

## Ultrastructural Comparison between Wet and Dry Bonding Techniques of Two Different Solvent-Based Adhesives

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### Abstract

The techniques of wet and dry bonding have been well accepted to facilitate clinicians under certain circumstances during bonding of composite restoration. Recent universal adhesive system with different solvent types have the ability to overcome excessive moist phenomena or dessication on the dentin surface and played an important role in bonding to dentin.

To investigate and compare the ultra-structure characteristics of resin-dentin interfaces among two (2) different solvent-based adhesives using wet and dry bonding technique.

Twenty (20) human molar teeth were cut to produce a flat dentinal surface then divided into two groups of 10 teeth each (G1: Tetric N Bond Universal, G2: Sprectrum Bond). Each group was subdivided into wet bonding (WD) and dry bonding, (DB). Composite (Diafil) buildups were incrementally applied with 4mm in height. All specimens were viewed under SEM at 2000x magnification. SEM pictomicrographs were taken from the interface to observe the bonding interface and analyzed by three calibrated examiners. Each micrograph was classified as follows 0=absence of resin tag, 1= ≤ 10 resin tag/field, 2= ≥ 10 tags/field and 3= 2+ abundant inter-tag complex.

There was a significance increase of resin tags morphology of the dentine/resin interface in G1 under wet bonding technique, revealing higher numbers of tag penetration as compared to G2 ( $P < 0.01$ ) (Kruskal-Wallis test). There was no statistically significant difference between wet and dry-bonding technique at the interface ( $p > 0.187$ ) (Mann-Whitney U-test,).

Acetone based adhesive produce a better water chasing ability as compared to ethanol-water based adhesive, thereby this factor had affected the quality pattern of resin tags. The results of this study suggested that morphology of resin tags depended on the types of solvent of the adhesive used.

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### Introduction

Over the years, a number of modifications have been made to dentin adhesives and the formulations were characterized as 'generations', mode of application steps and the

modification of smear layer.<sup>1-3</sup> A recent family of dental bonding agent, known as universal adhesive systems, has been introduced to ease the bonding procedure with lesser technique sensitivity<sup>4</sup>.

At present, there are two techniques which help to achieve adequate hybridization. The dry bonding<sup>5</sup> requires the substrate field is kept dry and adhesives with water-based primers are used to rehydrate the collagen network. These water-based primers have good penetration ability, but if present in excess, can inhibit polymerization.

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The other option is to keep the substrate surface moist and depend on the action of the water chasing capacity of solvent primers (acetone/ ethanol). The importance of leaving the water on the dentin surface is to maintain the exposed collagen web flexible and permeable prior to adhesive infiltration. In this way, the bond strength to dentin can be improved in the moist surface when compared to air dried surface as adhesive systems are blended together with their hydrophilic primers dissolved in acetone<sup>6,7</sup>. Air-drying of the etched dentin surface has been shown to led to shrinkage and collapse of the collagen network ,thereby preventing monomers of the primer and adhesive resin from wetting and infiltrating the conditioned surface<sup>8,9</sup>.

Acetone, with their high volatility, followed by other solvents such ethanol and water may displace surface moisture and facilitate the primer monomers into the microporosities of the exposed collagen network<sup>10,11</sup>. Hence there is need to study the effect of the solvents in the dentin bonding agents on the quality of the resin-dentin interface by comparative trials.

Thus, the purpose of this study is to investigate ultra-structure characteristics of resin-dentin interfaces among two different solvent-based adhesives on wet and dry bonding environment. The hypotheses tested were that the application of a different solvent-based adhesives would not influence morphology of the resin tags penetration at the interface.

