

AIN SHAMS DENTAL JOURNAL

Official Publication of Ain Shams Dental School December2022 • Vol. 28

Effect of dentin moisture and bonding strategies on micro-tensile bond strength using two chemically different universal adhesives

Mohamed Abdel-Halim Hamouda¹, Mohamed Mahmoud Kandil², Mohamed Salah Nassif³

Aim: To study the relevance of dentin moisture state on the micro-tensile bond strength (μ TBS) and bonded interface morphology when using two universal adhesives with different compositions.

Materials and Methods: Sixty-third molars randomly allocated into 12 groups based on adhesive types (Prime & Bond Universal [P&BU], Dentsply Sirona, New York, USA and Single Bond Universal [SBU], 3M ESPE, Neuss, Germany), adhesives application approach (etch and rinse or self-etch), and the amount of moisture found on surface of dentin (dry, wet & visible moist dentin surface). Thirty-six molars were divided into beams of composite and dentin with adhesive interface for μ TBS (n=20) after restoration. Twenty-four molars were used to create specimens for scanning electron microscope evaluation of the bonded interface (n=2). For statistical analysis, three-way ANOVA was used, followed by a multiple comparison with Bonferroni adjustment.

Results: In etch and rinse, P&BU had higher μ TBS than SBU when the dentin surface was dry, SBU had higher μ TBS when the dentin surface was wet. There were no differences between both adhesives on the moist dentin surface. In selfetch, SBU had higher μ TBS than P&BU on the moist dentin surface, there were no significant differences between the two adhesives on dry and on wet dentin surfaces. For both adhesives, SEM images revealed better hybrid layer morphology and integrity with a greater number of resin tags and deeper penetration in etch and rinse approach than self-etch. Moist dentin surface developed better interface morphology than dry dentin surface, but wet dentin surface had the poorest hybrid layer quality with scarce resin tags in both adhesives.

Conclusion: When dentin was moist or dry, universal adhesive systems demonstrated good adhesive performance. The response in wet dentin, on the other hand, is material composition dependent. Universal adhesives are better utilized in etch and rinse approach than the self-etch approach.

Keywords: Universal Adhesives, micro-tensile bond strength, SEM, hybrid layer, wet-bonding.

- 1. Master of Biomaterials Faculty of dentistry, Ain- Shams University, Cairo, Egypt.
- 2. Lecturer of Biomaterials, Department of Biomaterials, Faculty of Dentistry, Ain Shams University, Cairo, Egypt.
- 3. Professor of Biomaterials, Department of Biomaterials, Faculty of Dentistry, Ain Shams University, Cairo, Egypt.

Corresponding author: Mohamed Abdel-Halim Hamouda, email: mohamedabdelhalim89@live.com

Introduction

Dentistry industry has long sought to high-strength and long-lasting develop connections between restorative materials and tooth structures ¹. Adhesive systems are used to increase composite resin attachment to the dental structure, and they should behave similarly on both dentin and enamel². Dental bonding to enamel is regarded as long-lasting and steady clinical technique ^{3, 4}. Attempts to bind to dentin in the same way as enamel have been investigated ⁴. The dentin substrate is different from enamel, as it contains more organic materials, more fluid in its tubules, the smear layer, and intrinsic moisture ^{2,5}.

Because of difficulty to achieve optimal surface wetness in a variety of situations, the moisture condition of the dentin is regarded a significant aspect in the adhesion process ⁵. A dry dentin surface may induce collagen fibrils to collapse, reducing adhesive penetration, whereas a wet dentin surface may produce adhesive dilution 2 . A moist dentin surface is assumed to be the best condition when bonding to dentin³. The primary purpose of bonding restorations is to reach a close attachment between the dental tissues and restorative materials ⁶. To allow structural interaction, the liquid adhesive must soak the solid adherent for long-term attachments ⁷. Monomers with both hydrophilic and hydrophobic groups are used in adhesive systems. The hydrophilic group improves wettability for dental tissues, whereas the latter allows polymerization and reaction with restorative materials⁸.

Etch-and-rinse (3- or 2-step) adhesives, which are characterized by using inorganic acids (typically phosphoric acid) preparing the dental tissues for priming and bonding. In conjunction with the bonding agent, self- etch adhesives contain monomers that promote adhesion in self-etch primer (2or 1-step)⁹. Dentin bonding is problematic as the proper wetness level must be

collagen-matrix maintained to prevent allowing adequate collapse adhesive diffusion into demineralized substrate^{10, 11}. Acid etching could be performed as a discrete stage (etch and rinse approach) ¹⁰ or by using acidic monomers (self-etch functional materials) ¹². The etch and rinse technique involves completely removing the superficial hydroxyapatite and smear layer 5-9, then infiltrating sticky monomers into the microporosities to form the "Hybrid Layer" ¹³.

The SE method, on the other hand, renders the smear layer porous without completely eradicating it ¹⁴. Adhesive monomers are used to demineralize the underlying dentin by partially dissolving the smear layer ¹⁰.

