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# Influence of an Extra Bonding Layer on Immediate and Delayed Shear Bond Strength of Two Different Universal Adhesives to Dentin: An *In Vitro* Study

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## Abstract

**Aim:** To evaluate the effect of an extra bonding layer on immediate and delayed shear bond strength of two different universal adhesives to dentin.

**Materials and Methods:** Flat coronal dentin surfaces were prepared in 100 extracted human molars. Teeth were randomly divided into four experimental groups (n = 20) according to two different universal adhesives applied with or without an extra bonding layer (EBL) of Scotch Bond Multipurpose adhesive (SBM) and one control group: Group 1: Control (SBM etch and rinse adhesive); Group 2: Prime and bond universal (PBU); Group 3: PBU+ EBL; Group 4: Tetric N bond universal (TNBU); Group 5: TNBU + EBL. After composite restoration, samples were subjected to 24 hrs and 6 months shear bond strength evaluation. Statistical analysis was done using two-way ANOVA and post hoc Tukey's test ($p < 0.05$).

**Results:** PBU depicted comparable immediate bond strength to SBM. TNBU presented significantly lower bond strength than SBM. After 6 months, a significant fall in bond strength was observed for all adhesives without EBL. Mean difference between immediate and delayed bond strength was found to be significant for group 1 (0.007), group 2 ($p = 0.001$), group 4 (0.002), and group 5 ($p = 0.001$), while it was non-significant for group 3 ($p = 0.076$) with EBL.

**Conclusion:** Application of EBL improved the immediate bond strength as well as significantly preserved the bond strength of PBU over 6 months of storage. EBL application could not prevent the fall in bond strength for TNBU adhesive over 6 months.

**Clinical Relevance:** Application of EBL over universal adhesive may improve the durability of resin-dentin bond.

***Keywords:*** *Extra Bonding Layer; Prime and Bond Universal; Tetric N Bond Universal; Resin-Dentin Bond*

## Introduction

Although current dental adhesives effectively bond to dentin but they show poor long-term stability of the resin-dentin bond [1,2]. Greater reduction in bond strength has been observed for an adhesive that employs simplified application procedures with combined primer/adhesive resin and higher solvent content [3]. The universal adhesives simplified the concept of dental bonding in terms of less chair side time and versatile modes of application [4]. These universal adhesives can be applied either in etch and rinse or a self-etch application mode.

Similar to one-step self-etch adhesives, universal adhesives have water in their formulation to enable ionization of hydrophilic acidic monomers and their simultaneous permeation through the smear layer and underlying mineralized dentin [5]. Universal adhesive has increased concentration of solvents and hydrophilic monomer in the adhesive leading to a greater amount of residual solvent entrapped in the adhesive layer. Such residual solvent may decrease the polymerization and prevent the formation of a highly cross-linked polymer, leading to loss of adhesive-dentin bond strength over time [6,7]. Limited bond durability of universal adhesives may also be accounted to their thin film thickness, often below 15 μ*m* which amplifies the effect of oxygen inhibition. A thin adhesive layer fails to act as a good shock-absorber thereby reducing the adhesive layer's ability to absorb mechanical stresses [8,9]. All these factors account for accelerated bond degradation [5,6]. It has been previously reported that the long-term bonding efficacy of one-step self-etch adhesives can be improved by the placement of an extra bonding layer [5,10-12].

Similarly, such deficiencies of universal adhesives may be overcome by the application of an extra bonding layer (EBL) which increases the thickness of the adhesive layer and may help in reducing fluid flow across the adhesive interface [13,14]. Improved bond strength, reduced nanoleakage, and increased degree of conversion within adhesive layer, have been reported for universal adhesives applied with an extra bonding layer [14]. Therefore, it can be assumed that preservation of the long-term bond strength of universal adhesive may be achieved by the application of an extra bonding layer. However, few researchers have reported no significant effect of an EBL with universal adhesives on resin dentin bond durability or their clinical performance [15,16]. It is likely, that the effect of EBL on the stability of resin-dentin bond is not always predictable and may show different results due to the variations in the composition of different universal adhesives tested.

## Aim of the Study

The aim of the study was to evaluate the effect of an EBL on the immediate and delayed shear bond strength of two different universal adhesives to dentin. The null hypothesis tested was that EBL does not affect the bond durability of universal adhesives bonded to dentin in self-etch mode.

