

Enhancing bond stability: impact of adhesive thinning on resin-dentin bonded interfaces

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Abstract. – OBJECTIVE: This study aimed to investigate the effect of adhesive thinning on resin-dentin-bonded interfaces created by two simplified adhesives.

MATERIALS AND METHODS: Micro-tensile bond strengths and interfacial nanoleakage were evaluated within bonded dentin interfaces formed by Adper Single Bond 2 and Single Bond Universal after 24 hours and 6 months of water storage. The adhesives were subjected to three different techniques: air-thinning, brush-thinning, or application without thinning. Statistical analysis was performed using a multi-level analysis of variance followed by Bonferroni's post-hoc test, at a significance level of 0.05.

RESULTS: Adper Single Bond 2 demonstrated the highest immediate microtensile bond strengths (43.5 ± 1.3 MPa) and the lowest immediate nanoleakage ($49.8 \pm 2.8\%$) when air-thinning was employed. Single Bond Universal exhibited the lowest nanoleakage ($36.4 \pm 1.8\%$) when air-thinning was used, although there was no significant difference in immediate bond strengths between air-thinning and brush-thinning approaches, which both showed higher values compared to the no-thinning approach. After 6 months of storage, a significant decrease in bond strengths and a significant increase in nanoleakage were observed across most groups ($p < 0.001$).

CONCLUSIONS: While all groups displayed varying degrees of instability over a 6-month storage period, air-thinning of simplified etch-and-rinse and self-etch adhesives proved to provide clinically acceptable bonded interfaces. The findings suggest that adhesive thinning techniques can play a vital role in enhancing bond stability and longevity in resin-dentin-bonded interfaces.

Key Words:

Simplified adhesives, Microtensile bond strength, Nanoleakage, Air-thinning, Scanning Electron Microscopy.

interface¹. Most current adhesive systems develop high and efficient immediate bonding; however, it deteriorates dramatically over time, unfortunately². The properties of each adhesive system, as well as the technique of application, affect the durability of the bonded interface¹. Adhesive-thinning is used before light curing, and it plays an important role in influencing the effectiveness of the resultant bonds achieved during clinical procedures^{3,4}.

Simplified adhesives are mainly composed of a mixture of functional monomers, fillers, solvents, and water. To allow proper interaction with dentin, a complex blend of hydrophilic and hydrophobic components is important, which results in increased hydrophilicity of simplified adhesives⁵. Solvents present within bonded interfaces may interfere with resin polymerization and increase water sorption from the underlying wet dentin, which will eventually result in impaired resin-dentin interface durability^{1,6}. Consequently, air thinning of the adhesive before light curing is performed to remove solvents present within the bonded interface^{1,7-9}. However, strong air thinning of dental adhesives was reported¹⁰ to lower the bond strengths as it may greatly decrease the adhesive thickness such that the entire layer is oxygen-inhibited and does not polymerize.

In-vitro studies¹¹ have shown that the microleakage score was higher in the gingival margin at the enamel-adhesive interfaces and in the occlusal margin at the adhesive-metal bracket interfaces. Bracket debonding remains the main concern during orthodontic treatment, despite the new techniques. The 3-step etch-and-rinse and the 2-step self-etch adhesives have a separate bond step, which does not contain any solvent, and thus it is relatively hydrophobic¹. However, air thinning of such adhesives is still recommended to allow the adhesive to spread and penetrate the tooth structure and produce uniform adhesive thickness. The

Introduction

The longevity of adhesive restorations depends mainly on the durability of the resin-dentin

effect of adhesive thickness on bonding durability is controversial in the literature. It was reported that a thick adhesive layer may act as a stress absorber during resin composite polymerization, thus increasing bond strength and reducing interfacial nano leakage¹¹. However, other studies¹²⁻¹⁴ reported that thick adhesive layers either have no effect¹² or decrease bond strengths^{13,14}. Since dental adhesives have minimal filler loading, they have reduced mechanical properties, increased polymerization shrinkage, and water sorption, which adversely affect bond durability^{13,14}. Besides air thinning, the adhesive layer thickness can be greatly affected by the chemical composition of the bonding system used. Highly viscous adhesives will not be able to get to the tooth surface easily and are more likely to produce thick layers¹⁵.

The objective of this study was to evaluate the effect of adhesive thinning on dentin bond strength and nano leakage associated with two simplified adhesives immediately and after aging. The null hypotheses tested were: (1) Adhesive thinning protocols have no effect and do not affect immediate bond strength and nano leakage of both adhesives as compared to control, and (2) six-month-water storage does not affect the bond strength and nanoleakage of each adhesive.

Materials and Methods

Specimen Preparation and Bonding Protocols

Freshly-extracted 60 non-carious human molars were used according to the protocol (#229-03-21), approved by the Research Ethics Committee of King Abdulaziz University, Saudi Arabia. The teeth were preserved in 0.5% Chloramine T solution at 4°C for up to 2 months before use. Perpendicular to the long axis of each tooth, sectioning was done underneath the dentin-enamel junction using a low-speed diamond saw (Micromet AG, Munich, Germany) under copious

water irrigation. A 600-grit silicon carbide paper (Norton Saint-Gobain Abrasives, Worcester, MA, USA) was used for 1 min under water-cooling to obtain a standardized smear layer on the exposed flat mid-coronal dentin.