The most recent of these adhesives are the Universal adhesives also known as (Multi-mode adhesives), which offer the option of choosing between etch and rinse, self-etch, or selective enamel etching technique, combining the etch and rinse and self-etch approaches on enamel and dentin structure ^{15, 16}. Furthermore, these adhesives allow the bonding of different restorative materials ^{15, 17, 18}.

These are simplified adhesives that typically contain all of the components of bonding in one single bottle. However, these adhesives were linked to inferior in-vivo restoration durability and decrease of the invitro bond strength outcome ^{19, 20}. These findings are most likely the outcome of the complicated construction of those adhesives and their elevated amount of solvent, that might decrease the entire volatilization of the solvent, leading to inferior polymerization of the adhesive ²¹. Furthermore, the possibility of various degrees of moisture in the tooth cavity following etch, rinse, and drving actions affects the adhesion procedure, which is prejudiced by the components of the adhesive system ²².

A perfectly moist dentin surface should be reached in order to keep the

collagen fibers expanding, so as to be infiltrated with the monomers of the resin, and to improve the procedure of adhesion. However, when using dental adhesives, the actions of rinsing and drying are considered the most important aspects; additionally, deeper tooth cavities with more complexity may have different grades of wetness, where dry and extremely wet sections may occur on the same surface. Therefore, reaching the ideal moist dentin surface is problematic.

According to the above, this study was done for assessing the effects related to dentin surface hydration levels and bonding techniques on micro-tensile bond strength of resin composite to dentin when two Universal adhesive systems with different compositions were used in the bonding process.

Materials and methods

Universal Two adhesives were compared in etch and rinse and self-etch modes in dentin with varying moisture levels (dry, wet, visible moist). Prime & Bond Universal (P&BU, DENTSPLY Sirona) and Single Bond Universal (SBU,3M ESPE). Table (1) describes the manufacturers, compositions and lot numbers. In this study, 264 specimens were used: 240 for microtensile bond strength test and 24 for scanning electron microscopic assessment. Samples were made from sixty recently extracted free from caries wisdom molars (thirty-six teeth for micro-tensile bond strength testing with 3 molars prepared for each sub-group and twenty-four for scanning electron microscopic assessment). Molars washed and cleaned from debris before being kept in distilled water for one month. These molars were then divided randomly into twelve groups according to adhesive type (Single Bond Universal or Prime & Bond Universal), as well as etching approach (etch and rinse or self-etch) and to moisture condition of the dentin surface (dry surface, wet surface and

visible moist surface) where n=20 for microtensile bond strength test and n=2 for SEM evaluation.

Table 1: Materials that were	used in the research, including
their description, composition,	, and lot number

	Brand name and	Description	Composition	Lot no.
	manufacturer			
	Single Bond Universal	Mild Universal	MonomerMDP, HEMA,	00131A
	(3M ESPE, Neuss,	adhesive	Dimethacrylate resins	
	Germany) N.B.	(pH=2.7)	Vitrebond [™]	
	Scotchbond TM		Copolymer, Fillers,	
	Universal and Single		water, etahnol, Silane,	
	Bond Universal are the		initiators	
	same adhesive with			
	different names.			
	Prime&Bond	Mild Universal	MDP, PENTA,	200300
	universal TM Universal	adhesive (pH	Phosphoric acid	
	Adhesive	value > 2.5)	modified acrylate resin	
-	(Dentsply Sirona,New		Dentsply Sirona Active-	
	york,USA)		Guard TM technology,	
A	T		Initiator, Stabilizer,	
			Isopropanol, Water	
	Meta Etchant	Etchant delivery	37% Phosphoric Acid	MET2010061
	(METABIOMED	system	Semi Gel	
_	CO.LTD., Cheongju-si,			
	Chungcheongbuk-do,			
	Korea)			
	Polofil NHT flow	Flowable Nano-	Dimethacrylates	2047738
	(Voco, Cuxhaven,	Hybrid	(including BisGMA,	
	Germany)	Composite	UDMA, TEGDMA),	
		Shade(A2)	Fillers, Pigments,	
1			Catalysts and Stabilizers	

Specimen's grouping was performed as follows: Group A: (Single Bond Universal), Group B: (Prime & Bond Universal) each group was divided into subgroups according to the approach of application (etch and rinse 'i' or self-etch 'ii') then further sub-divided into three groups according to dentin surface moisture (dry 1, wet 2 and visible moist 3) so that the groups are:

A-i-1: Single Bond Universal etch and rinse for dry surface., A-i-2: Single Bond Universal etch and rinse for wet surface., A-i -3: Single Bond Universal etch and rinse for visible moist surface,

A-ii-1: Single Bond Universal self-etch for dry surface, A-ii-2: Single Bond Universal self-etch for wet surface, and A-ii-3: Single Bond Universal self-etch for visible moist surface. While for P&BU groups are as follow: B-i-1: Prime & Bond Universal etch and rinse for dry surface, B-i-2: Prime & Bond Universal etch and rinse for wet surface, B-i-3: Prime & Bond Universal etch and rinse for visible moist surface, B- ii- 1: Prime & Bond Universal self-etch for dry surface, B-ii-2: Prime & Bond Universal selfetch for wet surface, and B-ii-3: Prime &

3

Bond Universal self-etch for visible moist surface.

