Original Article

Effect of Solvent Removal on Microtensile Bond Strength of Etch and Rinse Systems under Vigorous Application to Wet and Dry Dentin

Mansoreh Mirzaei[1], Esmaeil Yassini[2], Farzaneh Aghagani[3], Narges Ezzati Givii[4], Mohammad Javad Kharazi Fard[5].

[1]Member of Dental Research Center and Assistant Professor, Department of Operative Dentistry, School of Dentistry, Tehran University of Medical Sciences, Tehran, Iran
[2]Member of Dental Research Center and Professor, Department of Operative Dentistry, School of Dentistry, Tehran University of Medical Sciences, Tehran, Iran
[3]Researcher & Member of Dental research center, School of Dentistry, Tehran University of Medical Sciences, Tehran, Iran
[4]Postgraduate Student, Department of Periodontology, School of Dentistry, Ahvaz University of Medical Sciences, Ahvaz, Iran
[5]Research Member of Dental Research Center Tehran University of Medical Sciences, Tehran, Iran.

Abstract

Background and Aim: Adhesive systems are commonly used for restorative dental procedures. Solvent removal my be effective for increasing the microtensile bond strength of etch and rinse systems to dentin. The aim of this study was to evaluate the effect of solvent removal on microtensile bond strength of etch and rinse systems to wet and dry dentin.

Materials and Methods: This in-vitro study was conducted on 40 intact human extracted third molars. A flat superficial dentin surface was exposed by wet abrasion. The specimens were randomly assigned to five groups. Two coats of the solvent-based (SB) and solvent-free (SF) adhesives were applied to dry (D) or rewetted (W) surfaces, under vigorous rubbing action after phosphoric acid etching according to the manufacturer's instructions. The examined groups included: group one: adhesive with solvent on dry dentin, group two: adhesive without solvent on dry dentin, group three: adhesive with solvent on wet dentin, group 4: adhesive without solvent on wet dentin and group 5 (control group). After light curing (600mW/cm²/10 s), composite build-ups were constructed incrementally and specimens were stored in water (37°C/24 h). They were longitudinally sectioned in the "*x*" and "*y*" directions to obtain bonded sticks (1mm²) for immediate testing at a crosshead speed of 1 mm/min. The resultant bond strength was expressed for different fracture patterns. The microshear bond strength test was carried out in a Universal Testing Machine. The data were analyzed using two-way ANOVA. All statistical analyses were performed using SPSS version 2 software. $p<0.05$ was considered significant.

Results: The highest bond strength was achieved in group 1 and the lowest in group 4; but the differences among groups in this respect were not statistically significant

Conclusion: Within the limitations of this study, the results showed that presence of solvent was not essential to achieve high bond strength values to dry and wet dentin when applied vigorously.

Key Words: Adhesive system, Solvent, Moisture, Bond strength

Corresponding author:
Esmaeil Yassini, Member of Dental Research Center and Professor, Department of Operative Dentistry, School of Dentistry, Tehran University of Medical Sciences, Tehran, Iran
yassini_e@yahoo.com

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Introduction

The efficacy of enamel bonding systems has been confirmed; however, controversial results have been obtained for bonding to dentin and cement [1]. Tubular structure of dentin, its permeability and organic composition compromise dentin bonding [2, 3]. Constant development of adhesive systems in the recent years has resulted in dentin bond strength similar to that of enamel [4, 5]. Application of phosphoric acid to dentin results in collagen fiber compaction and if accompanied by drying after dentin etching, leads to the collapse of collagen network and compromises resin penetration [6]. Consequently, lower immediate bond strength values are obtained [7]. Although hydration of fibers restores their plasticity [6], the level of reversibility remains unknown [8]. In general resin-dentin bond depends on the removal of liquid monomers from the interfibrillar space that used to be occupied by hydroxy apatite crystals [9]. In fact, micromechanical interlocking via the hybrid layer is known as the main mechanism of resin-dentin bond [4]. Although the hybrid layer plays a role in resin-tooth bonding, the tubular occluding ability of materials does not depend on the thickness of hybrid layer. What is important is the presence of a continuous, uniform hybrid layer with no interruption. Some believe that the hybrid layer is the site of bonding interface failure. In order to achieve an adequate bond, all porosities due to demineralization must be filled to prevent any micro porosity in this layer [6]. Hybrid layer can help preserve the integrity of hybrid dentin and protect it from the stresses of polymerization shrinkage. Also, it acts as a shock absorber and improves the bond strength. Higher tensile bond strength and less marginal microleakage due to the increased thickness of the adhesive layer and consequently improved distribution of load in the bonded complex as well as decreased strain due to composite resin polymerization shrinkage have been previously described.