The polished teeth were divided into 2 main groups (n=30) according to the type of adhesive used: 1) Adper Single Bond 2 (3M ESPE, St Paul, MN, USA). 2) Single Bond Universal (3M ESPE, Deutschland GmbH, Neuss, Germany). Each group was divided equally and randomly into 3 subgroups (n= 10), according to the adhesive thinning method; 1) control (no adhesive thinning), 2) adhesive gentle thinning with a dry brush prior to curing, 3) adhesive gentle air-thinning for 5 sec before curing. The chemical composition of the adhesives used is presented in (Table I).

Bonding procedure

A single operator (M.M.A-N.) performed all bonding steps according to the manufacturer's instructions for each adhesive system as follows:

- For the Adper Single Bond 2 group, acid etchant (3M ESPE Scotchbond™, Neuss, Germany) was applied to dentin for 15 seconds and then was rinsed for 10 seconds. The excess water was blot-dried using a cotton pellet, leaving the surface glistening without the pooling of water. Two consecutive coats of the adhesive were applied to etched dentin for 15 seconds with gentle agitation using a fully saturated applicator (Pearson Dental Supply, CA, USA). On the other hand, for the Single Bond Universal group, two consecutive coats of the adhesive were applied to dentin by scrubbing action for 20 seconds.

- For each adhesive system used, three scenarios were utilized. 1) No adhesive thinning was performed, and light-curing was done immediately for 10 sec. 2) A dry micro brush (Changzhou Medical Instrument Co., Ltd., Jiangsu, China) was used to perform gentle adhesive thinning before light curing. 3) Gentle adhesive air-thinning for 5 sec was performed before light-curing.

Table I. Dental adhesives used and their composition.

Adhesive System	Category	Composition	Manufacturer
Adper Single Bond 2	2-Step Etch and Rinse Adhesive	Dimethacrylate resins, 2-hydroxyethyl methacrylate (HEMA), methacrylate-modified polyalkenoic acid copolymer (Vitrebond Copolymer), filler, ethanol, water, initiators.	3M ESPE, St. Paul, MN, USA
Single Bond Universal	1-Step Self-Etch Adhesive	Methacryloyloxydecyl dihydrogen phosphate (MDP) phosphate monomer, methacrylate resins, HEMA, Vitrebond Copolymer, filler, ethanol, water, initiators, and silane.	3M ESPE, Deutschland GmbH, Neuss, Germany

After adhesive application to dentin, resin composite build-ups were constructed on top of the bonded dentinal surfaces in five 1-mm thick increments of nanohybrid resin composite (Filtek Z 350 XT, 3M ESPE, St. Paul, MN, USA) and each increment was light-cured for 20 s (Light Emitting Diode curing unit, 3M ESPE Elipar, Germany, 1,200 mW/cm², 430-480 nm).

All bonded specimens were stored in distilled water at 37°C for 24 hours. Then, each bonded tooth was cut perpendicular to the bonding interface into 16 (0.9 mm x 0.9 mm) sticks using the non-trimming technique¹⁶. The sticks of each tooth were kept in distilled water in separate containers. The specimens of each subgroup were further divided into 2 storage periods (n=5): 24 hours and 6 months storage in distilled water at 37°C in an incubator. From each tooth, 15 sticks were used for the microtensile bond strength test, and 1 stick was used for nanoleakage evaluation. The experimental design of the current study is presented in Figure 1.

Micro-Tensile Bond Strength Evaluation (μ TBS)

The dimensions of each stick were measured using a digital caliper (Dasqua tools, Sichuan, China), accurate to 0.01 mm, and were recorded.

Each stick was fixed to a micro-tensile testing machine (Bisco Inc., Schaumburg, IL, USA) using Zapit adhesive (Dental Ventures of America, Corona, CA, USA) and was stressed under tension at a crosshead speed of 1 mm/min until failure. The tensile force at failure was recorded and then divided by the cross-sectional area of each stick to calculate the micro-tensile bond strength in megapascals (MPa). The mean of the bond strength values of 15 sticks was determined to give a value for each tooth. Then, a grand mean of the 5 teeth of each group was obtained; where the tooth was the statistical unit of this study.

A stereomicroscope (Meiji Techno Co., Ltd., Tokyo, Japan) was used to examine all fractured specimens at 30x magnification to determine the modes of failure, which were classified as adhesive (A), cohesive (C), or mixed (M) failures.

Interfacial Nanoleakage Evaluation

Two layers of nail varnish (Shenzhen Meixin Industry Co., Ltd., Guangdong, China) were used to coat the entire bonded specimens except for 1 mm from the interface. The sticks were then immersed in 50% ammoniacal silver nitrate solution for 24 h, rinsed with water then placed in a photo-developing solution for 8 h under fluorescent light.

