Interference of preventive caries system with microshear bond strength of enamel surface bonded to etch &rinse or self etch adhesive system with nanofilled composite

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Abstract: Purpose: To evaluate the microshear bond strength of self etch and etch &rinse adhesives of nanofilled composite on enamel substrates after fluoride application. **Materials and Methods**: Forty Enamel samples were obtained from human premolars and randomly divided into 2 groups (n = 10) according to enamel substrates treatment first group samples are painted with fluoride varnish for 20 min and stored in artificial saliva for 24 hours , then divided into 2 subdivisions : first subdivision is treated with etch & rinse adhesive then. 2nd subdivision samples are treated with self etch adhesive. The second group divided into 2 subdivision : first subdivision are treated with etch & rinse adhesive. 2nd subdivision samples are treated with self etch adhesive. All prepared samples ,Prior to adhesive curing, a hollow cylinder (2.0 mm height/0.75mm internal diameter) was placed on the treated surfaces. A nanofilled resin composite was then inserted into the tube and cured. After artificial saliva storage for 24 hrs, the tube was removed and microshear bond strength was determined in a universal testing machine at a crosshead speed of 0.5 mm/min. **Results:** The mean and standard deviation values of micro-shear bond strength were 15.9 ± 4 MPa and 9.7 ± 3.8 MPa for normal and fluoridated enamel, respectively. Normal enamel showed statistically significantly higher mean micro-shear bond strength than fluoridated enamel. **Conclusions**: The microshear bond strength decrease in cases of recently fluoridated enamel.

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Key Words: preventive therapy, microshear bond strength**,** adhesion , nanofilled composite.

1. Introduction

Improvements in dental adhesive technology have extensively influenced modern restorative dentistry. Nowadays, extension for prevention' proposed by GV Black[1] in 1917 is no longer has any explain, also it has been replaced by the concept of 'minimal invasive dentistry'[.2] This modern approach focuses on the achievement of a more conservative cavity design. The subsequent restorative procedure relies on the bonding effectiveness of adhesive materials such as resin composites, which do not require the removal of sound dental structure for additional mechanical retention. Although these restorations tend to fulfill the main requirements of a more conservative and aesthetic treatment, their clinical longevity is still a topical issue, mainly due to the degradation of the adhesive interface over time.[3]

Initial enamel caries lesions are usually not treated operatively to avoid the sacrifice of sound hard tissues [4].Thus, preventive action at an early stage is important to prevent caries development. The maintenance of oral hygiene in conjunction with dietary advice , fluoride therapy and may prudent use of pit-and-fissure sealants has been shown to be a reliable preventive strategy in these populations [5].

In order to obtain long-term clinical success, the integrity of composite -enamel bond are important and the criteria for successful prevention of leakage of bacteria and oral fluids that initiate caries [6,7].

Recently, the microshear bond test has been developed as an alternative to the microtensile test [8]. The microshear bond test was further developed by Shimada and his group, who have researched the shear strengths of enamel and resin-based adhesives [9].

The objective of the present study was to compare the resin-enamel bond strength of etch &rinse and self etch adhesives of fluoridated and normal enamel. The null hypothesis was that there was no difference between the self-etching materials and etch-and-rinse adhesive in their bond strengths

2.Materials and Methods

I- Preparation of the Samples

A total of 40 caries-free permanent premolars, which were extracted for orthodontic reasons, were used in this study. Individual tooth surfaces were hand scaled to remove any remaining soft tissue. All teeth were stored in distilled water at -20°C. Crowns were separated from the roots 2 to 3 mm apical to the cementoenamel junction using a diamond saw (Isomet, Buehler, Lake Bluff, IL, USA) under water irrigation at a low speed. With their labial surfaces exposed, the crowns were then embedded in self-curing acrylic resin (Meliodent, Heraeus Kulzer, Dormagen, Germany) into Teflon molds. The convex enamel surfaces on the outermost buccal surfaces were reduced up to 0.5 mm by gently polishing on a 600-grit silicone carbide paper under running water to prepare a flat enamel surface. All enamel surfaces were examined under a stereomicroscope (Leica, MZ 12, Leica AG, CH-9435 Heerbrugg, Switzerland), and any specimens with cracks or hypoplastic defects were excluded.

II- Sample classification:

The total 40 Specimens were randomly divided into two groups.

Group I : 20 specimens are painted with fluoride gel for 30 minutes , rinsed and stored in artificial saliva for 24 hours Group II : 20 specimens are not painted with fluoride gel.

Each group is subdivided into 2 subdivisions

1st subdivision is treated with etch &rinse adhesive

2nd subdivision is treated with self etch adhesive

III- Application of Bonding Agents and Preparation of Resin Composite specimens:

Enamel surfaces were cleaned with water spray for 5 seconds and dried with oil and water-free compressed air for 3 seconds. Details of bonding adhesives and composite are provided in Table 1 . Prior to application of the bonding resin on each specimen, hollow cylinders 2.0 mm in height were cut from micro-bore tygon tubing (Norton Performance Plastic; OH, USA) with an internal diameter of 0.75mm and were placed on the treated surfaces. .