### Materials and methods

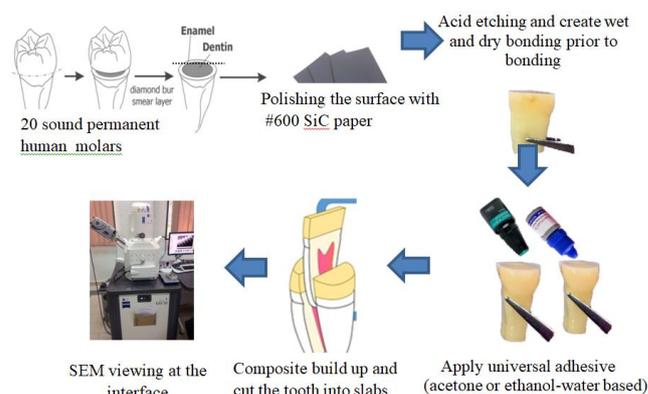
#### Tooth preparation

A summary of the procedure as previously is schematically illustrated in Fig. 1. Twenty (20) permanent and sound human third molars that were kept in 10% formalin at 4 °C were used in this study. The study was approved by the IIUM Research and Ethic Committee (ID Number:2019-060). The coronal part had been longitudinally at the cementum–enamel junction using a low-speed cutting machine under water spray (ISOMET ,Buehler ,IL) to produce a flat coronal dentin surface. The roots and the pulp tissue were then removed. A standardized smear layer was created by polishing the dentin surface with a 600-grit SiC .

#### Bonding procedure

Two universal single bottle adhesive systems with different solvents were used:

Spectrum Bond (Dentsply, Sirona) as acetone based and Tetric N Bond Universal (Ivoclar Vivadent),an ethanol-water based systems. The composition is listed in Table 1-2. Tooth specimens were randomly divided into two groups which were (Wet Bonding –WB) and dry bonding (DB).



**Figure 1.** Schematic illustration of the bonding workflow.

In WB ,the dentin surface was maintained moist after application of acid etching to mimic the wet bonding environment. While for dry bonding preparation,, the surface was blot-dried with absorbent paper after acid etching. Adhesive systems were then applied to the surface according to the manufacturer’s instruction. The prepared surface was left undisturbed for 20 seconds for solvent evaporation. Then, adhesive agent was applied light-cured for 10 seconds. A 4mm height of composite resin (Diafil, DiaDent) build up was applied to all samples after bonding procedure. Each 1mm increment was light-cured for 40 s with a light-curing unit (MiniLed, Satelec-Acteon) that delivered 600mW/cm<sup>2</sup>.

Material (Manufacturer)	Composition	Mode of application
Composite resin (Diadent)	UDMA, Bis-GMA, TEGDMA, Photo initiator, colorant ,filler	Light curing 20 seconds
Spectrum Bond (Dentsply)	UDMA, TEGDMA, Camphoroquinone, PENTA, BHT, Acetylamine, Acetone	Etch: 15 secs Rinse: 5 secs Apply adhesive 15 secs Mild air syringe: 5 secs light cure: 10 secs
Tetric N Bond Universal (Ivoclar)	Methacrylates, water, highly dispersed silicon dioxide, initiator and stabilizers ,Ethanol	Etch: 15 secs Rinse: 5 secs Apply adhesive 20 secs Gentle air syringe: 5 secs light cure: 10 secs

**Table 1.** Composition of materials used in this study.

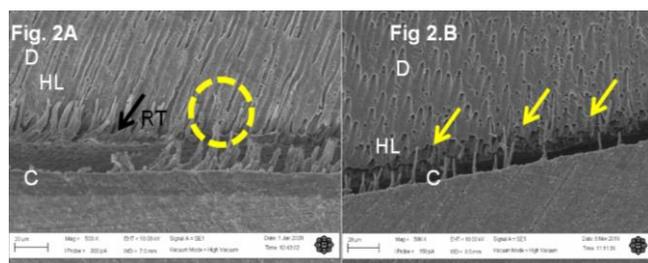
Score	Description
0	Absence of resin tag
1	< 10 tags per field
2	>10 tags per field
3	Abundant and continuous tags