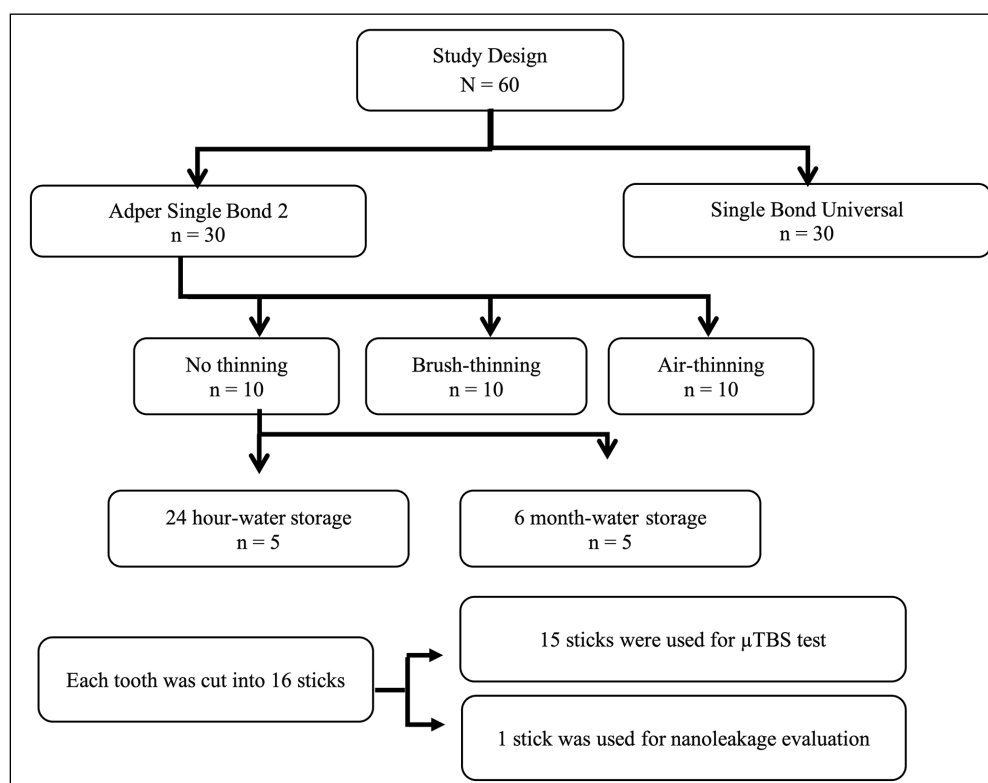


Figure 1. Flow chart presenting the experimental design of the current study.

The sticks were finally polished using silicone-carbide papers (600-1,200 grit) followed by 0.05 mm alumina particle suspension (Buehler, Lake Bluff, IL, USA) using a soft polishing cloth. Ultrasonic cleaning of all specimens in distilled water was done for 30 min (Ultrasonic Cleaning System 2014, L&R Manufacturing, Kearny, NJ, USA).

An environmental scanning electron microscope (SEM, Quanta 200 ESEM, FEI France, Mérignac, France), operated in the backscattered electron mode, was used to examine resin-dentin interfaces at 2,000x magnification. Quantitative analysis of the amount of silver nitrate present within the hybrid layer was performed using image analysis software (NIH Image, Scion Corp., Fredrick, MD, USA)¹⁷.

Statistical Analysis

Numerical data were explored for normality by checking the distribution of data and using Kolmogorov-Smirnov and Shapiro-Wilk tests of normality. All data were normally distributed, necessitating a parametric approach for the analysis. Data were presented as mean and standard deviation (SD) values. For the microtensile bond strength test, statistical analysis was performed using the tooth as the statistical unit ($n=5$), not the stick. A multi-level analysis of variance (ANOVA) test was used to study the effect of adhesive type, adhesive thinning approach, storage

time, and their interactions on micro-tensile bond strength and nano leakage percentage. Bonferroni's post-hoc test was used for pair-wise comparisons when the ANOVA test was significant. The Chi-square test was used to compare the various failure modes over both storage periods and different adhesive thinning protocols. The significance level was set at $p \leq 0.05$. Statistical analysis was performed with IBM SPSS Statistics for Windows system (Version 23.0. IBM Corp., Armonk, NY, USA).

Results

Microtensile Bond Strength Test

The mean values of μ TBS for both adhesives used with or without adhesive thinning after 24 h and 6 m of water storage are presented in Table II. There was a significant difference between the thinning techniques ($p < 0.001$) when Adper Single Bond 2 was used at both 24 h and 6 months storage periods. Pair-wise comparisons between techniques revealed that air-thinning showed the highest mean micro-tensile bond strength, followed by brush-thinning, while no thinning showed the lowest mean micro-tensile bond strength.

For Single Bond Universal after 24 h storage, a significant difference was found between thinning techniques ($p < 0.001$). On the other

Table II. The mean, standard deviation (SD) values, and results of the three-way ANOVA test for comparison between micro-tensile bond strength (MPa) values with different interactions of variables.