## Materials and Methods

One hundred freshly extracted molars were collected and examined under a stereomicroscope to exclude teeth with caries, cracks, and defects. Teeth were cleaned and stored in 0.1% thymol for no more than 3 months. Teeth were collected after obtaining the patient's consent under a protocol approved by the institutional ethics and review board under protocol number KDCRD/IERB/12/2020/17.

The crowns of all teeth were flattened occlusally using a low-speed diamond saw to expose superficial dentin. All teeth were embedded in an auto-polymerizing resin such that only the crown portion was visible with occlusal surface parallel to the acrylic resin surface.

Teeth were randomly assigned into one control group of etch and rinse adhesive and four experimental groups (n = 20) according to two different universal adhesive systems used with or without the application of an extra bonding layer. Each group was evaluated for shear bond strength at two different time periods: 24 hours (immediate) and 6 months (delayed) after storage in artificial saliva.

**Group 1: Scotch bond multi-purpose:** Dentin surface was acid-etched with 35% phosphoric acid for 15 sec, rinsed with distilled water for 20 sec and gently blot dried followed by application of primer and adhesive of Scotch bond multi-purpose according to manufacturer's instructions.

**Group 2: Prime and bond universal**: Application of prime and bond universal adhesivein self-etch mode according to manufacturer's instructions.

**Group 3: Prime and bond universal+ EBL:** Application of prime and bond universal adhesive in the self-etch mode followed by application of a thin layer of extra bonding layer from Scotch bond multi-purpose adhesive.

**Group 4: Tetric N bond universal:** Application of tetric N bond universal adhesive in self-etch mode according to manufacturer's instructions.

**Group 5: Tetric N bond universal + EBL:** Application of tetric N bond universal adhesive in the self-etch mode followed by application of a thin layer of extra bonding layer from Scotch bond multi-purpose adhesive.

Transparent plastic tubes (TYGON laboratory tubing, Saint Gobain, Akron, OH, USA) with 3 mm in diameter and 2 mm in height were precut and placed perpendicular to the previously etched, pre-treated, and bonded dentin surface. A nanohybrid resin composite (Spectrum® syringe, $A_2$ Dentsply Dental Products) was filled into the precut tubes. Each bonded specimen was light-cured for 20 seconds (Blue phase light curing unit; Ivoclar) at a light intensity of 600 mW/cm$^2$. The plastic tubes were gently cut and removed after light polymerization.

Ten samples from each group were stored in distilled water at 37°C for 24 hours before immediate shear bond strength testing. The other ten samples from each group of the delayed testing group were stored in artificial saliva (Wet Mouth, ICPA Heath Products Ltd.) at 37°C in an incubator for 6 months before the shear bond strength test.

### Shear bond strength testing

The specimens were placed in a universal testing machine (Instron, ADMET, Enkay Enterprises, New Delhi) stabilized by the jig, while a straight knife-edge rod (2.0 mm) was applied at the tooth restoration interface at a cross-head speed of 0.5 mm/min. The load was applied until restoration failure. All debonded samples were analyzed under a stereomicroscope at 10 X magnification to define the location of the bond failure, categorized as Adhesive (a), Cohesive (c) and Mixed (m).

### Statistical analysis

Data were normally distributed as tested using the Shapiro-Wilk W test (p-value was more than 0.05) Paired t-test (two dependent groups) was used for immediate and delayed bond strength. One-way ANOVA (two or more independent groups) and post hoc analysis were done using Tukey's test. The level of statistically significant was set at a p-value less than 0.05.

## Results

In this study, no statistically, significant difference was observed in immediate dentin shear bond strength among etch and rinse adhesive and PBU used in self-etch mode. At 24hrs, PBU depicted shear bond strength values comparable to the SBM (etch and rinse adhesives). However, TNBU showed significantly lower bond strength than SBM at 24hrs and at 6 months. Although PBU depicted higher bond strength than TNBU when applied according to the manufacturer's instructions, the difference was not statistically significant. After 6 months of storage in artificial saliva, a significant fall in bond strength was observed for all adhesives. However, the application of EBL

preserved the resin dentin bond strength at 6 months for PBU. It was seen that the mean difference between immediate and delayed bond strength was found to be significant for control group 1 of 3-step etch and rinse adhesive (p = 0.007), group 2 (p = 0.001) group 4 (0.002), and group 5 (p = 0.389). However, there was no significant difference between immediate and delayed bond strength for group 3 (p = 0.076).

