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# EVALUATION OF TENSILE BOND STRENGTH AND HYBRID LAYER OF NEWER GENERATION ADHESIVE WITH SINGLE AND MULTIPLE CONSECUTIVE APPLICATIONS- AN IN-VITRO STUDY

Dental Science


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## ABSTRACT

The objective of the study is to determine the effect of single and multiple consecutive coatings of all-in-one adhesive on tensile bond strength and hybrid layer formation. Forty two extracted human mandibular molars were selected. Specimens were divided into three groups based on the number of applications of Tetric N-bond all-in-one adhesive. Group 1: Single layer of adhesive; Group 2: Two layers of adhesive; Group 3: Four consecutive layers of adhesive were applied. Intermediate curing was done between each layer. Resin composite build-ups were made. 30 teeth were evaluated for tensile bond strength (TBS) using a universal testing machine, 12 teeth were used for hybrid layer evaluation using scanning electron microscope. TBS with two consecutive applications of all in one adhesive was significantly higher than with a single application, but application of further coatings caused a decrease in bond strength. There is no significant relationship between increase in number of adhesive layers and thickness of hybrid layer.



## KEYWORDS

Hybrid layer, Tensile bond strength, Self etch adhesive


## INTRODUCTION

The goal in adhesive dentistry is to achieve an adequately strong bonding of the restorative resin to the tooth structure so that there is optimum retention, minimal microleakage and, hence, better color stability and clinical longevity of the restoration. [1]

Bonding to enamel is considered as durable while bonding to dentin has been inconsistent because of its structure and chemistry. [2]

Self-etching system follow a trend towards simplification. They promote interdiffusion of the adhesive through the smear layer, providing procedures that are more reliable and less sensitive than the conventional adhesive techniques.[3]

Self-etch adhesive systems did not improve bonding effectiveness to dentin inspite of their purported reduction in technique sensitivity.[4]

To offset the limitations of self-etching adhesives, altered bonding protocols that increase resin-dentin bond quality were suggested;[5,6] those are the multiple applications of adhesive[5] or increased substrate contact time of the acidic primers.[6]

The purpose of this study is to determine the effect of single and multiple consecutive coatings of all-in-one adhesive on tensile bond strength (TBS) and hybrid layer formation.

## MATERIALS AND METHODS

### Sample preparation for tensile bond strength

Thirty freshly extracted caries free, unrestored human mandibular molars were selected and stored in distilled water. The occlusal surface was ground using a water cooled diamond disc (Buehler, Illinois, EUA, US) mounted on a slow speed micromotor handpiece until all occlusal enamel was removed. This resulted in exposure of flat dentin surface, with enamel at periphery. Horizontal indentations were placed with fissure bur on the radicular portion of the specimen to attach the specimen to the resin block. The teeth were embedded into self-curing resin leaving the dentin surface exposed. The specimens were randomly divided into three groups of 10 teeth each based on number of adhesive layers applied.

### Group 1 (single layer of adhesive)

Single layer of Tetric N-bond self-etch adhesive (Ivoclar Vivadent, Schaan, Liechtenstein) was applied to the exposed dentin surface using a fully saturated applicator tip of adhesive for 30 s and gently air-dried for 3 s then light-cured for 10 s using Blue Phase C8 LED unit (Ivoclar Vivadent, Schaan, Liechtenstein) at a light intensity of 800 mW/cm$^2$.

### Group 2 (Two layers of adhesive)

Two layers of adhesive were applied in the same manner as described in Group 1. Light-curing was done after the application of the each layer of adhesive.

### Group 3 (Four layers of adhesive)

In this group, four layers of adhesive were applied. Light-curing was done after the application of each layer.

A hollow polyvinyl cylinder with the inner diameter of 6 mm and height of 4 mm was placed on the treated dentin surface almost at the center of the specimen and Tetric N Ceram composite resin (Ivoclar Vivadent, Schaan, Liechtenstein) was condensed to 2 mm thickness and light-cured for 20 s. Another 2 mm thickness of composite resin was placed over the first placed composite increment.

A 26-gauge ligature wire was twisted at one end and a loop formed at other end. Twisted end was placed inside the 2 mm of uncured composite resin. The composite resin was then light-cured. Following complete curing; polyvinyl cylinder moulds were cut and removed, leaving the 4 mm of resin with ligature wire bonded to dentin. All the specimens were immersed in water for 24 hours. The loop end is then engaged to the hook of universal testing machine (Shimadzu, Japan) and pulled for measurement of tensile bond strength (TBS) at a cross head speed of 1 mm/min.