Time	Thinning	Adper Single Bond		Single Bond Universal		<i>p</i> -value (Between adhesives [§])
		Mean	SD	Mean	SD	
24 hours	No thinning	24.6 ^C	1.8	21.5 ^B	2.5	0.031*
	Brush-thinning	39.3 ^B	2.4	37.8 ^A	2.6	0.281
	Air-thinning	43.5 ^A	1.3	39.7 ^A	1.3	0.008*
<i>p</i> -value (Between thinning protocols)		<0.001*		<0.001*		
6 months	No thinning	18.7 ^C	2.5	15.1 ^C	2.9	0.013*
	Brush-thinning	32.1 ^B	2.6	29.6 ^B	2	0.076
	Air-thinning	38.7 ^A	2.2	37.2 ^A	1.4	0.280
<i>p</i> -value (Between thinning protocols)		<0.001*		<0.001*		
<i>p</i> -value (Between times [†])		<0.001*		<0.001*		
	No thinning	<0.001*		<0.001*		
	Brush-thinning	<0.001*		<0.001*		
	Air-thinning	0.001*		0.076		

Uppercase letters indicate a statistically significant difference ($p \leq 0.05$) in the same vertical column (different thinning protocols).

*Statistically significant at $p \leq 0.05$. [§]*p*-value for comparison between the two tested adhesives at the same time point. [†]*p*-value for comparison between different time points for the same adhesive and thinning protocol.

Table III. The mean, standard deviation (SD) values, and results of the three-way ANOVA test for comparison between nano leakage (%) values with different interactions of variables.

Time	Thinning	Adper Single Bond		Single Bond Universal		<i>p</i> -value (Between adhesives [§])
		Mean	SD	Mean	SD	
24 hours	No thinning	79.8 ^A	3.4	83.6 ^A	2.2	0.056
	Brush-thinning	58.6 ^B	2.8	53.3 ^B	2.4	0.008*
	Air-thinning	49.8 ^C	2.8	36.4 ^C	1.8	<0.001*
<i>p</i> -value (Between thinning protocols)		<0.001*		<0.001*		
6 months	No thinning	91.2 ^A	3.9	92.7 ^A	2.5	0.446
	Brush-thinning	75.4 ^B	3.4	72.5 ^B	3.8	0.141
	Air-thinning	60.2 ^C	3.9	43.2 ^C	2.9	<0.001*
<i>p</i> -value (Between thinning protocols)		<0.001*		<0.001*		
<i>p</i> -value (Between times [†])		No thinning	<0.001*	Brush-thinning	<0.001*	
		Brush-thinning	<0.001*	Air-thinning	<0.001*	
		Air-thinning	<0.001*		0.001*	

Uppercase letters indicate a statistically significant difference ($p \leq 0.05$) in the same vertical column. *Statistically significant at $p \leq 0.05$. [§]*p*-value for comparison between the two tested adhesives at the same time point. [†]*p*-value for comparison between different time points for the same adhesive and thinning protocol.

hand, pair-wise comparisons between techniques revealed that there was no significant difference between brush- and air-thinning; both showed higher mean micro-tensile bond strength than the no-thinning group. After 6 months of storage, a significant difference between thinning techniques was found ($p < 0.001$). Pair-wise comparisons between techniques revealed that air-thinning showed the highest mean micro-tensile bond strength, followed by brush-thinning, while the no-thinning group showed the lowest mean micro-tensile bond strength.

The mean micro-tensile bond strength values exhibited by all Adper Single Bond 2 groups dropped significantly after 6 months of water storage ($p < 0.001$). Air-thinned Adper Single Bond 2 exhibited an 11% bond strength reduction, brush-thinning resulted in an 18% bond strength reduction, while Adper Single Bond 2 without thinning exhibited a 24% bond strength reduction. On the other hand, the mean micro-tensile bond strength values exhibited by Single Bond Universal showed a significant drop after 6 months of water storage for no thinning and brush-thinning groups ($p < 0.001$). However, there was no significant difference for the air-thinning groups between both testing periods ($p = 0.076$). Bond strength reduction of Single bond universal groups after 6 months of water storage of air-thinning, brush-thinning, and no-thinning groups were 6%, 22%, and 30 %, respectively.

