on orthodontic bond strength of human enamel.

Conclusion

An experimental study was undertaken to evaluate the tensile and shear bond of orthodontic brackets with acidulated phosphate fluoride application before and after acid etching.

A SEM study was also conducted to detect the changes on the enamel surface after de-bonding with the fluoride application before and after etching.

The results showed that there is no statistically significant difference in bond strength with the APF application before acid etching but there is significant decrease in bond strength with APF application after acid etching compared to the control group.

In the SEM APF gel produced surface coating appeared to consist of densely packed small spherical globular structure present in before and after acid etching.

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There are no financial support and conflicts of interest

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