Micro-tensile bond strength test Specimen's preparation

Molars were placed inside acrylic blocks two millimeters under the cemento-enamel junction. By using a slow speed diamond saw, the enamel was split horizontally 1 mm beneath the dentin-enamel junction (DEJ) until dentin was exposed (Isomet 4000, Buehler Ltd., Lake Bluff, IL, USA). The uncovered surfaces of dentin were refined for 60 seconds on wet 600-grit SiC paper to produce standardized smear layer ²³. Dentin surface was etched for fifteen seconds with a thirty-seven percent phosphoric acid solution (meta-etch) and rinsed away with waterspray. The dry surface groups had the dentin surface dried by using compressed air free from oil for 20 seconds at a distance of 5 cm away from the dentin surface, while wet groups had the dentin substrate kept wet without surface drying. The moist dentin surface groups were produced by lightly drying the dentin surface with paper points for 5 seconds until a glossy surface was obtained ²⁴.Then the prepared surface was treated by the intended adhesives in strict agreement with the producer's commands, as shown in table (2). Dentin surface was not pre-etched in self-etch mode.

Following bonding, each tooth received a composite restoration (Polofil NHT flow, Voco, Cuxhaven, Germany) in three layers, each layer is about 2mm. The LED light cure device was used to irradiate each layer for twenty seconds (Elipar S10 free light, 3M ESPE, USA).

The restored teeth are soaked in thirty-seven degrees Celsius distilled water for twentyfour hours, then sectioned with slow-speed saw (diamond) longitudinally in the buccolingually and mesio-distally to produce beams with cross sectional area around one mm2 as determined by a digital-caliper.

 Table 2: Bonding technique for the two adhesives in the two

 modes (etch & rinse, and self-etch)

	SBU	P&BU
Self-etch	 A micro-brush was used to apply the adhesive to the exposed dentin surface, which was rubbed for 20 seconds. For 5 seconds, mild stream of air was passed on the adhesive until it stopped moving, indicating complete solvent evaporation. The adhesive was cured for 10 seconds using LED light curing system (Elipar S10 free light, 3M ESPE, USA) with output intensity 1200 mW/cm². 	I. New Applicator Tip was used. Z. For 20 seconds, the adhesive was lightly agitated. G. Clean and dry air from air- water syringe was used to distribute the adhesive and remove the solvent. 4.A moderate air flow was applied to each surface for at least 5 seconds, resulting in a glossy and homogenous coating. Using an excessively strong air flow will result in dry areas and adhesive splashing. 5. The adhesive was cured for 10 seconds using LED light curing system (Elipar S10 free light, 3M ESPE, USA) with output intensity 1200
Etch and rinse	 For 15 seconds, the etchant (Meta Etchant) was used to etch the dentin surface. The etchant was washed for 15 seconds using flowing water. The adhesive was then applied in the same means as it was for the self-etch mode. 	 For 15 seconds, the etchant (Meta Etchant) was used to etch the dentin surface. The etchant was washed for 15 seconds using flowing water. The adhesive was then applied in the same means as it was for the self-etch mode.

Each beam was made of composite and dentin, with an adhesive interface. Then, each beam was attached using cyanoacrylate glue to a stainless steel notched Geraldeli's jig ²⁵ and tested under tension using universal test machine (Instron, MA, USA) at a rate of 0.5 mm/min crosshead speed till fracture. The broken bonded area of specimens and load were recorded. Micro-tensile bond strength was calculated with (Bluehill Lite software, Instron, MA, USA) in MPa.

Evaluation of the tooth-restoration interface by Scanning electron microscope:

Specimens' preparation

In twenty-four recently extracted third molars free from caries (two for each experimental condition as described for micro-tensile bond strength test), occlusal surfaces were removed until dentin was exposed. The adhesive and resin composite were then applied to the exposed dentin in the same manner defined previously in specimen preparation for micro-tensile bond strength test.

Then teeth were sectioned into two halves bucco-lingually using a diamond disc under copious air-water coolant spray. The sections were flattened and smoothed using silicon carbide papers with sequential grit of 600, 800,1000,1200,1500,2000 and 2500.The specimens were acid etched for five seconds with 37 percent phosphoric acid gel and rinsed for another ten seconds before being immersed in 3 percent NaOCl for ten seconds and washed under running water for 1 minute. This was done to clean up any debris and to achieve a clean, glossy surface on the bonded interface. No gold sputtering was done as environmental scanning was used. The adhesive-dentin interface was assessed by scanning electron microscope (Quanta 3D 200 I).