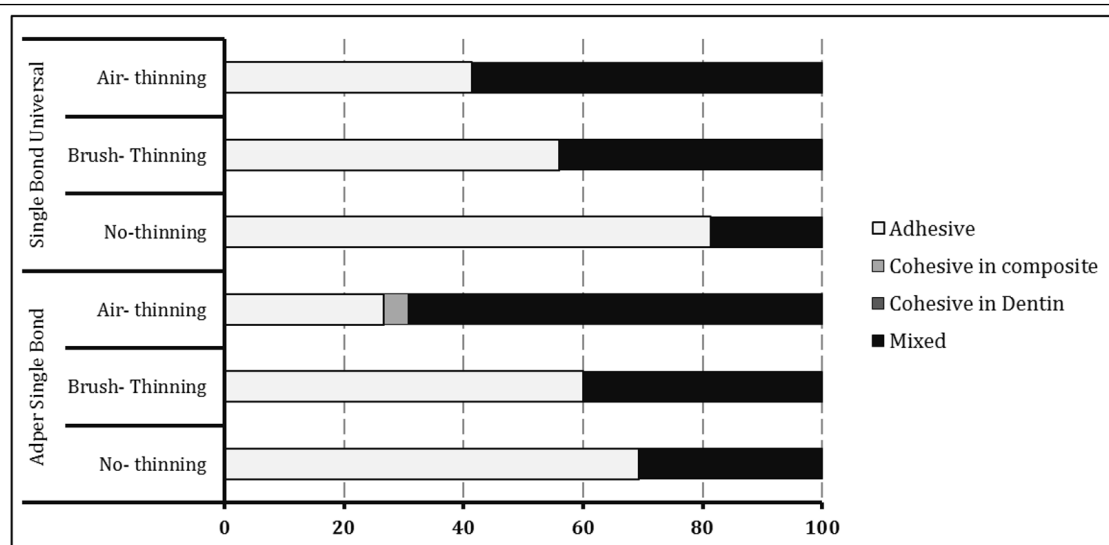
Failure Mode Analysis

The frequency (%) of fracture patterns is presented in Figure 2 A-B. There was a significant difference among the adhesive thinning groups ($p < 0.001$), as well as between 24 hours and 6 months' time points ($p = 0.0029$). There was no effect for the type of adhesive ($p = 0.109$).

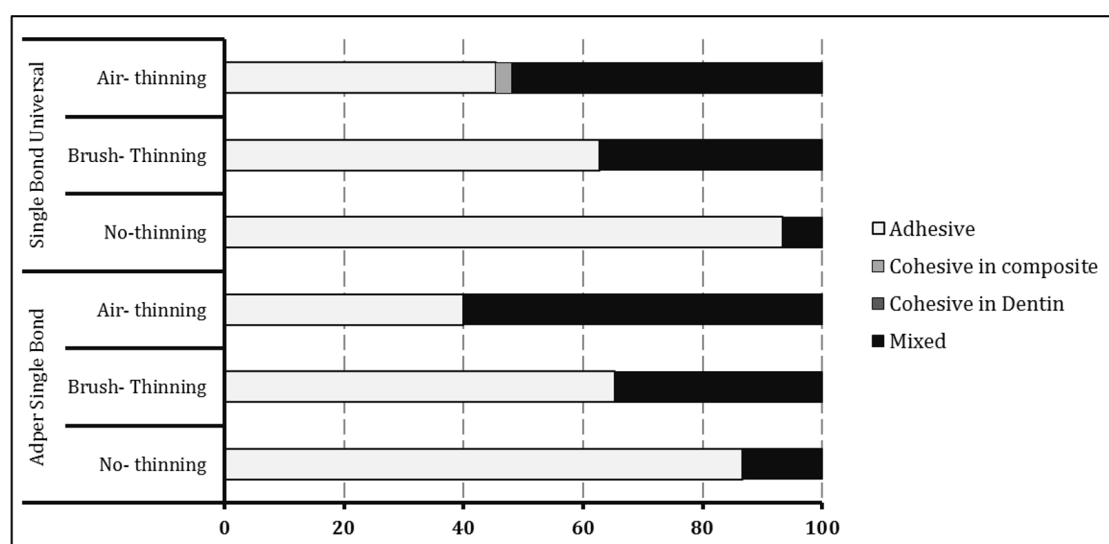
Adhesive failure was the dominating type of failure in all 24 h groups that received no-thinning and brush-thinning approaches. For the air-thinning approach groups, the mixed type of failure was predominating. After 6 months of water storage, adhesive failure increased for all groups and remained the dominating type of failure in both the no-thinning and brush-thinning approaches; however, the mixed type of failure was higher for the air-thinning approach.

Interfacial Nanoleakage Percentage

The mean values of nanoleakage percentage for both adhesives used with or without adhesive thinning after 24 hours and 6 months of water storage are presented in Table III. Adper Single Bond 2 specimens at each storage period (24 hours and 6 months) showed a significant difference between the 3 thinning approaches ($p < 0.001$). Pair-wise comparisons between the three approaches revealed that the no-thinning approach showed the highest mean nanoleakage %, followed by brush-thinning, while air-thinning showed the lowest mean nano-



A



B

Figure 2. A, Failure mode distribution (%) for each group following micro-tensile bond strength after 24 h of water storage. B, Failure mode distribution (%) for each group following micro-tensile bond strength after 6 m of water storage.

leakage %. Similarly, for Single Bond universal at both storage periods (24 hours and 6 months), there was a significant difference between the three thinning approaches ($p < 0.001$). Six months of water storage of Adper Single Bond 2 and Single Bond Universal specimens using each thinning approach (no-thinning, brush-thinning, and air-thinning) resulted in a significant increase in nanoleakage % when compared to 24 h storage specimens ($p < 0.001$).

Representative SEM pictures of nano leakage % of different groups at 2,000x magnification are shown in Figures 3 and 4. Among all 24 h groups, extensive silver deposits were observed within the hybrid layer in representative specimens of the no-thinning approach (Figure 4-a1) when compared to other approaches used (Figure 4-b1, c1). Moreover, silver deposits increased clearly in the 6 months representative specimens (Figure 4-a2, b2, c2) when compared to 24 h specimens (Figure 4-a1, b1, c1).

