ORIGINAL RESEARCH

Effect of different evaporation periods on microtensile bond strength of an acetone-based adhesive to dentin

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ABSTRACT

Objective: Solvent content of a contemporary dental adhesive affect the bonding process, especially in the case of acetone based adhesives. The aim of this study was to evaluate the effect of different air-drying periods on microtensile bond strength (MTBS) of a total-etch adhesive to dentin. **Materials and Methods:** Prime & Bond NT (Dentsply-USA) was used with different air-drying periods (0, 2, 5, 10, 30sec) for bonding a composite resin to prepared dentin. The specimens were then subjected to a tensile force until fracture and the MTBSs of the samples were recorded. Failure modes of the fractured samples were also determined using stereomicroscope and scanning electron microscopy. Data were analyzed using ANOVA and Bonferroni tests ($P = 0.05$). **Results:** With increasing the air-drying periods, the MTBSs were increased until the 5 second air-blowing; after that, with increasing the air-drying periods, the MTBSs decreased. Both, the most complicated failure and the strongest bond were seen in the 5 sec air-drying group. **Conclusion:** There is an optimum air-drying time for acetone based adhesives which results in the strongest bond to dentin.

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Contemporary total-etch adhesives are a combination of monomers and solvents, which are used as an adhesive for bonding the composite restoration to enamel and/or dentin. Solvent acts as a transport medium, carrying reactive agents from the storage container to the tooth,[1] lowers resin viscosity and allowing formation of resin impregnation phase in the bonding process.[2,3] During adhesive application, most solvents evaporate quickly, leaving behind a mixture of polymerizable monomers enveloping collagenous, demineralized dentin.[2,3]

Upon placement, the clinician is instructed to direct a steam of air over the film in order to evaporate remaining carrier solvent. The stated purpose in doing so is to minimize, if not totally eliminate, residual solvent because of its potential interference with polymerization of the adhesives. Because of clinical limitations and the inability to directly assess residual solvent content and the need to provide dental treatment in minimal time, clinicians often minimize time spent in air drying the adhesive, potentially leaving unknown amount of solvent in the polymerized layer.[4]

Bonding efficacy and marginal seal of contemporary total-etch adhesives may be affected by technique sensitivity of these adhesives.[5,6]

Incomplete evaporation of solvent results in dilution, poor polymerization or phase separation of the resin components.[7] after light curing of the adhesives, residual water or solvents may become pathways for water movements within the hybrid or resin layers, increasing the permeability of the resin dentin interfaces and their subsequent susceptibility to degradation via resin hydrolysis.[8]

Acetone, ethanol, and water are the most common solvents in adhesives. Adhesives with acetone solvents can volatilize too quickly after being dispensed, leaving a very viscose fluid that dose not penetrate exposed collagen well.[9] This solvents can also evaporate easily from its container if left open too long and multiple application coats may be required, 4 therefore, solvent content in acetone-based adhesives is more critical.

Recent studies have shown that bond strength and other mechanical properties of the adhesives may be affected by different solvent evaporation periods.[8,10,11] Other

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investigators have emphasized on the importance of solvent content and air-drying in bonding process.[12-16]

The purpose of this study was to evaluate the effect of different evaporation periods on microtensile bond strength of an acetone based total-etch adhesive to dentin.

MATERIALS AND METHODS

Twenty seven caries-free human molars were used in this study. After extraction, the teeth were washed and stored in 0.2% thymol solution for two months prior to the study. Following the cleaning of the teeth, the enamel caps of the teeth were removed using high speed instrument and diamond bur with water spray coolant until the superficial dentin was exposed. The superficial dentin then were conditioned using 37% phosphoric acid (Etchant liquid, Alpha-Dent, USA) for 15 seconds and were washed for 20 seconds and air dried for 2 seconds until the pooled water were displaced.

Prime and Bond NT (Dentsply, USA) was used as adhesive for restoring the teeth [Table 1]. The adhesive was actively rubbed on the conditioned area using microbrush for 20 seconds. The primed dentin surface was then air-dried with oil-free compressed air with an air pressure of 4 kgf/cm^2 from 5 cm above the dentin surface for: 0 (no evaporation), 2, 5, and 10 and 30 seconds. The adhesive layer was then irradiated for 20 seconds with a light-curing unit (Arialux, Apadana Tak, Iran) with a light output about 400 mW/cm^2. Composite build-ups were performed using three 2 mm increments of resin composite (Synergy, Coltene, Switzerland) which were individually light cured for 40.