**Table 2.** Score classification of resin tags.

*SEM observation of resin- dentine surfaces*

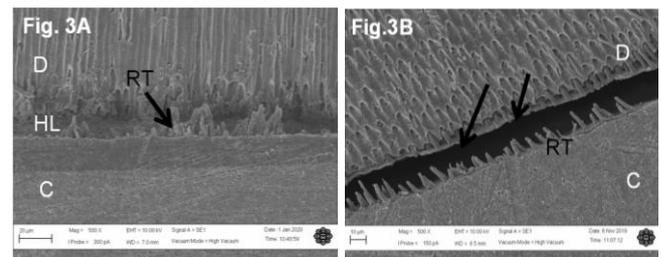
After the bonding procedure and completion of restoration, the teeth were cut into slab with approximately 3 mm in thickness by using low speed cutting machine. Central slab from each tooth were selected and stored in distilled water at room temperature for 24 hours. All the slabs were then dehydrated in ascending grades of ethanol (25% for 20 mins, 50% for 20 mins, 75% from 20 mins, 95% for 30 mins & 98.9% for 60 mins). In order to observe the resin tags penetration morphology, the slabs were immersed in 1M hydrochloric acid for 30 seconds and 5% sodium hypochlorite for 5 minutes, followed by rinsing with warm water. Then, the samples were fixed with hexamethyldisilazane (HMDS) for 20 minutes, placed on a filter paper inside a covered glass vial and air-dried at room temperature. Specimens were later mounted on aluminum stubs, sputter coated with gold-palladium (LEICA EM SCD005, Germany) and examined under scanning electron microscope (SEM- ZEISS Model SEM EVO50) at 2000x magnification. SEM pictomicrographs were taken at the interface up to five shots per sample to observe the interface and analyzed by three calibrated observers. Each pictomicrograph was evaluated according to the four categories as follows:

Differences in score values were analyzed using Kruskal-Wallis test, followed Mann-Whitney-U test for difference between wet and dry bonding. Statistical significance was set at  $p = 0.05$

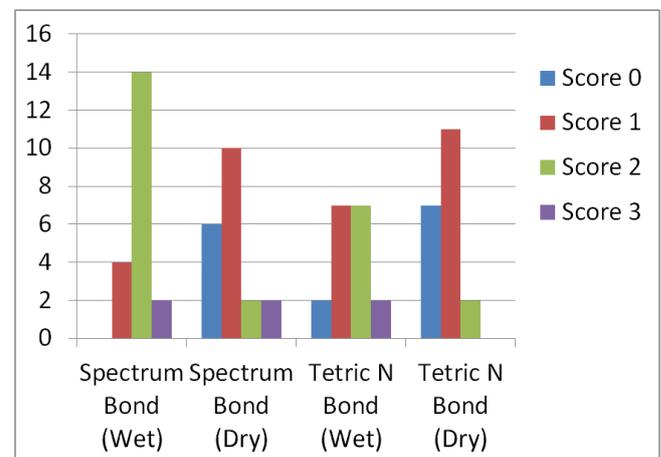


**Figure 2.** Representative SEM images (2000x) of resin dentin interface from acetone based

adhesive. Fig.2A showed abundant and continuous resin tags pattern in wet bonding technique (Tags appeared as long, conical shape—in circle). Fig. 2B showed much lesser tags penetration and presence of few voids (yellow arrow) present within the hybrid layer in dry bonding. (D: dentin, HL: Hybrid layer, C: Composite, RT: tags).



**Figure 3.** Representative SEM images (2000x) of resin dentin interface of ethanol-water based adhesive. Figure 3A revealed much more uniform hybrid layer and the tags were shorter in wet bonding. While in dry bonding (Fig.3B), voids, gaps were seen in hybrid layer (arrow) and the resin tags were observed as short and less penetrating. (D: dentin, HL: Hybrid layer, C: Composite, RT: tags)



**Figure 4.** Comparison of two groups (Tetric N-Bond and Spectrum Bond) with respect to their mean score value.

**Results**

All the tested adhesives gave variety of resin tags pattern under two different environments. Representative SEM photomicrographs in Fig.2 & 3 illustrated the morphology of resin-dentin interfaces produced by the two adhesive systems.

The mean score distributions are shown in Figure 4. Kruskal Wallis indicated that when Spectrum Bond prepared under wet bonding environment, there was a significant increase of Score 2 ( $p < 0.01$ ) when compare to Tetric N-Bond. On the other hand, there was no significant difference of resin tags pattern between wet and dry bonding technique at the interface among these two adhesives ( $p > 0.01$ ) (Mann-Whitney U).