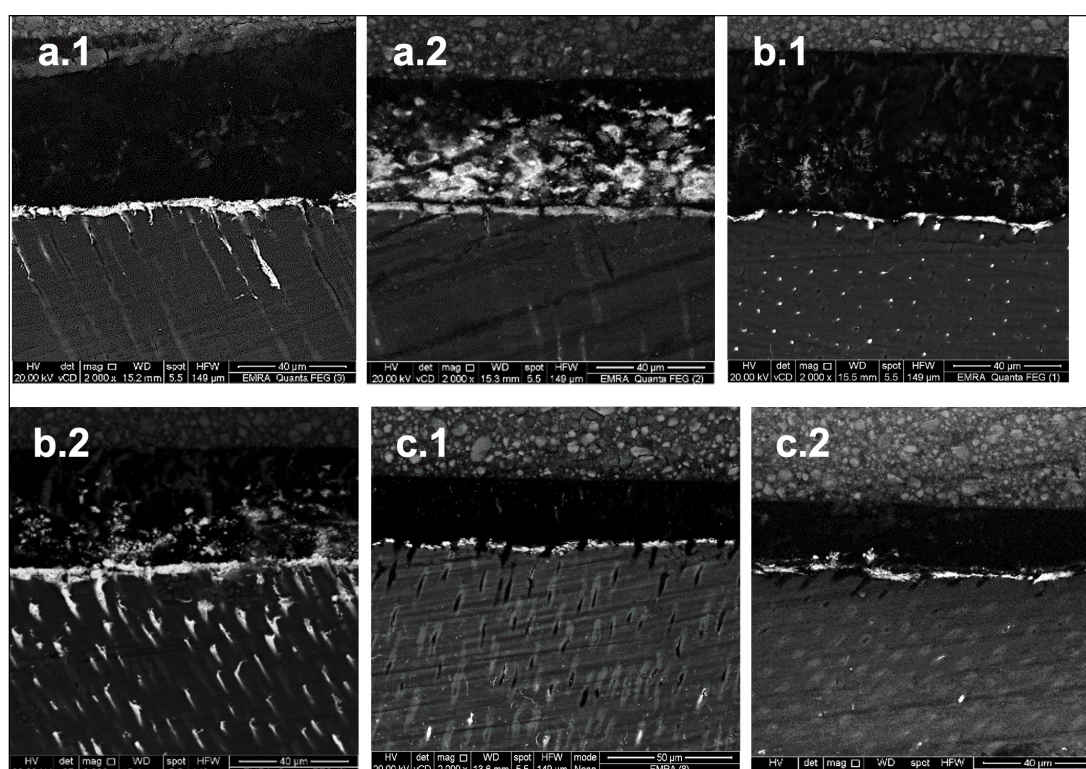


Figure 3. Back scattering representative scanning electron microscope (SEM) images of ammoniacal silver nitrate-stained resin-dentin interfaces demonstrating interfacial nano leakage for Adper Single Bond 2 groups. **a1**, Control (no thinning after 24 h of storage); **(a2)** Control (after 6 months of storage) **(b1)** Brush thinning after 24 h of storage; **(b2)** Brush thinning (after 6 months of storage) **(c1)** Air-thinning after 24 h of storage. Magnification: 2,000X; **(c2)** Air-thinning after 6 months of storage. Magnification: 2,000X.

Discussion

In the current study, two simplified adhesives were selected: Adper Single Bond 2 and Single Bond Universal. Adper Single Bond 2 is a 2-step etch- and-rinse adhesive, while Single Bond Universal is used as a 1-step self-etch adhesive. The separate etching step would indeed provide a stronger micromechanical attachment of etch-and-rinse adhesives to tooth structure than self-etch ones. However, Single Bond Universal is characterized by the presence of 10-Methacryloyloxydecyl dihydrogen phosphate (MDP), which is a monomer that bonds chemically to the hydroxyapatite of the tooth structure and thus was reported^{18,19} to resist hydrolysis and provide strong ionic bonds with calcium.

Other than this composition difference, both adhesives have nearly the same chemical composition; therefore, the application technique may be the major factor that affected the results of the current study. Adper Single Bond 2 and Single Bond Universal exhibited the highest im-

mediate bond strengths and lowest nanoleakage % when they were air-thinned prior to curing ($p < 0.001$). Gentle air-thinning may accelerate solvent evaporation from the adhesive and facilitate resin penetration into the dentinal surface, producing a better-quality hybrid layer, improving the seal of the interface, and raising the bond strength²⁰⁻²².

Although complete solvent evaporation is not clinically possible, it is highly important to reduce the solvent present within the adhesive during polymerization to avoid polymerization reaction retardation^{23,24}, which may affect both initial and long-term dentin bond strength. Several reports^{9,25,26} demonstrated conflicting results regarding the influence of air-thinning on dental adhesives' clinical performance. It was reported^{9,26,27} that resin-dentin bond strength increases with gentle air-thinning; however, strong air-thinning decreases the bond strength due to over-thinning of the adhesive layer, as well as increased oxygen inhibition, which reduces monomer conversion. Moreover, the number

of applied layers, as well as temperature and speed of air, may also affect the adhesive bonding performance significantly²⁵.

Air thinning allows for obtaining adhesive layers with uniform thickness. Forman et al²⁵ stated that decreasing the adhesive layer thickness by half would double the solvent evaporation rate. They stated that bond strength values are affected by many factors, such as phase separation, oxygen inhibition (depending on adhesive layer thickness), and defective adhesive penetration into the dentin. Component volatility is another factor that may affect the clinical performance of current adhesive systems²⁵.