The specimens were then stored in distilled water at room temperature for 24 h, they were sectioned perpendicular to the adhesive interfaces with a diamond saw (Vafayi, Iran) to produce resin-dentin slices, each with 1 mm diameter. The slices were then prepared using high speed diamond bur with water spray as a coolant to produce hourglass slices with an adhesive area of 1 mm^2.

Assessment of shear bond strength

The specimens were then attached with syanoacrilate adhesive to a testing jig, and a tensile load was applied with a universal testing machine (DARTEC Universal testing machine/HC10, Instron-UK) at a crosshead speed of 1 mm/min until fracture occurred.

Eighteen samples were examined in each group

The MTBSs were expressed in MPa, dividing the apply force (N) at the time of fracture by the bonded area (mm^2).

Statistical analysis

Data were analyzed using One-Way ANOVA at a significant level of $P = 0.05$. For Bonferroni tests P values were adjusted for ten segments of multiple comparisons using an overall experiment wise error rate of $\alpha = 0.05$. The adjustment was 0.05/10 = 0.005.

Failure modes of the fractured specimens were observed using a stereomicroscope (ZTX-3E, Zhejiang, China) at ×20 magnifications. Three samples of each group were sputter coated with gold (SCD125, Germany) and the resin-dentin interfaces were analyzed in a Scanning Electron Microscope operated in the secondary electron mode (Phillips XL20, Netherland). All observations were conducted by one person.

RESULTS

Microtensile bond strength analysis

A total of 90 specimens from 27 teeth were available for microtensile test (Twenty one samples were debonded before the test.) The mean MTBSs for the data in MPa are presented in Table 2. ANOVA test showed a significant difference ($P < 0.05$) between the groups. Bonferroni tests showed that all groups have significant statistical differences with each other ($P < 0.005$) except for the group I and V. The highest MTBS (27.9 ± 4.3) was observed in 5 second air-drying group. Other evaporation periods showed significant lower MTBS than 5 sec air-drying group.

Failure mode analysis

- The majority of the samples in group 1 were fractured from the interface; also cohesive failure in the adhesive layer was seen [Figure 1].
- In group 3 mixed failure (in adhesive, interfacial and in dentin) was the predominant failure [Figures 2 and 3].
- Most of the samples in group 2 and group 4 showed interfacial failure and cohesive failure in dentin [Figure 4].
- Pure interfacial failure was seen in the majority of the samples of group 5 [Figure 5].

DISCUSSION

Contemporary total-etch adhesives are routinely used in general dentistry for bonding the restorative materials

Table 1: Prime and bond NT and its composition

Material	Manufacturer	Composition
Prime and bond NT	Caulk/dentsply Int., Ink., milford DE, USA	PENTA, UDMA, resin-T, resin-D, bisphenol a dimethacrylate, acetone, nanoscale filler cetylamine hydroflouride

Table 2: Results of microtensile bond strength tests

Group	Time (sec)	Numbers in groups	Mean bond strength (MPa)	Std. deviation	Debonded samples
I	0	18	8.2 16.7 27.9	2.6	7
II	2	18	20.9	1.8	4
III	5	18	11.4	4.3	3
IV	10	18	-	2.4	2
V	30	18	-	2.4	5

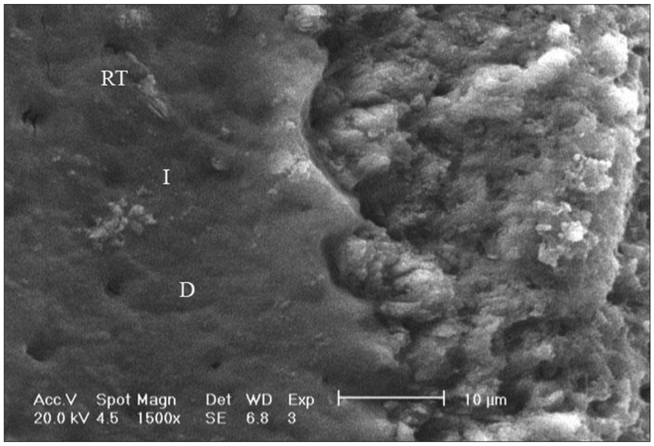

Figure 1: SEM photomicrograph of fractured surface of a sample from group 1, showing the dentin (D), interfacial failure (I) and remnants of resin tags, categorized as interfacial failure and cohesive failure in adhesive