Results

I. <u>Micro-tensile bond strength results:</u>

Different adhesives exhibited minimal effects on micro-tensile bond strength (p = 0.110), according to a three-way ANOVA. In contrast, Technique (Etch and rinse vs. Selfetch), and water content (Dry vs. wet, moist) and relations between all the variables showed a significant outcome on the micro-tensile bond strength (p < 0.001).

In case of etch and rinse technique. In dry condition Prime & Bond universal had higher mean micro-tensile bond strength than Single Bond universal, but in wet condition the micro-tensile bond strength for Single Bond universal was higher than Prime & Bond universal. In moist dentin surface there was no difference in micro-tensile bond strength between the two adhesives as shown in table (3)

On the other hand, in self-etch technique, Single Bond universal had higher micro-tensile strength than Prime & Bond universal when the dentin surface was moist. In dry and wet dentin surfaces there was no difference between both adhesives in micro-tensile bond strength as shown in table (3).

Table	3:	Mean	and	standard	deviation	of	micro-tensile	bond
strength for different tested adhesives								

		Single Bon	Single Bond Universal		Prime & Bond Universal	
		Mean	SD	Mean	SD	1
Etch and	Dry	21.3 ^{b, A}	5.2	29.3 ^{a, A}	6.9	0.0001*
rinse	Wet	16.3 ^{a, B}	4.0	10.3 ^{b, B}	2.5	0.001*
	Moist	17.1 ^{a, AB}	4.1	13.8 ^{a, B}	3.4	0.064 NS
Self-etch	Dry	18.6 ^{a, A}	4.6	16.8 ^{a, A}	3.0	0.308 NS
	Wet	10.0 ^{a, B}	1.6	11.7 ^{a. B}	2.7	0.353 NS
	Moist	18.3 ^{a, A}	3.6	12.8 ^{b. AB}	3.1	0.002*

*=Significant. NS= non-significant. Different small letters within each row indicates significant difference, Different capital letters in each column indicates significant difference for each mode of bonding techniques (etch and rinse & self-etch).

II. <u>Scanning Electron microscope (SEM)</u> results evaluation:

Scanning electron microscopic images (2000X) were done for assessment of the tooth-restoration interface for different experimental groups.

a. <u>Etch and rinse group:</u>

• The Scanning electron microscopic photomicrographs shows that In-case of dry dentin surface there is collapsed collagen with few penetrations of resin in dentinal tubule. Single Bond Universal had good integrity at the adhesive- dentin interface with intact and thick hybrid layer formation but not uniform (red arrows) but Prime &Bond Universal showed thin hybrid layer and more uniform with slightly more resin tags as shown in figure (1).



Figure (1): SEM image 2000X showing tooth /restoration interface for SBU and P&BU in etch and rinse approach (dry dentin surface) (A) Single Bond Universal, (B) Prime &Bond Universal (D) dentin, (C) composite, red arrows marking the hybrid layer, White arrows marking the resin tags.

• Scanning electron microscopic photomicrographs reveled when the dentin surface was wet, both adhesives showed that there was no enough penetration of resin in dentinal tubules due to excess water with less uniform hybrid layer and presence of defects filled with water between dentin and adhesive as shown in figure (2).



Figure (2): SEM image 2000X showing tooth /restoration interface for SBU, P&BU in etch and rinse approach (wet dentin surface) (A) Single Bond Universal, (B) Prime &Bond Universal, (D) dentin, (C) composite, red arrows marking the hybrid layer, White arrows marking the resin tags.

- In case of moist dentin surface there is abundant amount of resin tags in both adhesives with uniform and thin hybrid layer with average thickness for the two adhesives as shown in figure (3).
- <u>Self-etch group</u>
- In case of self-etch mode, a less distinct hybrid layer is seen with few and short resin tags. In dry dentin surface Single Bond Universal showed thicker and less homogenous hybrid layer with deeper resin tags, while Prime &Bond Universal showed thinner and more uniform hybrid layer with shallower depth of resin tags as shown in figure (4).



Figure (3): SEM image 2000X showing tooth /restoration interface for SBU and P&BU in etch and rinse approach (moist dentin surface) (A) Single Bond Universal, (B) Prime &Bond Universal (D) dentin, (C) composite, red arrows marking the hybrid layer, White arrows marking the resin tags.



Figure (4): SEM image 2000X showing tooth /restoration interface for SBU and P&BU in self-etch approach (dry dentin surface) (A) Single Bond Universal, (B) Prime &Bond Universal (D) dentin, (C) composite, red arrows marking the hybrid layer, White arrows marking the resin tags.

• In wet dentin surface Single Bond Universal showed less uniform hybrid layer with average thickness and bubbles of water in the hybrid layer with average number of shallow resin tags. While Prime &Bond Universal showed more uniform and thinner hybrid layer with no water bubbles or voids inside the hybrid layer as shown in figure (5).



Figure (5): SEM image 2000X showing tooth /restoration interface for SBU and P&BU in self-etch approach (wet dentin surface) (A) Single Bond Universal, (B) Prime &Bond Universal (D) dentin, (C) composite, red arrows marking the hybrid layer, White arrows marking the resin tags, green arrows marking water bubbles.