Researchers believe that the adhesive layer thickness is potentially effective on the bond strength of the current bonding systems [10]. Single-step adhesives contain organic solvents in their composition added for the purpose of surface wetting and enhancement of the effect of adhesive. They increase bond strength especially when the dentin is moist or has been re-wetted [1]. To improve dentin bonding, adhesive systems contain a primer solved in an organic solvent [6]. Due to low molecular weight and high diffusion ability, these primers decrease the viscosity of the solution and increase hydrophilicity [11]. These solvents include acetone, ethanol and water. Each solvent has a different effect on the stability of adhesives. Thus, it can be concluded that use of ethanol results in products with a stability in between that of water and acetone. Water in combination with acetone or ethanol provides greater stability than products using an organic solvent alone [4]. Acetone-containing adhesives compared to water- or ethanol-containing ones are more sensitive to repeated use and reduction of bond strength [12]. However, almost all three solvents are completely eliminated after 75 days; in fact, the speed of evaporation varies by time [13]. A noteworthy issue is that when adhesives are applied the highest amount of solvent possible must be vaporized because the solvent remnants can prevent polymerization and compromise the fracture resistance [14]. There is a possibility that vigorous application of adhesive results in better penetration of monomer into the collagen network while the solvent is pushed out and removing the excess water improves the mechanical properties of the hybrid layer [15]. Previous laboratory findings have demonstrated that vigorous application of adhesive increases both immediate and 6-month bond strength compared to its gentle application. However, retention was not significantly different between methods of application at 24 months and the amount of retention at 24 months after vigorous application was 92.5% and almost similar to the baseline value (100%). However, this rate was lower in other application methods (about 82.5%) [16]. In other words, the immediate bond strength of Total Etch systems to dentin depends on both adequate surface moisture and type of solvent [5]. However, ideally, all the solvent and water must be vaporized prior to curing; otherwise they are trapped in the adhesive resin during the process of curing [17]. Due to higher technical sensitivity of the bonding in presence of moisture and solvent, obtaining a system that enables adequate bond strength in absence of dentin moisture or solvent or allow more efficient solvent removal would be optimal. It appears that SF bonding systems have adequate clinical function. The amount of solvent in adhesive bottles significantly decreases following frequent opening and closing the bottle during the day. Thus, the adhesive used has a probably greater concentration than the product originally manufactured by the company [18]. This study aimed to assess the effect of solvent removal on the μTBS of etch and rinse systems when adhesive is vigorously applied to wet and dry dentin.

Materials and Methods

This in vitro experimental study evaluated One Step Plus (OS P, Bisco, Schaumburg, IL, USA) filled adhesive with acetone solvent from the etch and rinse system.

Two bottles of the adhesive were used. Solvent was eliminated from one bottle as follows: The adhesive was placed on a glass slab with a known weight and weighed. The glass slab was then placed in a dry oven at 37°C and weighed at one hour time intervals to reach a fixed weight indicative of complete vaporization of solvent.

All specimens were protected from light by filters to prevent possible polymerization prior to the experiment. A total of 40 intact human third molar teeth were collected after obtaining patient consents and restored in 0.5% Chloramine T solution for less than a week. The teeth were then stored in distilled water at 4°C. The teeth had been extracted less than 6 months earlier. The specimens were then prepared as follows: The occlusal enamel was removed by 180 grit silicon carbide papers along with water coolant to expose the dentin. To achieve a standard smear layer, the teeth were ground with 320 and 600 grit silicone carbide papers for 30 seconds each. Specimens were then etched with 32% etchant (UNI-ETCH, Bisco, Schaumberg, IL, USA) for 15 seconds followed by 20 seconds of rinsing with water and drying for 10 seconds from a 20cm distance with oil and water free air spray. The specimens were then randomly divided into 5 groups of 8:

Group 1: Specimens were remained dry and a layer of adhesive containing acetone solvent was applied.

Group 2: Specimens were remained dry and a layer of acetone-free adhesive was applied.

Group 3: Specimens were rewetted by a micropipette containing 2.5 µL water for 10 seconds and a layer of adhesive containing acetone was applied.

Group 4. Specimens were rewetted by a micropipette containing 2.5 µL water for 10 seconds and a layer of acetone-free adhesive was applied.

Group 5. Specimens were prepared according to the manufacturer's instructions (control group).