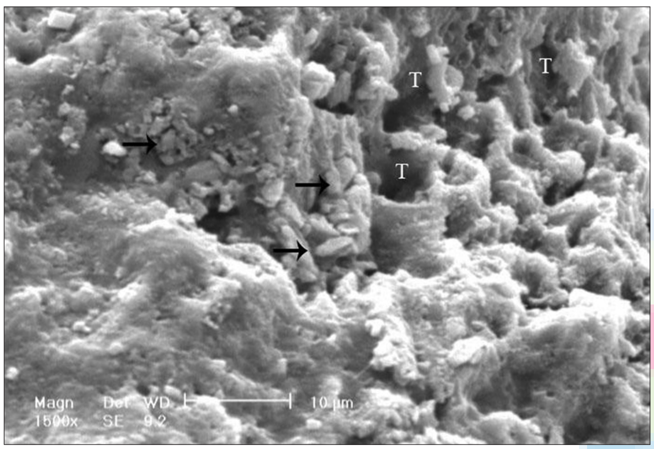

Figure 3: The circle in Figure 2 with higher magnification, showing the fractured dentin (D) and dentin tubules (T) with the remnants of the fractured dentin on it (arrows)

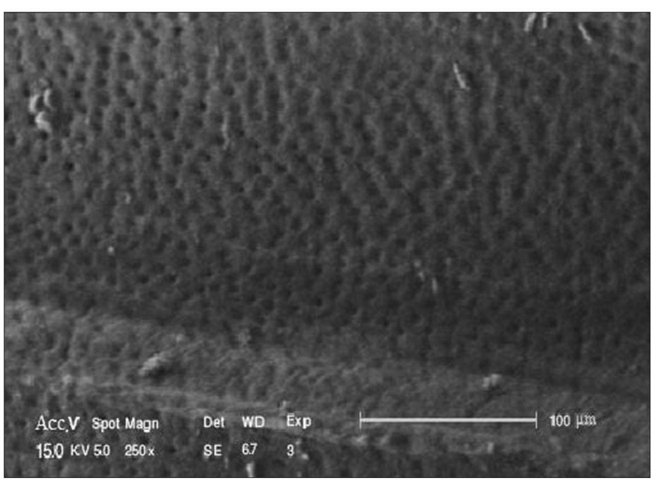

Figure 5: SEM photomicrograph of a sample from group 5 showing the interfacial failure. The adhesive layer was completely removed from the dentin side

to enamel and dentin. These adhesives mainly contain monomers in a solvent such as water, alcohol or acetone.

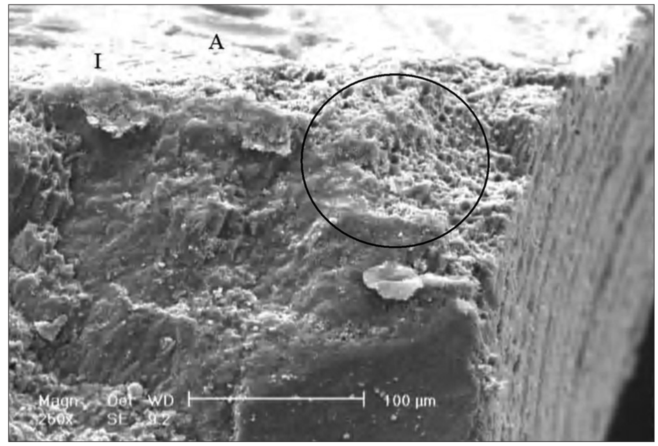

Figure 2: SEM photomicrograph of the fractured surface on the dentin side of a sample of group 3. The fracture occurred within the adhesive layer, interface and in dentin. The failure categorized as mixed failure. Dentin (D), adhesive layer (A) and interface (I)

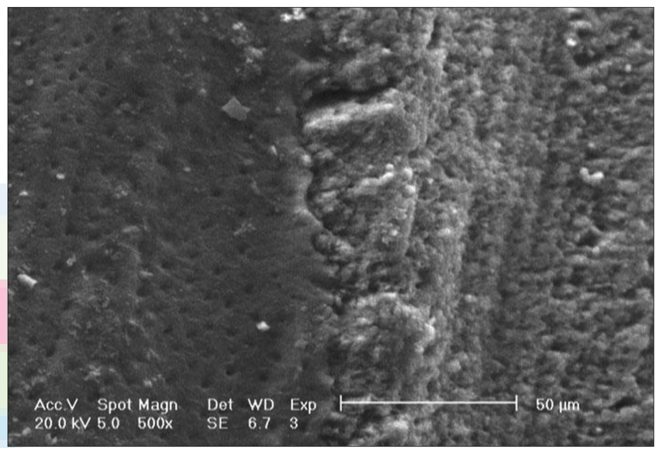

Figure 4: SEM photomicrograph of a sample from group 4, showing the interfacial failure and cohesive failure in dentin. Left side of the photograph shows the conditioned dentin with open tubules and the right side shows fractured dentin