The results of the current study showed a significant drop in the bond strength exhibited by Adper Single Bond 2 ($p < 0.001$), as well as a significant increase in nanoleakage with both adhesives when a brush was used for thinning ($p < 0.001$). Brush-thinning may also lead to over-thinning in some areas, which may cause reduced bond strength. Too thin adhesive layers may lead to suboptimal polymerization and low degree of monomer conversion⁹. Moreover, the adhesive layer acts as a stress relief layer that absorbs stresses

generated during composite polymerization, thus reducing stresses created at the bonded interface^{28,29}. Over-thinning of the adhesive layer in some areas may increase the stresses at the interface, weakening the bond strength. Furthermore, brush thinning may not allow proper solvent evaporation which may adversely affect the bond strength.

On the other hand, brush thinning of Single Bond Universal did not result in a significant drop in bond strength as compared to Adper Single Bond 2, which showed a significant drop. Since the chemical composition of both adhesives is very close, the main difference is probably in the mechanism of bonding. Etch and rinse mechanisms may result in deeper demineralization of the dentin, which may not be completely penetrated by the resin, leaving nanospaces at the base of the hybrid layer. Single Bond Universal is a mild self-etch adhesive (pH = 2.7)³⁰; thus, it provides more superficial dentin demineralization and simultaneous resin penetration to full depth. This may result in a thinner hybrid layer of better quality and may allow better solvent evaporation even with brush thinning, which may not happen with Adper Single Bond 2.

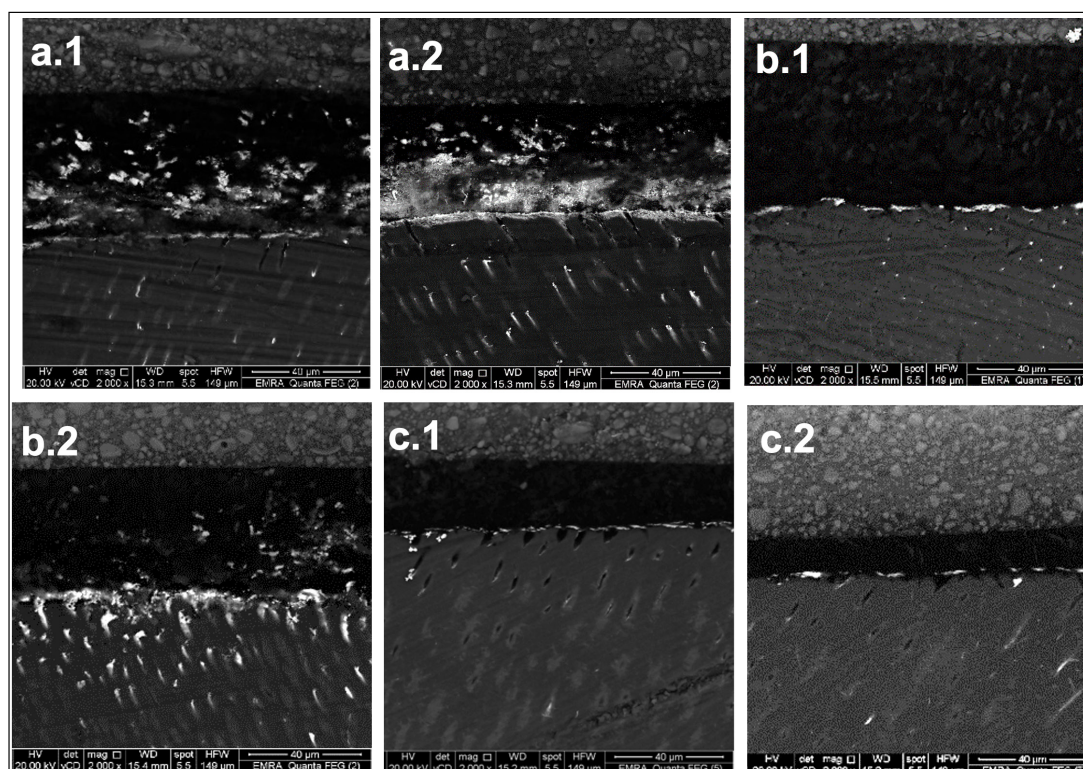


Figure 4. Back scattering representative scanning electron microscope (SEM) images of ammoniacal silver nitrate-stained resin-dentin interfaces demonstrating interfacial nano leakage for Single Bond Universal groups. **a1**, No thinning after 24 h of storage (Control); **a2**) No thinning after 6 months of storage (Control) **(b1)** Brush-thinning after 24 h of storage; **(b2)** Brush-thinning after 6 months of storage **(c1)** Air-thinning after 24 h of storage. Magnification: 2,000X; **(c2)** Air-thinning after 6 months of storage. Magnification: 2,000x.

When both adhesives were applied without thinning, a significant reduction in bond strength and a significant increase in nano leakage occurred ($p < 0.001$). This may be attributed to the presence of a too-thick adhesive layer, which negatively affected the bond strength as the adhesive layer is considered the weakest point in the resin-dentin interface. Furthermore, the increased adhesive thickness may hinder proper solvent evaporation. Also, proper curing of the adhesive before resin composite placement is critical. Too-thick adhesive layers may result in improper light curing, which can be further hindered by water and solvents present within the adhesive. This would significantly adversely affect the marginal seal and bond strength^{13,14}.