• In moist dentin surface, Single Bond Universal showed uniform hybrid layer with uniform thickness with abundant resin tags that extend deep in dentin. In Prime &Bond Universal the hybrid layer is uniform and thin with average amount of resin tags that also extend deep in dentin as shown in figure (6).

AST

Discussion

latest generation of the modern adhesive systems are Universal adhesives, founded on the all-in-one perception. General practitioner can adjust these group of bonding agents to the clinical scenario by using them in both techniques either etch and rinse or self-etch , according to their assessment of what seems to be most suitable in the clinical circumstance ²⁶. Dentin bonding still has issues with determining the proper humidity level for improved hybridization, pulp protection, and cavity margin sealing^{27, 28}.



Figure (6): SEM image 2000X showing tooth /restoration interface for SBU and P&BU in self-etch approach (moist dentin surface) (A) Single Bond Universal, (B) Prime &Bond Universal (D) dentin, (C) composite, Red arrows marking the hybrid layer, White arrows marking the resin tags.

Investigating the morphological characteristics of dentin-adhesive interface had been used to determine the patterns of hybridization given by numerous adhesives with a variety of dentin surface circumstances ^{22,23,28}. Hence, in the present study the microtensile bond strength of two different bonding adhesives (Single Bond Universal and Prime & Bond universal) was assessed applying both etch and rinse and self-etch techniques when dentin surface was dry, wet and visibly moist. Studies employing universal adhesives and various dentin surface moistures revealed mixed outcomes with few variables ^{29, 30, 31, 32}. Hence, that is the reason why we utilized dry, wet and moist substrate surfaces.

According to the present study, for dry dentin surface, Prime & Bond Universal demonstrated higher micro-tensile bond strength (29.1 MPa) than Single Bond Universal (21.3 MPa) in the etch and rinse technique. This could be because the isopropanol solvent found in the Prime & Bond Universal solution can aid in the regaining the expansion of the collapsed network of collagen, and the resin mixture which has low viscosity can effectively infuse to the inter-fibrillar spaces, creating a suitable hybrid Layer while used on the dried demineralized dentin surfaces. Two-steps etch and rinse adhesive containing tertbutanol solvent previously showed the same performance ³¹. This is in agreement with Kumagai et al ³² where Prime and Bond Universal had higher micro tensile bond strength (91.7 MPa) than Single Bond Universal (77.3 MPa) in etch and rinse condition when dentin surface was dry.

However, in wet dentin surface by using the same bonding technique (etch and rinse), Single Bond Universal demonstrated greater micro-tensile bond strength (16.3 MPa) than Prime & Bond Universal (10.3) MPa). This might be due to the fact that ethanol and water which are solvents in many adhesive systems such as SBU, their capability to establish hydrogen bonds with the collagen fibers peptides impacts their ability of re-expanding, keeping the demineralized dentin extended throughout monomer intrusion ³³. Because ethanol and water in SBU have solubility parameters that are similar, they can be mixed easily to produce a solution. This solution had a similar solubility parameter to dentinal fluid found in dentinal tubules, which improves the adhesive's bonding performance ³⁴.

It's unclear why Prime & Bond Universal had lowered micro-tensile bond strength in wet dentin surface using the etch and rinse approach. This could be owing to the fact that the water content of the P&BU is around 20%, compared to 15% in the SBU (10–15%). This higher water content in P&BU is linked to a increased water content on the dentin surface (wet), this might explain why P&BU has a lower binding strength value than SBU ^{30, 35}. The previous results agree with the study done by Münchow et al ³⁶ which showed that Prime & Bond 2.1 having the same amount of water content in its composition as P&BU had decreased micro-tensile bond strength when used on wet dentin surface.

The findings contradict the findings of Figueredo de Siqueira et al ³⁰ which revealed that the micro-tensile bond strength of Single Bond Universal was lower than that of Prime & Bond Universal using etch and rinse technique when the dentin surface was over saturated with water.

There was no difference in microtensile bond strength between the two adhesives when the dentin surface was visibly moist with the application approach of etch and rinse. That might be due to the fact that moist dentin is believed to have the optimal water content after etching. Over drying can cause the collagen network to collapse, while over wetting can dilute the adhesive ³⁷. This is in agreement with Tsujimoto et al ²⁹ who found that there is no difference in bond strength between Prime & Bond Universal (19.1 MPa) and Single Bond Universal (20.2 MPa) when dentin surface was moist utilizing etch and rinse technique.

In self-etch technique micro-tensile bond strength showed no significant difference between the two adhesives when dentin surface was dry and when dentin surface was wet. That might be due to the fact that both adhesives are mild adhesives. The pH of P&BU is around (2.5) and SBU is (2.7), their ability to penetrate the smear layer is the same as in the case of self-etch, which is greatly reduced in wet conditions due to dilution of the adhesives ³². This is in agreement with Cengiz and Ünal³⁸ that stated that there was no remarkable difference between the two adhesives in micro-tensile bond strength when used in self-etch approach with two different self-adhesive composites.