**Sample preparation for SEM analysis**

Twelve freshly extracted human mandibular molars stored in distilled water were used. These teeth were mounted in blocks of acrylic resin and the occlusal third of crown was removed using slow speed micromotor handpiece equipped with a diamond impregnated disc under water coolant. Subsequently remaining surface was polished with 180 grit, 240 grit and 600 grit silicon carbide sand paper until no enamel remained. Teeth were then cross-sectioned to obtain 1 mm thick dentin disks. Outer surface of each disk was polished with wet 600 grit silicon carbide sand paper to create a uniform smear layer. The samples were randomly divided in to 3 groups of 4 each. Specimens were treated with adhesive as mentioned above in three groups. After the completion of bonding procedure, 2mm of composite was applied over these dentin disks and light cured for 20 seconds , then samples were stored in distilled water at 37ºC for 24 hours. These disks were fractured in the center with a chisel for cross sectional viewing. All specimens were then immersed in 5% hydrochloric acid for 30 seconds and then washed for 30 seconds under running water. Sections were then transferred to 70% ethanol and dehydrated in increasing concentration of ethanol for 10 seconds each. Each specimen was then mounted on a aluminium stub with double sided carbon conductivity tape, and a thin layer of gold coat over the samples were done by using an automated sputter coater and examined under Scanning Electron Microscope (SEM) (Model: JEOL-JSM 5600) at a magnification of 1000X.

The mean values were subjected to statistical analysis (ANOVA with post hoc Tukey honestly significant difference test). P value set for the significance level of 0.001.

**RESULTS**

The mean tensile bond strength values were reported in Table 1. Hybrid layer thickness in Table 2 and Figs.1, 2, 3

**Tensile bond strength**

There was a significant difference in the mean tensile strength among the study groups. Mean values of group 2 were higher than mean values of group 3 and group 1.

There was an increase in bond strength as the number of coatings increase from one to two, followed by a decrease in bond strength with successive coatings.

**SEM analysis**

Under SEM, very thin hybrid layer (less than 0.5µm thick) was observed. It was irregular and non uniform in thickness with some areas being extremely thin while others were relatively thick in all three groups. The increase in number of adhesive layers did not appear to affect the thickness of the hybrid layer.

**Table 1: Comparison of mean TBS values among three groups**

| Group | n | Mean | SD | P | Post hoc test |
|---|---|---|---|---|---|
| 1 | 10 | 6.94 | 0.71 | < 0.001 | 2>1,3 |
| 2 | 10 | 11.39 | 0.71 | | |
| 3 | 10 | 7.20 | 0.52 | | |

SD = standard deviation TBS= Tensile bond strength

**Table 2: Width of Hybrid layer in µm**

| Group | Minimum | Maximum |
|---|---|---|
| 1 | 0.44 | 0.49 |
| 2 | 0.39 | 0.45 |
| 3 | 0.42 | 0.5 |

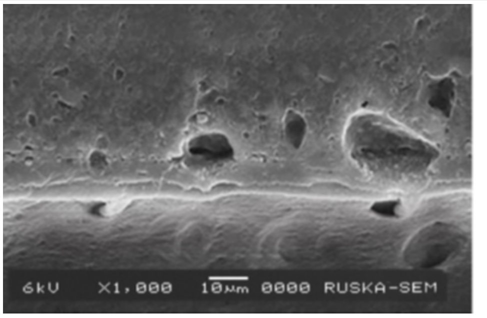


**Fig1. SEM image of Group 1**

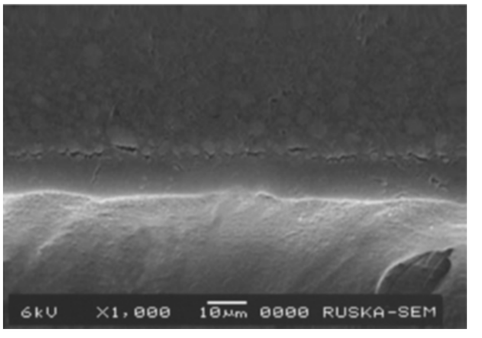


**Fig2. SEM image of Group 2**

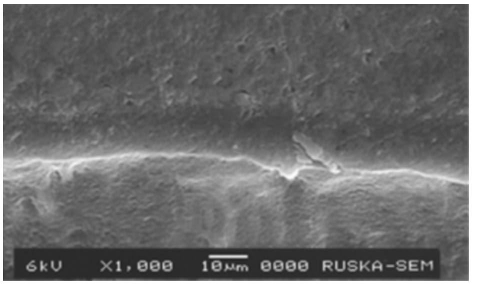


**Fig3. SEM image of Group 3**

**DISCUSSION**

Bonding to dentin represents a challenge to clinical scientists, as the substrate is an intrinsically wet organic tissue, penetrated by tubular structures that communicate with the pulp. Most of the modern adhesives are often regarded as technique-sensitive with the smallest error in the clinical application procedure being penalized either by rapid debonding or early marginal degradation.[6] As a consequence, the demand for simpler, more user-friendly and less technique-sensitive adhesives have led to the introduction of self-etch adhesives. The present study was conducted to evaluate the effect of multiple consecutive coatings of all-in-one self-etch adhesive on dentin bond strength.