Adhesive was applied with 34.5±6.7 g manual force to the entire surface for 10 seconds except for the control group where adhesive was applied with mild pressure to dentin surface for 10 seconds. The teeth surfaces in all groups were then air dried from 20cm distance for 10 seconds. The second layer of adhesive was applied similar to the first layer. The time spent on dentin surface wetting and application of adhesive was equal to the time required for opening the adhesive bottle, soaking the micro-applicator and application of adhesive to the tooth surface. The adhesive layer was then light cured using a light curing unit (Blue Phase, Ivoclar Vivadent, FL-9494 Schaan, Lichtenstein) with an intensity of 450 mW/cm^2 recommended by the manufacturer for 10 seconds. Composite build-ups (AELITE All-Purpose Body, Dark Opaque, A3.5-O, Bisco, Schaumberg, IL, USA) were fabricated on each specimen as three increments of one millimeter each. Each layer was cured by 450 mW/cm^2 light intensity for 30 seconds as recommended by the manufacturer. All steps were carried out by a single operator at room temperature and relative humidity. Eight teeth based on the surface moisture and presence of solvent in each group were used. Specimens were stored in distilled water at 37°C for 24 hours. In groups where SF adhesive was applied, only the amount of adhesive required for one day of work was subjected to solvent removal. Specimens were mounted in polyester material and then sectioned perpendicular to the adhesive interface in X and Y axes in such way that a cubic stick with one mm^2 surface area was obtained. The specimens were then subjected to loading in Microtensile Testing Machine (Bisco, Schaumburg, IL, USA) at a crosshead speed of 1mm/min until fracture. Specimens were mounted in the machine in such way that the interface was at the midline of load application. Thus, the stress accumulation area exactly corresponded to the interface. The mode of failure was recorded and the results were analyzed using two-way ANOVA.

Results

This study aimed to assess the microtensile bond strength of etch and rinse adhesives with and without solvent to wet and dry dentin by vigorous application of adhesive. One-way ANOVA was applied and after eliminating the control group, presence or absence of solvent was compared using two-way ANOVA. Data were analyzed using SPSS 2 and type 1 (α) error was considered as 0.05.

The highest bond strength was achieved in group 1 (SB adhesive on dry dentin) and the lowest in group 4 (SF adhesive on rewetted dentin); however, two-way ANOVA found no significant difference among groups.

Mode of failure: Thirty specimens in groups 1, 3 and control (75.93%) and 31 specimens in groups 2 and 4 (87.96%) showed failure at the interface. By eliminating the control group, type of dentin (p=0.284), presence or absence of solvent (p=0.196) and the interaction of the two (p=0.843) had no significant effect on bond strength (Table 1).

Table 1. Mode of failure of specimens

-	Adhesive failure	Cohesive failure	
		Dentin	Composite
Control group	30(%93/75)	%0	2(%6/25)
1	30(%93/75)	%0	2(%6/25)
2	31(%96/87)	%0	1(%3/12)
3	30(%93/75)	%0	2(%6/25)
4	31(%96/87)	%0	1(%3/12)

Discussion

Since the introduction of the concept of wet bonding to dentin, dentin drying is not much popular. Drying the demineralized dentin results in some changes in the collagen network that prevent monomer penetration [19]. It has been reported that in these conditions, collagen fibrils contact each other and hydrogen bonds are formed between them resulting in a compact matrix allowing very small penetration of adhesive resins [20]. These conditions were provided in this study by changing the manufacturer's instructions and air-drying the demineralized dentin.

It is believed that the only way to overcome this issue is to recreate interfibrillar spaces in dried, demineralized dentin [21]. Thus, several researchers have obtained high bond strength by maintaining the demineralized dentin wet prior to the application of adhesive; this process is known as wet bonding [22]. In agreement with these results, previous studies have shown that diffusion of resin bonding agent in the hybrid layer in acetone based systems decreases by 50% if applied on dry instead of wet dentin [23]. Studies have shown that the amount of water required for maximum bond strength is different based on the system used and acetone-based systems require a wetter surface [7]; however, recent studies have questioned this concept.