Micro-tensile bond strength regarding Single Bond Universal (18.3 MPa) was better than Prime & Bond Universal

(12.8 MPa) when the dentin surface was visibly moist by using self-etch technique. This might be due to the use of ethanol as a solvent in Single Bond Universal and isopropanol as a solvent in Prime & Bond Universal. According to several studies, adhesive systems containing ethanol had decreased sensitivity to dentin moisture levels ^{39, 40}, however, it may be true only when dentin surface is moist. Isopropanol is also less hydrophilic and has a slightly higher viscosity than ethanol ⁴¹. The previous findings are in agreement with Figueredo de Siqueira et al ³⁰ in which Single Bond Universal had higher micro-tensile strength than Prime & Bond Universal in the same conditions.

In terms of the scanning electric microscope findings for evaluating the generated interface between the adhesives and dentin, etching of dentin in the etch and rinse approach resulted in the smear layer removal, increase in depth of infiltration of both adhesives into dentin, creation of lengthy resin tags and thicker hybrid layers, and improved morphology of dentin-resin interface 42 as previously shown in figures (1,2 &3).

Although morphological characteristics of the interface was improved after acid etching, there is no connection between the micro-tensile bond strength and hybrid layer quality ⁴³. When compared to etch and rinse groups, the hybrid layer of the self-etch groups was relatively thin with very few resin tags for both adhesives, which could be because smear plugs blocked the majority of the dentinal tubules ⁴⁴ as shown in figures (4,5 &6).

Furthermore, both adhesives have a mild pH (2.5,2.7 for P&BU and SBU, respectively), which reduces the capacity of monomer to permeate dentinal tubules and create deep, dense, and well-defined resin tags ³². These discoveries are in approval with findings of study done by Takamizawa

et al.⁴⁵ which showed that the hybrid layer for five universal adhesive was thicker and more uniform with resin tags longer and deeper in etch and rinse technique than self-etch technique. Cengiz and Ünal ³⁸ SEM investigations, showed deeper and more consistent resin tags with thicker hybrid layer in the etch and rinse groups than self-etch groups for four universal adhesives tested with two different types of composites.

The thickness, quality, and regularity of the hybrid layer, as well as number of tags and the depth of resin penetration in the dentinal tubules, are all affected by the moisture condition of the dentin. Dry dentin surface had moderate hybrid layer integrity, with an average amount of resin tags and shallow penetration in dentinal tubules, according to SEM data shown in figures (1&4). The wet dentin surface had a scarce resin-tags with extremely shallow dentin penetration, and the hybrid layer was full of voids and bubbles and not uniform shown in figures (2&5). The most uniform, greatest quality, and thickness of hybrid layer was found on the moist dentin surface, with abundant resin-tags that are thick with deep extension into the dentin for both adhesives in the two techniques etch and rinse and selfetch shown in figures (3&6).

These outcomes could be attributed to the fact that over-drying of the dentin surface collapsed the fibrils of the collagen matrix, limiting penetration of monomers inside the dentinal collagen network ³⁶. In wet dentin surface, it's possible that too much water diluted the adhesive ingredient, making the hybrid layer porous³⁶. Excess water may also limit removal of excess water, preventing entire monomer entry into demineralized dentin or possibly adhesive polymerization is hampered ^{22,46}. Moist dentin surface avoids collagen fibrils collapse and offers a suitable surface for the hydrophilic resins to easily penetrate into the dentinal tubules ⁴⁷. This is in approval with SEM results of Perdigao et

al ⁴⁸ where moist dentin surface showed better integrity of hybrid layer and deeper resin tags than dry and wet dentin surfaces.

The adhesive composition is another factor that influences the SEM results. In the etch and rinse mode, P&BU had a more uniform and thinner hybrid layer with more resin tags than SBU on the dry dentin surface shown in figure (1). This could be because the P&BU adhesive solution contains isopropanol, which can aid the collagen system that was collapsed to re-expand, allowing resin mix which has low viscosity to penetrate the interfibrillar spaces, creating an acceptable hybrid layer when used on dried demineralized dentin surface ³¹. While the presence of HEMA in SBU composition replaces water in dentinal tubules, which is present in minimal amounts in the dry state, limiting rehydration of collapsed collagen and reducing resin infiltration ³². These SEM results are in approval with Kumagai et al³² SEM results which showed that P&BU had more uniform hybrid layer with deeper resin infiltration than SBU in dry dentin surface.

Conclusion

According to the study's findings, the best condition for bonding both adhesives is a moist dentin surface, followed by a dry dentin surface. Avoid dentin surfaces that are wet. The two adhesives showed better performance in the etch and rinse approach than self-etch approach.

Conflict of interest

The authors do not have any financial interest in the companies whose materials are included in this article.

References

1. Stangel I, Ellis TH & Sacher E (2007) Adhesion to tooth structure mediated by contemporary bonding systems Dental Clinics of North America 51(3) 677-694.