Molars were preferred as flat dentin surface could be produced which would give a wider area of dentin to be treated and bonded to resin substrate.

The rationale behind selection of Tetric N-Bond Self-etch adhesive is that it contains hydrolytically stable methacrylamide monomers instead of the common reactive dilutent 2-hydroxyethyl methacrylate (HEMA) which is particularly instable in aqueous acid due to formation of hydrolysis-prone associates.[7] Salz and Bock [7]compared the adhesive properties and storage ability of methacrylamide monomers to methacrylate-based adhesive formulations and reported that HEMA-free formulations performed more reliably, with the fully acrylamide-based adhesive consistently giving the highest dentin bond strength values.

In the present study, the exposed dentin surface in group 1 was treated with adhesive according to manufacturer's instructions. The manufacturer's instructions for Tetric N-bond self-etch are the application of a single layer for 30 s followed by thorough air drying and light-curing for 10 s. This may result in a layer that is too thin for successful photoploymerization. In group 2 and group 3, multiple consecutive coats were applied with light-curing after each coat.

The method of light-curing after application of each coat was selected, as for simplified adhesive systems, which possess solvents in their composition; the improved adhesive thickness makes it more difficult to volatilize the solvent before light-curing, and this result in lower bonding values.[8] Moreover, the effect of repeated light-curing to the first coat of bonding resin may be able to increase the conversion of the adhesive resin, enhancing bond strength.

Normally in *in vivo* conditions, dental adhesives used are more likely to be subjected to shear forces, but the ability of the adhesive resin to retain on the tooth surface ultimately depends upon the resistance it offers to tensile forces, so tensile bond strength was evaluated in this study.

For evaluation of “hybrid layer”, Scanning Electron Microscopic study was done. Due to short wavelength of electron beams, SEM offered sufficient resolution to identify the formation of few micrometer wide hybrid layers.[9]

In the current study, the highest mean TBS was obtained with two consecutive applications of adhesive. The probable reason for the lower TBS for group with single coat application is single coat results in a layer that is too thin for photopolymerization which is been inhibited by oxygen.[10] Pashley et al.[5] observed that an additional application of bonding agent could seal the nonpolymerized oxygen inhibition layer, thus enabling it to be adequately polymerized.

Furthermore, the HEMA-free self-etch adhesive system used in the present study contains MDP monomer which is speculated to have chemical interaction with hydroxyapatite crystals forming stable calcium-phosphate and calcium-carboxylate salts, respectively, along with only a limited surface-decalcification effect (“Adhesion-Decalcification concept”). This additional chemical interaction is also thought to particularly improve bond durability.[11]

In accordance with the present study, Mandava et al.[1] demonstrated increase in bond strength for two consecutive applications of adhesive followed by decrease in bond strength with successive coatings.

In the present study, there was decrease in bond strength when more than two multiple consecutive coatings were applied. This might be due to increase in the thickness of the adhesive layer, which acts as a weak interface and resulted in total cohesive failure.[12]

The thickness of the hybrid layer and its influence on bonding durability is still uncertain. Some authors have suggested that the dimensions of the hybrid layer may be taken as an indicator of the strain-absorbing capacity of the corresponding interface.[13] This elastic buffer could be of utmost importance for absorbing the stresses originated from composite resin polymerization shrinkage. Nevertheless, no correlation was established between thickness of the hybrid layer and bond strength in the present study. The thickness of the hybrid layer observed was similar for all the three groups of about 0.5μm and did not present any relation to the values of bond strength. This is in agreement with other studies.[14,15,16]

If too thick, an adhesive layer would negatively influence the mechanical physical properties of the restoration with a risk of total cohesive failure in adhesive thickness; therefore, it would be advisable not to apply an excessive number of adhesive coats.

D'Arcangelo et al[8] stated that the ideal adhesive thickness is certainly variable and depends on the adhesive system used. Clinicians should consider the intrinsic properties of each bonding system when using a multilayering technique.

## CONCLUSION

Within the limitations of this *in vitro* study, the highest values for bond strengths were achieved following two consecutive applications of all in one adhesive compared to single and four layer applications with no significant relationship between increase in number of adhesive layers and thickness of hybrid layer.

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