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Evaluating the Effect of Collagen Removal on Shear Bond Strength of Excite Dentin Adhesive System (Using NaOCl Treatment) In Vitro Study

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ABSTRACT

Background: To evaluates the effect of collagen removal from acid etched dentin surface (using 10% sodium hypochlorite (NaOCl) for one minute on bond strength between composite resin and dentin surface using Excite adhesive system using two mechanism (wet and dry).

Material and Method: thirty sound human upper first premolars teeth recently extracted for orthodontic purpose were selected. 2mm from the buccal surface of the blocked tooth were grounded to obtain flat superficial dentin surface. The bonding systems were applied according to the following groups. (Each group consists of ten teeth), Group 1: Acid plus Excite bond, Group 2a: Acid/NaOCl (wet surface) plus Excite, Group 2b: Acid/NaOCl (dry surface) plus Excite.

Results: Shear bond strength is determined by using Instron testing machine and the results show that: There is a significant increase in shear bond strength after treatment of acid etched dentin with 10 % NaOCl for 1 min . There is no significant difference in the shear bond strength between dry and wet acid etched deproteinized dentin groups of the Excite adhesive system.

Keywords: *shear bond strength, sodium hypochlorite (NaOCl), collagen removal, Excite adhesive system.*

1. INTRODUCTION

Composite restorative materials were introduced to the dental profession in the early 1960s &quickly became the principal anterior restorative materials.

By following improvements in composite technology, these materials were also developed as aesthetic posterior restorative materials but were initially hampered by problems of polymerization shrinkage and wear resistance [(1)]. Commercial dentin bonding systems differ in chemistry &complexity of application, but most of them depend upon the removal or modification of the smear layer, demineralization of the superficial dentin & dentin penetration by a hydrophilic, bifunctional comonomer to form the hybrid layer

(2, 3).

The bonding mechanism is mainly based on micromechanical retention. The hybrid layer, resin tags & adhesive filling of lateral branches of dentinal tubules have been suggested as the essential mechanisms of adhesion [(4)].

Strong acid (37%. Phosphoric acid) at low pH denature proteins such as collagen, could leads to a change in their dimensions, or even to fragmentation. This may result in a less optimal surface for bonding compared to a surface with intact collagen fiber[(5)].

The presence of the collagen network resulting from acid –etching has been constantly questioned as an important structure for dentin bonding, since it has been shown that resin monomers do not fully diffuse through it to reach intact dentin [(6)]. This incomplete penetration produces a porous layer of exposed collagen subjected to hydrolysis & degradation [(7, 8)] by proteolytic enzymes [(9)], which were released from leukocytes, salivary glands, &bacteria in plaque [(10, 11)] resulting in microleakage &failure over time [(12)].

Sodium hypochlorite is a non–specific proteolytic agent that effectively removes organic compounds [(13)] and has been frequently applied for collagen removal after acid –etching with various and controversial results [(14, 15)].

Thus elimination of the collagen fibril might be essential for the increase of the longevity of resin restoration.

2. MATERIAL AND METHOD

Thirty-sound human upper first premolar teeth, recently extracted for orthodontic purpose (the patient's age range from 13-20 years) of comparable size &shape, were selected after examining all teeth using magnifying lens (X10) & by transillumination using polarization light to identify any visible cracks, then the teeth were cleaned from debris by using slurry of pumice in a rubber cup used in low-speed hand piece, then washed with distilled water.

The teeth were stored in deionized distilled water (DDW) in the refrigerator at 4C˚ until sample preparation [16].

The roots of the teeth were sectioned by diamond disc with workshop hand piece at the cement-enamel junction.

The acrylic block construction was done by using a brass mold with an internal stone mold of 24,20,16 mm dimensions. A depression (half ball) was made at the base of the one stone mold of 6mm diameter &2mm depth**.**

The separating medium (separating fluid/Ivoclar) was varnished to the walls of the mold then the sample was placed at this depression with the buccal surface to be treated faced downward & wax was used to fill the remnant space, the self cure acrylic was loaded into the mold& after completion of the polymerization, the two parts of mold were separated &excess acrylic was removed. 2mm from the buccal surface of the blocked teeth were grounded to obtain flat superficial dentin surface with 600-grit silicon carbide paper fixed on a roater (rotoflix) machine under a stream of running water. Then the samples were stored in (DDW) at 37°C for one hour. [16]**Group 1- :** The dentin surface will be etched with **phosphoric acid 37%,** received combination of **Excite** single component adhesive system &corresponding composite.