| | Mean (MPa) Immediate | Delayed | p-value |
|---|---|---|---|
| Group SBM | $44.46^{a} \pm 3.14$ | $33.24^{a} \pm 5.45$ | 0.007*, sig |
| Group PBU | $40.36^{ac} \pm 4.00$ | $31.19^{ac} \pm 5.62$ | 0.001*, sig |
| Group PBU+EBL | $41.04^{ac} \pm 4.42$ | $38.24^{a} \pm 5.71$ | 0.076, ns |
| Group TNBU | $35.6^{bc} \pm 7.24$ | $25.43^{bcd} \pm 7.87$ | 0.002*, sig |
| Group TNBU+EBL | $34.76^{bc} \pm 8.45$ | $28.58^{ad} \pm 4.53$ | 0.001*, sig |

***Table 1:*** *Intergroup comparison of immediate and delayed shear bond strength of different groups.*

*Paired t-test, the level of significance set at p < 0.05.*

*Ns: Non-Significant, Sig: Significant.*

*Same superscript letter indicates no statistically significant difference within a column.*

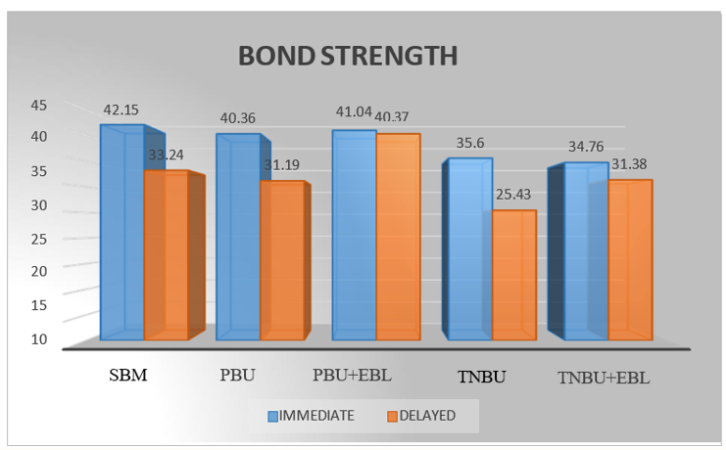


***Figure 1:*** *Comparison of immediate and delayed bond strength among various group.*

## Fracture mode analysis

After immediate shear bond strength testing, all the groups depicted maximum number of mixed failures. After 6 months, number of adhesives failures increased in all the groups regardless of EBL application.

| | | Adhesive | Cohesive | Mixed | P-value |
|---|---|---|---|---|---|
| Group SBM | Immediate | 3 (30%) | 0 (0%) | 7 (70%) | 0.314, ns |
| | Delayed | 5 (50%) | 1 (10%) | 4 (40%) | |
| Group PBU | Immediate | 4 (40%) | 0 (0%) | 6 (60%) | 0.580, ns |
| | Delayed | 4 (40%) | 1 (10%) | 5 (50%) | |
| Group PBU+EBL | Immediate | 3 (30%) | 1 (10%) | 7 (70%) | 0.091, ns |
| | Delayed | 4 (40%) | 0 (0%) | 6 (60%) | |
| Group TNBU | Immediate | 4 (40%) | 1 (10%) | 5 (50%) | 0.890, ns |
| | Delayed | 5 (50%) | 1 (10%) | 4 (40%) | |
| Group TNBU +EBL | Immediate | 5 (50%) | 0 (0%) | 5 (50%) | 0.590, ns |
| | Delayed | 6 (60%) | 0 (0%) | 4 (40%) | |

***Table 2:*** *Fracture analysis of the tested groups.*

*Chi-square test, Ns: Non-significant.*

*There was no significant difference in the modes of fracture between the groups.*

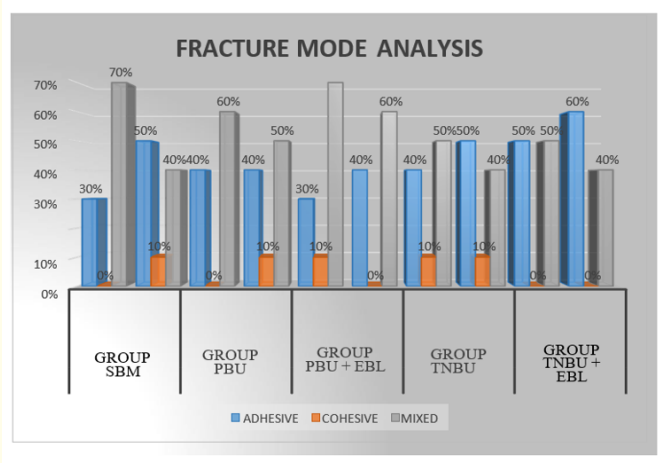


***Figure 2:*** *Comparison of fracture mode analysis among various group.*

## Discussion

Bond strength of universal adhesives decreases over time [1,7,15]. Adhesive interfaces formed by these simplified adhesives act as semi-permeable membranes, leading to hydrolytic degradation of demineralized collagen fibrils [17]. Application of a hydrophobic, solvent-free extra bonding layer can reduce hydrolytic degradation and increase the longevity of resin-dentin bond [11,13,18].