2. Susin Ah, Vasconcallos Wa, Cury Saad Jr & Oliveira Junior Ob (2007) Tensile Bond Strength of

Self EtchingVersus Total Etching Adhesive Systems under Different Dentinal Substrate Conditions Brazilian Oral Research 21(1) 81-86.

3. Ritter AV, Heymann HO, Swift EJ, Perdigao J & Rosa BT (2000) Effects of different rewetting techniques on dentin shear bond strengths Journal of Esthetic Dentistry 12(2) 85-96

4. Frankenberger R, Perdigao J, Rosa BT & Lopes M (2001) _No-bottle' vs _multi, bottle' dentin adhesives—A microtensile bond strength and morphological study, and Dental Materials 17(5) 373-380.

5. Perdigao J New developments in dental adhesion Dental Clinics, and North America 51(2)2007 333-357.

6. Baier RE. Principles of adhesion. Oper Dent. 1992; Suppl.5:1–9

7. Eick JD, Cobb CM, Chapell RP, Spencer P, Robinson SJ. Thedentinal surface: its influence on dentinal adhesion part I.Quintessence Int. 1991;2:967–77.3

8. Van Landuyt KL, Snauwaert J, De munck J, Peumans M, Yoshida Y, Poitevin A, et al. Systematic review of thechemical composition of contemporary dental adhesives.Biomaterials. 2007;28:3757–85.

9. Van Meerbeek B, De MunckJ, Yoshida Y, et al. Buonocore memorial lecture. Adhesion to enamel and dentin: current status and future challenges. Oper Dent 2003;28: 215–235

10. Pashley DH, Tay FR, Breschi L, Tjaderhane L, Carvalho RM, Carrilho M, et al. State of the art etchand-rinse adhesives. Dental materials: official publication of the Academy of Dental Materials. 2011;27(1):1-16.

11. Münchow EA, de Barros GD, Alves LS, Valente LL, Cava SS, Piva E, Ogliari FA (2014) Effect of elastomeric monomers as polymeric matrix of experimental adhesive systems: degree of conversion and bond strength

12. Van Meerbeek B, Yoshihara K, Yoshida Y, Mine A, De Munck J, Van Landuyt KL (2011) State of the art of self-etch adhesives. Dent Mater 27:17–28

13. Sanket Nagarkar, Nicole Theis-Mahon, Jorge Perdigão 2019 in Wiley Online Library . Universal dental adhesives: Current status, laboratory testing, and clinical performance

14. Sofan E, Sofan A, Palaia G, Tenore G, Romeo U, Migliau G. Classification review of dental adhesive systems: from the IV generation to the universal type. Annali di stomatologia. 2017;8(1):1.

15. Hanabusa M, Mine A, Kuboki T, Momoi Y, Van Ende A, Van Meerbeek B, De Munck J. Bonding effectiveness of a new 'multi-mode' adhesive to enamel and dentine. J Dent 2012;40(6): 475–484

16. Antoniazzi BF, Nicoloso GF, Lenzi TL, Soares FZ, Rocha Rde O. Selective acid etching improves the bond strength of universal adhesive to sound and demineralized enamel of primary teeth. J Adhes Dent 2016;18(4):311–316.

17. Kim JH, Chae SY, Lee Y, Han GJ, Cho BH. Effects of multipurpose, universal adhesives on resin bonding to zirconia ceramic. Oper Dent 2015;40(1):55–62.

18. Van Meerbeek B, Frankenberger R. Editorial: What's next after "universal" adhesives, "bioactive" adhesives? J Adhes Dent 2017; 19(6):459–460.

19. De Munck J, Van Landuyt K, Peumans M, Poitevin A, Lambrechts P, Braem M, Van Meerbeek B (2005) A critical review of the durability of adhesion to tooth tissue: methods and results. J Dent Res 84:118–132

20. Tuncer D, Yazici AR, Ozgunaltay G, Dayangac B (2013) Clinical evaluation of different adhesives used in the restoration of non-carious cervical lesions: 24-month results. Aust Dent J 58:94–100

21. 3 M ESPE. ScotchbondTM Universal Adhesive. Date access: November 25, 2014. Available from: http://

multimedia.3m.com/mws/media/754753O/scotchbon d-universal-

adhesive.pdf?fn=scotchbond_uni_brochure.pdf

22. Tay FR, Gwinnett AJ, Wei SH. The overwet phenomenon: a transmission electron microscopic study of surface moisture in the acid-conditioned, resin-dentin interface. Am J Dent. 1996; 9: 161-6.

23. Melissa Al, Patrícia M, Daphne Câ, Clovis P, Carlos RT, Bond Strength of Adhesive Systems with Different Solvents to Dry and Wet Dentin. The Journal of Contemporary Dental Practice, 2013;14(1):9-13

24. Kanca J, 3rd. Improving bond strength through acid etching of dentin and bonding to wet dentin surfaces. J Am Dent Assoc. 1992; 123: 35-43.