Each adhesive system was applied according to the manufacturer's instructions as follows:

Etch & rinse adhesives :

The enamel surface was etched (using Scotchbond) for 15 s with 37% phosphoric acid, and rinsed with water spray for 15 s. Excess water was removed with cotton pellet or mini sponge leaving the enamel moist. Bond (Adper Single bond 2)was applied with a disposable brush, 2 to 3 consecutive coats for 15 s with gentle agitation using a fully saturated applicator . Gently air thin for five seconds in evaporative solvents . light cured for 10 s using a halogen light source (Visulux curing unit, Vivadent; Schaan, Liechtenstein). The output of the light curing unit was regularly checked (500 mW/mm2).

Self Etch adhesives :

Adper Easy one : The adhesive was applied to the enamel surface for 20 s, blown with mild air for 5 s and light cured for 10 s. A nanofilled restorative composite (Filtek Z350 Enamel Shade A2, 3 M , USA) was carefully inserted into the tubing lumens and irradiated for 40 s according to the manufacturer's instructions. The specimens were then stored in artificial saliva at 37°C for 24 hrs. After removal from artificial saliva, the tygon tubing around composite cylinders was removed by gently cutting the tube into two hemi cylinders using a feather-edge blade.

IV- Microshear bond strength testing:

These tests were performed using NEXYGEN from Lloyd Instruments. Each acrylic embedded molar tooth with its own bonded composite micro-cylinders was secured with tightening screws to the lower fixed compartment of a materials testing machine (Model LRX-plus; Lloyd Instruments Ltd., Fareham, UK) with a loadcell of 5 kN and data were recorded using computer software (Nexygen-MT Lloyd Instruments). A loop prepared from an orthodontic wire (0.014" in diameter) was wrapped around the bonded micro-cylinder assembly as close as possible to the base of the microcylinder and aligned with the loading axis of the upper movable compartment of the testing machine.

A shearing load with tensile mode of force was applied via materials testing machine at a crosshead speed of 0.5 mm/min. The relatively slow crosshead speed was selected in order to produce a shearing force that resulted in debonding of the microcylinder along the substrate-adhesive interface. The load required to debonding was recorded in Newton.

Micro-Shear bond strength calculation;

- The load at failure was divided by bonding area to express the bond strength in MPa :

$$\tau = P/\pi r^2$$

where ; **τ** =bond strength (in MPa), **P** =load at failure (in N), **π** =3.14

r = radius of micro-cylinder (in mm)

A total of 40 bond strength values were recorded for two adhesives.

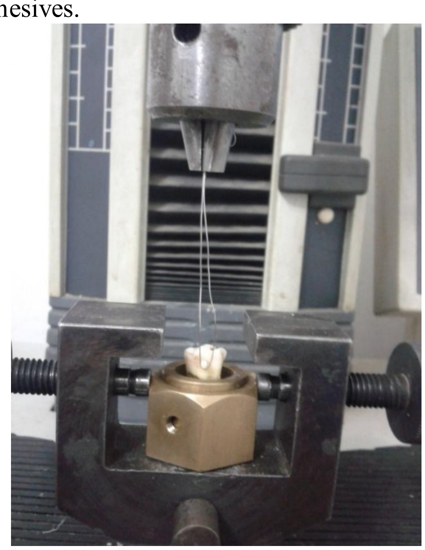

Fig.(1): lloyd universal testing machine with microshear bond strength sample.

Table (1) Composition, lot number, and manufacture of the tested materials.

Material	composition	Lot number	Manufacture
Topex DuraShield	5% sodium fluoride varnish	#0411131105	Sultan Health Care
Scotchbond etchant gel	35 % phosphoric acid	N 110268	3M ESPE
Adper Single bond 2	(10% colloidal nanofiller) BisGMA, HEMA, dimethacrylates, ethanol, water, a novel photoinitiator system and a methacrylate functional copolymer of polyacrylic and polyitaconic acids	N353034	3M ESPE
Adper Easy Bond	2-hydroxyethyl methacryate (HEMA) Bis-GMA Methacrylated phosphoric esters 1,6 hexanediol dimethacrylateMethacrylate functionalized Polyalkenoic acid (Vitrebond™ Copolymer) Finely dispersed bonded silica filler with 7 nm primary particle size Ethanol Water Initiators based on camphorquinone Stabilizers	434163	3M ESPE
Filtek Z350 XT	(20 nm silica filler 4-11 nm zirconia filler) as 72.5% by w filler bis-GMA, UDMA,TEGDMA , PEGDMA and bis-EMA resins	N339145	3M ESPE

V-Statistical Analysis:

Data were presented as mean and standard deviation (SD) values. Regression model using two-way Analysis of Variance (ANOVA) was used in testing significance for the effect of adhesive system, enamel condition and their interactions on mean micro-shear bond strength.