The role of acetone in adhesive solutions is based on three mechanisms: acetone decreases the viscosity of adhesive and increases the diffusion of bonding agent into the demineralized and collagen-rich dentin. Due to the decreased surface tension of water, acetone acts as a water chaser and eventually acetone increases the vapor pressure of water. A consensus has been reached that elimination of water increases surface collagen and is later substituted by acetone and then adhesive resin [24, 25]. Due to higher vapor pressure and low boiling temperature, acetone needs precise maintenance and care especially in products where acetone has a higher percentage than other ingredients [4]. Due to the volatile nature of these carriers, their concentration in single-bottle adhesives decreases over time. Frequent applications and high temperature can facilitate this process leading to the formation of a weak hybrid layer and subsequently decreased bond strength [26]. The effect of frequent use of single-bottle adhesives on bond strength reduction has been confirmed to some extent [4, 27]. Also, acetone-containing adhesives compared to those with ethanol and water are more susceptible to frequent application and decrease in bond strength [12]. Reis and Dal-Bianco demonstrated that application of bonding resin to the dentin surface has a more significant effect than the moisture of the acid etched dentin [15, 28]. The longer the process of resin application and the higher the pressure when applied to dentin, the higher the immediate and long-term bond strength irrespective of the dryness or wetness of dentin. In our study, adhesive was vigorously applied to dentin; which may explain the higher bond strength of adhesive to dried acid etched dentin. One possible theory is that by following the manufacturer's instructions resin monomers, especially those with a molecular weight higher than that of adhesive, have limited penetration into the demineralized moist dentin. Thus, vigorous application of adhesive enables better penetration of monomer into the collagen network. At the same time the solvent is pushed out and by increased removal of the excess water, the mechanical properties of the hybrid layer improve [15]. On the other hand, there is a possibility that by vigorous application of adhesive, the collapsed collagen network is squeezed like a sponge and by removing the pressure, it bounces back into its baseline position and the adhesive is suctioned into the collapsed collagen network [25]. These researchers have claimed that vigorous application decreases the vaporization of acetone to the extent that the adhesive destructively looses its flow and forms a gel-like structure like HEMA that cannot penetrate in between the nanometer-scale spaces in between collagen fibrils. The current study showed that the bond strength of acetone-based adhesive increased by its vigorous application to dentin surface. Solvents are used in etch and rinse adhesives to enable adequate monomer penetration into the moist collagen network. At the same time, they decrease the viscosity of adhesive improving its diffusion into the micron-scale porosities in dentin surface [29]. Ries et al. demonstrated that the degree of conversion in SF single-step and one-step adhesives did not change or increased compared to that in SB adhesives and both achieved bond strength as high as that of SB adhesives. Such result was not reported in previous studies by Reis [7]. Reis discussed that presence of solvent is necessary to achieve an effective bond to dentin but did not report how the adhesive was applied and just mentioned that adhesive was used according to the manufacturer's instructions. The difference between our study and that of Reis may be due to the method of application of adhesive. When the adhesive is applied to the surface of demineralized dentin vigorously, monomers are pushed into the nanometer-scale spaces in between collagen fibrils enabling better infiltration of resin. Loguercio demonstrated that the effect of solvent removal was not equal on the bond strength of different adhesives to dentin. Opti-Bond Solo was adversely affected by the solvent removal. Resin-dentin bond strength and the degree of conversion were significantly lower in SF adhesives. One main difference of these adhesives with other materials was that the manufacturer claims that they are filled by 24wt%. Their viscosity is significantly higher than that of other products and this may explain their lower performance after solvent removal. Increased viscosity adversely affects the flexural bond strength and the degree of conversion of adhesives. These results are different from our findings. It appears that SF one step adhesives have a clinically superior efficacy. It has been demonstrated that the solvent content of adhesive bottles decreases over time as the result of frequently opening and closing the bottle and clinicians do not pay attention to this fact. Thus, it is highly likely that in routine clinical practice, adhesive with a concentration higher than that produced by the manufacturer is applied [18]. One Step Plus bonding contains approximately 10% of glass filler. Based on the results, it can be stated that this fact is not a matter of significance until non-filled or slightly filled adhesives are applied vigorously to dentin surface. Based on the results, solvent removal from the adhesive is not recommended and further studies are required in this respect. The effect of solvents and their concentration on the immediate bond of adhesives must be further evaluated because the remaining solvent may compromise polymerization due to dilution of monomer and result in a polymer with low mechanical properties, high water sorption and low hydrolytic stability. Moreover, it is clinically difficult, if not impossible, to vaporize the water and solvent remnants trapped in the demineralized dentin as the result of short period of air-drying. Further studies are required to evaluate the micromorphology of the adhesive surface and the long-term bond strength. This study showed that if adhesive is applied vigorously, high bond strength may be obtained even in case of decreased solvent and dentin moisture.

Conclusion

Within the limitations of this study, the results showed that if adhesive is applied vigorously, high bond strength to dentin may be achieved even in case of decreased solvent or dentin moisture. Thus, factors other than the method of application of adhesive, such as air pressure, temperature, angle of the air spray and its distance from the surface, type of solvent present in the adhesive and solvent vaporization are inevitably important for achieving high bond strength values.

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