After applying the primer on the conditioned dentin and penetrating the monomers into the nanospaces of collagen fibers, the evaporation of solvent must be done. The importance of the primer application method in the resultant bond strength of adhesives have been emphasized in previous studies;[8,17] but the specific effect of different air blowing periods on the acetone based total-etch systems has not been previously investigated. This study assessed the effect of different air-drying periods on the microtensile bond strength of an acetone based total-etch system. Prime and Bond NT, a total-etch adhesive used in this study, has consistently demonstrated a high bond strength in previous studies,[18,19] so it was used in this investigation as well.

Manufacturer's recommendation has advised 5 sec air-drying for this adhesive. In different clinical situations, the application of 5 sec air-drying is not usually possible. In other hand, this is probable that different areas of the cavity receive more air blow, thus the evaporation occurs more than the manufacturer's recommendation. According to a pilot study performed prior to the investigation different air-drying periods (0, 2, 5, 10, 30 sec) were used.

Different evaporation periods between 0 to 40 sec have been examined in previous studies.[10,14,17,20] it seems that for acetone based adhesives such as Prime and Bond NT, 30 sec air-drying period results in complete evaporation of the solvent and there is no need for examining the longer periods.

Mean bond strengths of the adhesive used in the study were between 5-35 MPa. Other studies reported the same bond strength range for this adhesive.[21]

Based on the results of the study, maximum bond strength belonged to group 3 (manufacturer's recommendation air-drying period) which was significantly higher than the other groups. The lowest bond strength was seen in group 1 (no evaporation). With increasing the air-drying periods the bond strength increased until 5 sec air-drying; after that increasing the air-blowing, decreased the bond strength.

Previous studies have emphasized on the importance of solvent evaporation in the resultant bond strength of different adhesives to dentin.[10,14,20]

Miyazaki *et al*.[17] have shown that maximum shear bond strength of ScotchBond MP to dentin was observed in 1-5 sec air-drying time and for Imperva Bond the optimum time for drying was 10-30 sec. Sadr *et al*.[10] showed that the bond strengths of self-etch adhesives to dentin were increased until 5 sec air-drying. Ten sec air-drying did not show any significant difference with the 5 sec group. These studies confirmed our results that there is an optimum air-drying time for achieving maximum bond strength.

Jacobson *et al*.[20] found out that increasing the air-drying time of self-etch adhesives increases the shear bond strength. Our results did not confirm this finding; probably because of the differences in the adhesives and the bond strength methods used in these studies.

In no evaporation group, most failures were interfacial and cohesive in adhesive [Figure 1]. Indeed, incomplete solvent evaporation or the entrapment of solvent within resin-dentin interfaces may result in dilution and/or phase separation of the adhesive resin tags.[8] Low bond strengths in group 1 were probably a result of weak adhesive layer.

Groups 2 and 4 showed an interfacial and cohesive failure in dentin that indicates a strong bond to dentin [Figure 4]. More complicated failure mode was seen in group 3, accompanied with the strongest microtensile bond strength between groups [Figures 2 and 3]. Low microtensile bond strength and a pure interfacial failure belonged to group 5. In fact, strong air blowing may have caused over removal of the adhesive resin causing incomplete enveloping of the collagen fibrils. Another reason for the poor adhesion in this group is that when strong air blowing is used, the solvent is evaporated quickly resulting in a viscous resinous material with entrapped air bubbles, remaining on the dentin surfaces. This would lead to weaker mechanical properties resulting in lower bond strength.[11]

The differences found between microtensile bond strengths of the groups were confirmed by morphological findings.

It is speculated that there is an optimum air-drying time for the adhesive used in this study. The strongest microtensile bond strength and the most complicated morphological bond failure belonged to 5 sec air-drying period group. No evaporation and excessive evaporation (30 sec) could lead to weak bonding.

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