Group 2: -

Subgroup 2a:- Etched with **phosphoric acid 37%** , then the surface will be treated with **NaOCl** then the **Excite** will be applied on **wet surface**, then composite will be applied.

Subgroup 2b:-Etched with **phosphoric acid 37%** , then the surface will be treated with **NaOCl** then the **Excite** will be applied on **dry surface**, then composite will be applied.

The adhesive application was done according to the manufacturer.The exposed surface of each tooth was washed with (DDW) spray for 15 seconds then dried with an air syringe for 5 seconds). A circular hole, 4mm in diameter, was punched by a specially designed paper puncture in adhesive tape, which was positioned on the ground dentin surface to demarcate the bonding region [17]. The adhesive application was done according to the manufacturer instructions.

Regarding Excite adhesive system, the exposed surface was acid etched with a 37 % phosphoric acid gel for 15 seconds, then rinsed for 15 seconds, gently dried for 2 seconds at a distance of approximately 1cm (in a manner mimicking application performed by a dental unit) from the preparation surface in order to remove excess water, (The dentin surface should be slightly moist)

(18, 19).

A generous amount of Excite was applied on dentin surface using a Vivadent Applicator for 10 seconds, then evaporating any remaining solvent with a gentle air stream for 3 seconds at a distance of approximately 5mm from the preparation surface, then cured with light cure for 20 seconds at a distance of approximately 1mm from the preparation surface.The dentin surface should have a visible glossy appearance. **Regarding NaOCl solution** ,in order to remove the collagen network &interfere with the formation of conventional (hybrid layer), the teeth were treated with 10 % NaOCl solution for 60 seconds using Vivadent Applicator & rinsed for 60second

[4, 15] leaving **1-** a **moist** dentin surface (**subgroup2a)** by drying the surface with gentle air stream for 1 second at a distance approximately 1cm from the preparation surface or **2- dry** dentin surface (**subgroub 2b)** by drying the surface with gentle air stream for 10 second, at a distance approximately 1cm from the preparation surface [20]. Then the adhesive systems was applied according to the manufacturer instructions.

Regarding the composite restoration, application of the composite was done for all the samples following manufacturer instructions and using a device specially designed for the research for the standardization of the composite application (Al-Shawee device). The composite application standardization device is composed of a metal board with a fixed handle to hold the light cure gun in a way that the tip comes in a 90° angle to the sample. Also there is a circular movable base on which the sample was placed, under it there are six direction which are indicators for the curing directions. With the presence of a line drawn on the movable base, it is easy to ensure a 6 directions curing.

Near the base there is a holder supporting a 400-g weight on to which a 3.9mm in diameter, metal covered with plastic, condenser attached to it. There is a screw indicator in the upper end of the condenser which has a diameter wider than the glass tube (4 mm) present under it which prevents the condensers from going deeper than required, leaving a 2 mm space below the lower end of the condenser in which the composite was placed .By this device we have a standardized distance and curing in six directions, the time for each curing in each direction is 40 seconds, the total curing time is 240 sec, the composite was applied according to manufacturer's instruction.

Aging & Thermocycling

The specimens of all groups were stored in deionised distilled water at 37C° for 24 hours.

Thermal cycling allows bonded specimens to extreme temperatures which simulate intraoral condition. Therefore, all specimens were subjected to 250 thermal cycles using two water baths set at 5C° & at 55C° & with dwell time of 30 seconds in each bath with a transfer time of 5 second, using a thermocycling machine, that was manufactured by (Al-Khafaji & Al-Qayssi, 1993).

The specimens were again stored in deionised distilled water at 37C° until testing [21].

Testing Procedure (Shear Test). Shear bond strengths were tested with Instron testing machine using a stainless steel chisel shaped rod, which was used to deliver the shearing force with a cross head speed of 0.5mm per minute (ISO TR 11405), the load cell was set at 100 Kg. The tested specimens were placed in a specially designed acrylic block holder which was placed on the testing machine. The sample with its surrounding acrylic block was held in a vertical position in such a way that the long axis of the chisel-shaped rod was parallel to the flat prepared bonding site and perpendicular to the long axis of composite cylinder. The chisel end of the rod was positioned at the interface between the sample surface and composite cylinder. The specimens were loaded until they fractured. The forces were recorded in Newton which has been divided by the surface area 12.56 mm^2 to obtain shear bond strength calculated in MPa.

Mode of failure (debonding)

The fractured surface of specimens were examined using a stereomicroscope by two examiners to classify the type of fracture that occurred during the debonding procedure. They could be:

Adhesive (cohesive in adhesive interface failure).