In our study, there was no statistically significant difference observed in immediate dentin shear bond strength among etch and rinse adhesive SBM and universal adhesive PBU used in self-etch mode. Similar to our study Pradeep., *et al*. also reported comparable shear bond strength of PBU and etch and rinse adhesive [19].

When used according to the manufacturer's instructions prime and bond universal depicted higher immediate bond strength than tetric N bond universal, although the difference was not statistically significant. Compositional differences between adhesives may play an important role in adhesive performance. Both universal adhesives contain 10 MDP monomer capable of chemically interacting with the hydroxyapatite by the formation of a stable nano-layering at the adhesive interface [17]. PBU has phosphoric acid-modified acrylate resin PENTA and 10 MDP. Both monomers etch dentin releasing calcium ions which form irreversible calcium phosphate complexes in the hybrid layer. This may explain good performance of PBU [20].

In our study, TNBU showed significantly lower bond strength than SBM at 24 hrs ($p = 0.001$) and 6 months ($p = 0.049$). Munoz., *et al*. [13] also reported that this universal adhesive exhibited inferior microtensile bond strength values compared to control conventional adhesives.

Tetric N bond universal contain bisphenol A diglycidyl methacrylate which is a highly viscous monomer that results in poor penetration into demineralized dentin and HEMA present in TNBU can also lead to hydrolytic degradation, resulting in reduced bond strength values with TNBU adhesive [21].

In our study after 6 months of storage in artificial saliva, a significant fall in bond strength was observed for all adhesives used according to the manufacturer's instructions. SBM showed the reduction in SBS values after 6 months, despite being a 3-step adhesive. Among the tested adhesives, SBM contains more HEMA (30 - 40 wt%) than the other adhesives. Although hydrophilic HEMA enhances resin monomer penetration into demineralized dentin, it is thought to be susceptible to hydrolytic degradation over time [22,23]. Therefore, this 3-step etch &rinse adhesive also depicted a significant fall in bond strength over 6 months.

In this study, PBU with the application of an extra bonding layer showed higher bond strength at both 24hrs and 6 months of water storage. Ermis., *et al*. [24] also reported that the bond durability of universal adhesives, used in self-etch mode, improved when an extra hydrophobic adhesive layer was applied over the cured universal adhesive.

The application of an extra bonding layer improves the adhesion of universal adhesives to dentin by adding hydrophobic monomers to the adhesive interface, thereby, decreasing the relative concentration of residual solvents. This decreases the permeability of the adhesive layer and leads to improved monomer conversion which makes the hybrid layer more resistant to long term degradation [13-15,24].

However, TNBU with EBL group demonstrated low bond strength in 6 months (delayed) testing. TNBU contains (HEMA) which acts as a solvent improving the monomer solubility. However, HEMA retains water and interferes with the polymerization of adhesives. HEMA stimulates osmotically driven water movement from the underlying dentin, leading to water droplets on the adhesive surface which behaves as a semipermeable membrane [25]. This hydrophilicity weakens the mechanical strength of the polymerized adhesive and makes it more prone to degradation. Additionally, HEMA reduces the nano-layering of 10 MDP at the adhesive interface [26,27]. However PBU (HEMA Free) adhesive has been considered advantageous in removing those water-prone hydrophilic domains [20].

Vinagre., *et al*. [15] stated that the effect of an extra bonding layer on the immediate and aged dentin bond strength of the universal adhesives was dependent on the specific adhesive and its application mode.

## Conclusion

Within the limitations of this study, it can be concluded that tetric N bond universal adhesive presented significantly lower bond strength than etch and rinse SBM. The application of an extra bonding layer improved the immediate bond strength as well as significantly preserved the bond strength of prime and bond universal adhesive over 6 months of water storage. Extra bonding layer application could not prevent the fall in bond strength for TNBU adhesive over 6 months. Further clinical trials are required to evaluate the effect of extra bonding layer application on the bonding performance of universal adhesives.

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