25. Perdigão J, Geraldeli S, Carmo AR, Dutra, HR. In vivo influence of residual moisture on microtensile bond strengths on one-bottle adhesives. J Esthet Restor Dent. 2002;14:31-8.

26. Cristina Gré, Mauro de A, Sylvio Mo J. Microtensile bond strength of a universal adhesive to deep dentin. Braz Dent Sci 2016 DOI: 10.14295/bds.2016.v19i2.1259

27. De Goes MF, Pachane GCF, Garcia-Godoy F. Resin bond strength with different methods to remove excess water from the dentin. Am J Dent 1997;10(6):298-301.

28. Nakabayashi N, Sami Y. Bonding to intact dentin. J Dent Res 1996;75(9):1706-15.

29. Tsujimoto A, Shimatani Y, Nojiri K, Barkmeier WW, Markham MD, Takamizawa T, et al. Influence

of surface wetness on bonding effectiveness of universal adhesives in etch-and-rinse mode. Eur J Oral Sci 2019127:162–9.

30. Siqueira, F., Pinto, T.F., Carvalho, E.M., Bauer, J., Gonçalves, L., Szesz, A., Reis, A., Cardenas, A.M., & Loguercio, A. Influence of dentinal moisture on the properties of universal adhesives. International Journal of Adhesion and Adhesives, 2020 101, 102633

31. Machado LS, Oliveira FG, Guinossi TA, et al. Effects of dentin dry/- moist condition on the immediate adhesive performance of different solvent-based etch-and-rinse adhesive systems. Int J Adhes Adhes. 2013;43:1-6.

32. Kumagai RY, Hirata R, Pereira PNR, Reis AF. Moist vs over-dried etched dentin: FE-SEM/TEM and bond strength evaluation of resin-dentin interfaces produced by universal adhesives. J Esthet Restor Dent. 20191–8

33. Manso AP, Marquezini L Jr, Silva SM, Pashley DH, Tay FR, Carvalho RM. Stability of wet versus dry bonding with different solvent-based adhesives. Dent Mat 2008 Apr;24(4):476-82.

34. Miller RG, Bowles CQ, Chappelow CC, Eick JD. Application of solubility parameter theory to dentinbonding systems and adhesive strength correlations. J Biomed Mater Res. 1998;41:237-243.

35. FGM. FGM produtos odontologicos. Tecnología APS (advanced polymerization system). 2018

36. Münchow, E., Valente, L.L., Bossardi, M., Priebe, T.C., Zanchi, C.H., & Piva, E. (2014). Influence of surface moisture condition on the bond strength to dentin of etch-and-rinse adhesive systems. Brazilian Journal of Oral Sciences, 13, 182-186

37. Ahmed MH, De Munck J, Van Landuyt K, Peumans M, Yoshihara K, Van Meerbeek B. Do universal adhesives benefit from an extra bonding layer? J Adhesive Dent 2019;21:117–32

38. Cengiz T, Ünal M. Comparison of microtensile bond strength and resin–dentin interfaces of two selfadhesive flowable composite resins by using different universal adhesives: Scanning electron microscope study. Microsc Res Tech. 2019;1–9

39. Finger WJ, Balkenhol M. Practitioner variability effects on dentin bonding with an acetone-based one-bottle adhesive. Journal of Adhesive Dentistry. 1999;1(4).

40. Reis A, Loguercio AD, Azevedo CL, de Carvalho RM, da Julio Singer M, Grande RH. Moisture spectrum of demineralized dentin for adhesive systems with different solvent bases. J Adhes Dent. 2003; 5: 183-92.

41. Dentsply Sirona, Prime and Bond Universal _Scientific Compendium_EN.pdf; 2018.

42. Wagner A, Wendler M, Petschelt A, Belli R, Lohbauer U. Bonding performance of universal adhesives in different etching modes. Journal of dentistry. 2014;42(7):800-7.

43. Priyadarshini B, Selvan S, Lu T, Xie H, Neo J, Fawzy A. Chlorhexidine nanocapsule drug delivery approach to the resin-dentin interface. Journal of dental research. 2016;95(9):1065-72.

44. Nagpal R, Manuja N, Tyagi SP, Singh UP. In vitro bonding effectiveness of self-etch adhesives with different application techniques: A microleakage and scanning electron microscopic study. Journal of conservative dentistry : JCD. 2011;14(3):258-63

45. Takamizawa T, Imai A, Hirokane E, et al. SEM observation of novel characteristic of the dentin bond interfaces of universal adhesives. Dent Mater. 2019;35(12):1791-1804

46. Spencer P, Wang Y. Adhesive phase separation at the dentin interface under wet bonding conditions. J Biomed Mater Res. 2002;62(3):447-56.

47. Kanca J, 3rd. Wet bonding: effect of drying time and distance. Am J Dent. 1996;9(6):273-6.

48. Perdigao J, Araujo E, Ramos RQ, Gomes G, Pizzolotto L. Adhesive dentistry: Current concepts and clinical considerations. J Esthet Restor Dent. 2021;33: 51–68.

Ain Shams Dental Journal