## Discussion

The recent universal adhesive systems have been introduced to expand the opportunity for clinicians to decide which adhesive strategy to use of either etch-and-rinse or self-etch technique<sup>12-14</sup>. This versatile new adhesive system incorporated hydrophilic components that are diluted in organic solvents like acetone or ethanol in their composition. During bonding, a certain amount of moisture is needed to maintain the collagen network integrity in an expanded state thereby preserving the porosity necessary for interdentin resin penetration. The bifunctional molecules with one hydrophobic and one hydrophilic resin end are dragged to the bottom of the demineralized zone assisted by acetone, ethanol, both of which have affinity for water. Thus the organic solvents can chemically displace fluid from the moist collagen network and promote the resin monomers infiltration through the collagen web.

The findings of this study showed both resin tags as well as hybrid within the resin interface in the two experimental groups. In our study, Spectrum Bond (acetone-based dentin bonding system) produced a homogenous, continuous gap-free hybrid layer with fewer voids and conical resin tags with lateral branches compared to the other adhesive systems. This is consistent with Marco Ferrari *et al* in 1995 who investigated resin-dentin interdiffusion zone following the application of Prime & Bond NT (PBNT-acetone based adhesive) on both conditioned and unconditioned dental substrates. Their results revealed that the dentin samples treated with 36% phosphoric acid showed a characteristic hybrid layer, reverse cone shaped tags and adhesive lateral branches. On the other hand, M. Hashimoto *et al* in 2002, also observed the resin-dentin interface produced by two acetone-based adhesives (Prime & Bond NT & One-Step) under dry and wet bonding conditions

by laser Raman spectroscopic analysis. Their results suggested better good resin impregnation in specimens when the excess water was blotted from the dentin surface, leaving the surface visibly moist (wet-bonding) compared to when the specimens were completely dried with compressed air (dry bonding). Spectrum Bond is a nano-technology adhesive, contains a very low viscosity nanoscale filler (5-7nm), a high resin concentration and very small resin hydrophilic molecules (PENTA). This makes the bonding agent stronger against wet environment, create better penetration to dentin and improved marginal integrity. Water-chasing properties of this acetone-based adhesive may be responsible for the intimate adaptation of monomers to the etched peritubular and intertubular dentin<sup>17</sup>. We suggested that this is another factor Spectrum Bond showed a relatively good resin impregnation in specimens when surface was left moist.

The ethanol-water based dentin bonding system, Tetric N-Bond consistently produced a hybrid layer with a relatively non-uniform ultrastructure, and with lesser resin tags penetration. Void-like spaces, previously occupied by water along the hybrid layer, phase separation between hybrid layer and overlying composite and incomplete resin-infiltration were observed in some samples.

The findings is supported by other study<sup>18</sup> in which they revealed that when water is remained trapped inside the collagen network, the remaining water competes for space with the resin inside the demineralized dentin and causing incomplete polymerization of the adhesive within the hybrid layer.

The presence of voids in the hybrid layer may be due to blister-like areas and formation of resin globules caused by presence of trapped air bubbles which originated from the water and alcohol of bonding systems<sup>19</sup>.

It has been speculated that poor infiltration of the adhesive resin into the collagen-rich area of the demineralized dentin had led to formation of gaps in the hybrid layer that are vulnerable to degradation<sup>20</sup>. Water may well occupy these spaces if an incomplete penetration occurs, therefore producing hydrolysis of exposed collagen peptides. This exposed collagen is not only susceptible to degradation from water, but also from bacterial enzymes that cause collagen breakdown jeopardizing the

bonding integrity of one restoration.

The findings of this qualitative in-vitro study do not represent an exact hybridization standard for the adhesive systems used and may not be directly correlated to true clinical effectiveness, therefore, mechanical properties related studies and clinical trials are necessary to substantiate these results and establish the superiority of one over the other.

### Conclusions

Within the limitation of our study, we concluded that both acetone-based and ethanol based adhesive produced a relatively more uniform and better resin tags penetration in wet condition when compared to that in dry condition. Hence, further study is required to evaluate the relationship between tags penetration and mechanical property of the adhesive material.

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### Declaration of Interest

The authors report no conflict of interest pertaining to any of the products or companies discussed in this article.

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