Despite the marked progress of dentin adhesives during the last decades, the poor stability of hybrid layers remains highly alarming^{1,2}. Numerous studies^{2,4,30,31} have confirmed this finding by demonstrating the rapid decrease of dentin bond strength over time^{2,30,31}, using both etch-and-rinse and self-etch adhesives⁴.

The results of the current study confirmed these findings, as there was a significant drop in the bond strength of both adhesives after 6 months of storage. However, the greatest drop in bond strength occurred in the specimens with no thinning (Adper Single Bond = 24%, Single Bond Universal = 30%), followed by the brush thinning specimens (Adper Single Bond 2 = 18%, Single Bond Universal = 22%). This may be due to the great amount of solvents and water that failed to evaporate during the bonding procedure as well as the presence of unreacted monomers, which may accelerate resin degradation and jeopardize the durability of the bonded interfaces^{23,24,32,33}. This also may explain why the least drop in bond strength was in the air-thinned groups.

On the other hand, within the air-thinned groups, the bond strength dropped significantly after 6 months for the Adper Single Bond 2 group (11%, p -value = 0.001), while in the Single Bond Universal, there was a 6% drop in the bond strength but with no significant difference between the 2 testing periods ($p = 0.076$). Acid-etching superficially demineralizes dentin and completely removes the hydroxyapatite crystals. Practically, the resin cannot completely infiltrate all exposed collagen, resulting in voids at the base or within the hybrid layer, which make the bonded interface susceptible to degradation over time^{1,9}. This is not the case for Single Bond Universal, which provides a better quality hybrid layer. Moreover, Single Bond Universal is a mild self-etch adhesive; thus, its formulation is less hydrophilic (3M ESPE. Sin-

gle Bond Universal Adhesive, Technical Product Profile, St. Paul, MN, USA: 3M Oral Care, 2016) and therefore less prone to undergo hydrolytic degradation, leading to improved durability.

The failure pattern often reflects the bond strength at the interface; as cohesive failure is often associated with higher bond strength, while adhesive failure is associated with weaker bonds⁹. This supports the predominance of adhesive failure in all 24 h groups that received no-thinning or brush-thinning, while the mixed type of failure predominated in the air-thinned groups; indicating a better bond strength in comparison to the groups with adhesive failure. Moreover, the mixed type of failure decreased in all aged experimental groups. Therefore, a shift in failure mode patterns from mixed to adhesive failure, indicating a weaker bond condition, confirmed some degree of degradation after aging in all adhesive groups.

Following many previous types of research^{2,17,30-33}, the results of this study showed a marked increase in nanoleakage within the bonded interfaces of all groups after 6 months of water storage; however, still, the greatest nanoleakage was found in the groups that received no-thinning followed by the groups that received brush-thinning, while least nanoleakage was found in air-thinned groups. Nanoleakage results followed the microtensile bond strength results of the current study.

According to the results of the current study, we reject the first null hypothesis as adhesive thinning had a significant effect on immediate bond strength and nanoleakage of both adhesives as compared to the control. In addition, we reject the second null hypothesis as six-month-water storage resulted in a significant decrease in bond strength and a significant increase in nanoleakage exhibited by all groups except for the air-thinned Single Bond Universal, where there was no significant difference in bond strength between both storage periods.

Limitations

Similar to other *in-vitro* studies, there are some limitations of the current investigation that include a lack of full simulation of the oral environment such as the presence of saliva, dentinal fluid, pulpal pressure, pH, temperature changes, and masticatory forces. Furthermore, specimens did not simulate typical dental restoration designs.

Conclusions

Within the limitations of the current study, it can be concluded that the air-thinning protocol

of simplified etch-and-rinse and self-etch dentin adhesives should be performed before light curing to provide a clinically acceptable bonded interface. Brush thinning of the self-etch adhesive provided initially a similar clinical performance to air-thinning. Furthermore, a lack of stability with varying degrees of the bonded interface was manifested regardless of the adhesive thinning protocols. Still, there is an urgent need for continuous development of new dentin adhesives that may result in more stable bonded interfaces with more clinically successful resin composite restorations.

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Ethics Approval

Ethical approval of protocol (#229-03-21) was obtained by the Research Ethics Committee, King Abdulaziz University, Saudi Arabia.

Informed Consent

The written informed consent was obtained from all patients from whom 60 non-carious human molars were extracted used according to the protocol (#229-03-21) approved by Research Ethics Committee, King Abdulaziz University, Saudi Arabia.

Conflict of Interest

The authors declare that they have no competing financial interests or personal relationships that could have appeared to influence the work reported in this manuscript.

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Data Availability

The data are all present in the manuscript, and raw data can be requested to the corresponding author for genuine reasons.

Authors' Contributions

MMA-N: experimental studies, data acquisition, statistical analysis, manuscript preparation. HMN: concepts, design, data analysis, manuscript review, guarantor. HMS: literature search, definition of intellectual content, manuscript editing.

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