Cohesivein dentin (dental substrate failure).

Cohesivein resin-based composite (resin-based composite failure).

Mixed (cohesive & adhesive failure) [22].

Statistical Analysis

Statistical analyses were done using SPSS version 7.5 computer software (Statistical Package for Social Sciences).

Frequency distributions for selected variables were done first. The data of each experiment will be presented as range, mean, SD and SE.

The statistical significance of difference in mean of a continuous response variable which is known or assumed to be normally distributed (like power needed to break the composite) between 2 groups was assessed by independent samples t-test. the resulting P value was multiplied by the number of comparison needed for each group (since each group will have to be compared to only 2 other group.

P value less than the 0.05 level of significance is considered statistically significant.

3. RESULTS

Regarding the Excite adhesive system, the use of wet NaOCl technique is associated with a statistical significant increase in mean power needed to break the composite of (6.4 MPa) over the conventional technique.

Table 1: The difference between Conventional and Wet NaOCl procedure in power need to break the composite in Excite products.

Power needed to break the composite	Control (Conventional)	Wet NaOCl procedure	difference between control and wet	95% confidence interval	P (ttest)	Adjusted P value
Excite						
Minimum	8.7	15.9				
Maximum	20.0	27.6				
Median	15.8	20.5				
Mean	14.7	21.1	6.4	(2.9-9.8)	0.001	0.003

SD	3.6	3.7				
SE	1.14	1.19	1.64			

Regarding the Excite adhesive system, the use of dry NaOCl technique is associated with a statistical significant increase in mean power needed to break the composite of (6.6MPa) over the conventional technique.

Table 2: The difference between Conventional and Dry NaOCl procedure in power needed to break the composite in Excite products .

Power needed to break the composite	**Control (Conventional)**	**Dry NaOCl procedure**	**difference between control and dry**	**95% confidence interval**	**P (t-test)**	**Adjusted P value**
Excite						
Minimum	8.7	13.6				
Maximum	20.0	30.1				
Median	15.8	22.2				
Mean	14.7	21.3	6.6	(2.5-10.6)	0.004	0.012
SD	3.6	5				
SE	1.14	1.57	1.94			

Regarding the Excite adhesive system shows no statistically significant differences between dry NaOCl procedure & wet NaOCl procedure.

Table 3: The difference between Dry and Wet NaOCl procedure in power needed to break the composite in Excite products.

Power needed to break the composite	**Wet NaOCl procedure**	**Dry NaOCl procedure**	**difference between wet and dry**	**95% confidence interval**	**P (t-test)**	**Adjusted P value**
Excite						
Minimum	15.9	13.6				
Maximum	27.6	30.1				
Median	20.5	22.2				
Mean	21.1	21.3	0.2	(-3.9 to 4.3)	0.93*[NS]*	1*[NS]*
SD	3.7	5				
SE	1.19	1.57	1.96			

4. DISCUSSION

The results of the shear bond strength study indicate that one-minute application of NaOCl to the conditioned dentin using wet or dry condition significantly increased the shear bond strength (table 1, 2).

Collagen depletion with NaOCl results in a more permeable substrate [23, 24] by removing the organic components of the dentin &enlarging the tubules near the outer dentin surface that enhances the spreading & diffusion of adhesive monomers through dentin, in addition the collagen depletion with NaOCl improve the surface energy of dentin, because the hydroxyapatite is a high surface energy substrate while collagen has a low–energy surface

(25).

The results also agree with Prati et al (1999)[26] who showed that high bond strength can be achieved after NaOCl treatment via reverse hybrid layer formation which proposed new mechanism of micro-mechanical resin retention & improved mechanical properties of the substrate.

There is no significant difference between acid etched deproteinized dry dentin and acid-etched deproteinized wet dentin when using Excite (ethanol based adhesive) (table 3).This may be explained by Tay etal [27] who considered that acetone & alcohol effectively displace water therefore better facilitators of resin primer infiltration as compared to water based adhesive systems.

Mode of Failure

The failure modes after shear bond strength are mainly adhesive, and only 2 specimens show cohesive failure in composite, so this means that the results are preferable as Hara et al (2001)[22] who stated that the shear bond strength test is preferable when the result in more adhesive failure occur at the adhesive resin /dentin interface.

Conclusions

1-There is a significant increase in shear bond strength after treatment of acid etched dentin with 10%NaOCL for 1 min .

2-There is no significant difference in the shear bond strength between dry and wet acid etched deproteinized dentin groups of the Excite adhesive system.

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