The significance level was set at $P \leq 0.05$. Statistical analysis was performed with IBM®SPSS® Statistics Version 20.

3.Results

Two-way ANOVA results

The results showed that enamel condition and the interaction between the two variables had a statistically significant effect on mean micro-shear bond strength.

Table (2): Regression model results for the effect of different variables on mean micro-shear bond strength

Source of variation	Type III Sum of Squares	df	Mean Square	*F*-value	*P-value*
Adhesive system	23.5	1	23.5	2.3	0.154
Enamel condition	134.3	1	134.3	13.4	0.004*
Adhesive system x Enamel condition	77.9	1	77.9	7.8	0.018*

df: degrees of freedom = (n-1), *: Significant at $P \leq 0.05$

Effect of enamel condition:

The mean and standard deviation values of micro-shear bond strength were 15.9 ± 4 MPa and 9.7 ± 3.8 MPa for normal and fluoridated enamel, respectively. Normal enamel showed statistically significantly higher mean micro-shear bond strength than fluoridated enamel.

Table (3): Comparison between micro-shear bond strength of the two enamel conditions regardless of adhesive system

Normal		Fluoridated		*P*-value
Mean	±SD	Mean	±SD	
15.9	±4	9.7	±3.8	<0.001*

*: Significant at $P \leq 0.05$

Effect of different interactions:

The statistically significantly highest mean micro-shear bond strength was found with (Self-etch & normal enamel). The statistically significantly lowest mean micro-shear bond strength was found with (etch & rinse & fluoridated enamel) and (Self-etch & fluoridated enamel) with no statistically significant difference between the two groups. From the interactions table, we can also conclude the following:

- **Detailed comparisons between adhesive systems:**
  - With normal enamel; self-etch showed statistically significantly higher mean micro-shear bond strength than etch & rinse.
  - With fluoridated enamel; there was no statistically significant difference between the two adhesive systems.
- **Detailed comparisons between enamel conditions:**
  - With total etch; normal enamel showed statistically significantly higher mean micro-shear bond strength than fluoridated enamel.
  - With self-etch; normal enamel showed statistically significantly higher mean micro-shear bond strength than fluoridated enamel.

Table (4): Comparison between micro-shear bond strength of different variables' interactions

Adhesive system	Enamel condition	Mean	SD	*P*-value
Total etch	Normal	12.3 [b]	1	0.018*
	Fluoridated	10.8 [c]	4.1	
Self-etch	Normal	19.5 [a]	0.8	
	Fluoridated	8.7 [c]	3.7	

*: Significant at $P \leq 0.05$, Different letters are statistically significantly different

4.Discussion :

The null hypothesis that there are no differences in the bond strength of the enamel substrates and different resin adhesive materials was rejected. In this study, differences were noted between the Microshear of normal enamel and fluoridated enamel to resin adhesive. The fluoride application of 5% NaF varnish decrease microshear bond strength of enamel to different types of adhesives.

The use of a varnish as a vehicle for topical fluoride application was chosen in this study due to its prolonged period of contact with the enamel surface to allow greater uptake of fluoride ions into the enamel and making it more resistant to demineralization [(10)].

Shimada and his group who have researched the shear bond strengths of enamel and resin-based adhesives. They also who developed microshear bond strength [(9)] . The microshear bond test is considered more useful for testing bond strengths to enamel, as the microtensile method is not easy to use on this substrate and there is a high probability that the enamel will be pulled off the dentin when a tensile stress is applied to such small specimens. Also the microshear bond test is less demanding in terms of specimen production, and bond test areas can be much better controlled by the use of known diameter microbore tubing.

Little is known about the microshear bond strength of resin composite to fluoridated enamel. In current study normal enamel showed statistically significantly higher mean micro-shear bond strength than fluoridated enamel, in accordance with other studies have indicated that topical fluoride application fills the interprismatic spaces occupied by $Ca_5(PO)_3$ and CaF_2 and reduces the bonding capacity of adhesives.[(11,12)] On the other hand, studies have shown that shear bond strength is not significantly different in groups with and without fluoride pretreatment [(13-15)] . In these studies, researchers saw globular structures only on the prism cores of ground enamel surfaces etched with H3PO4 containing higher fluoride concentrations; they did not observe adverse effects on the bond strength of bonding resin to etched enamel.

5. Conclusion:

It can be concluded that the microshear bond strength was significantly influenced by fluoride application of 5% NaF varnish . The microshear bond strength decrease in cases of recently fluoridated enamel .

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