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Effect of Thermocycling, Degree of Conversion, and Cavity Configuration on the Bonding Effectiveness of All-in-One Adhesives

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Clinical Relevance

The poor results of some all-in-one dentin adhesives tested may indicate that clinicians should be cautious with selection of this class of materials for high-C-factor or deep-class II applications.

SUMMARY

The aim of this study was to compare five all-in-one bonding agents with respect to microleakage, microtensile bond strength (μTBS), degree of conversion (DC) and the impact of cavity configuration. The materials tested were Adper Easy Bond, Clearfil S3 Bond, iBond, Optibond All-in-One, Xeno IV, and Adper Single Bond Plus as a control. The DC of each adhesive was measured on the surfaces of dentin discs (n=5) by attenuated total reflectance Fourier transform infrared spectroscopy. One hundred and forty-four extracted human molars were randomly divided and assigned to one of the five tested adhesives and the control group. The μTBS to dentin was measured on flat occlusal dentin with and without thermocycling and to the gingival floor dentin of class II cavities (n=8). All specimens were restored with Filtek Z250 resin composite. Class II samples were immersed in a 5% methylene blue dye solution for 24 hours, and microleakage was examined under a stereomicroscope. Micromorphological analysis of demineralized/deproteinized specimens was done using scanning electron microscopy. The DC and microleakage data were statistically analyzed by one-way analysis of variance (ANOVA) and μTBS data by two-way ANOVA followed by a Bonferroni multiple comparison *post hoc* test (α=0.05) and Weibull-distribution survival analysis. The relation between differ-

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ent variables and μTBS and microleakage was tested by the Pearson correlation coefficient and regression statistics. A moderate direct relation between DC and μTBS durability was found for all the adhesives tested. Significant wide variations exist among the results obtained for single-bottle adhesives tested regarding their μTBS and microleakage. Some of the all-in-one materials tested have shown significantly inferior results under a high C-factor or after aging. The use of these materials should be carefully considered.

INTRODUCTION

In the past few decades, there has been a significant increase in patient demand for esthetic restorations, and in response, direct resin composites have been widely used not only in the anterior area but also in various cavities in the posterior teeth. The major shortcoming of those adhesive restorations is their limited durability *in vivo*.[1] A weakened bond, poor marginal adaptation, and subsequent gap formation at the resin-dentin interface are common with these restorations, which may be followed by failure of the restoration in the form of microleakage, hypersensitivity, and recurrent caries.[2]

Establishing adequate bonding to dentin has proven to be a challenging task. Self-etch adhesives were introduced to overcome the technique sensitivity that might be encountered with the etch-and-rinse systems. These systems incorporate acidic components to partially demineralize and infiltrate dentin simultaneously.[3] Two-step self-etch adhesives show good and durable bonding to dental tissues.[4] On the contrary, various studies report conflicting results on the performance of one-step self-etch (all-in-one) adhesives.[5] Although excellent immediate and short-term bonding effectiveness of this class of dental adhesives has been revealed, the durability and stability of the bonded interfaces on dentin of some all-in-one bonding systems remain questionable.[6] The poor performance was attributed mainly to the hydrophilic nature of these materials and their liability to degradation by water or enzymatic activity of matrix metalloproteinases in the long term.[7] Optimal infiltration of the adhesive into the demineralized substrate and a high degree of conversion (DC) are essential in establishing long-lasting bonds.[8] Moreover, polymerization contraction exposes restoration-tooth interfaces to tensile stresses, leading to interface degradation and decreases in microtensile bond strength (μTBS), especially when restoring cavities with a high C-factor.[9,10]

The purpose of the present study was to 1) compare bond strength and microleakage of different all-in-one adhesives, 2) determine the effect of aging by thermocycling and cavity configuration on the bonding effectiveness, and 3) determine the relation between degree of conversion (DC), cavity configuration, microleakage, and bond strength. The null hypotheses tested in this investigation were that there is no significant difference among products tested and that the bond strength and microleakage of those adhesives are not a function of aging, degree of adhesive cure, or cavity configuration.

METHODS AND MATERIALS

Five all-in-one adhesives and one etch-and-rinse adhesive as the control group were evaluated in this study. Names and compositions of the tested materials are listed in Table 1, and their application procedures are listed in Table 2.

Degree of Conversion

Thirty 1.0-mm-thick dentin discs were prepared from extracted sound human molars. Teeth were cleaned from stains, calculus, and soft tissues with an ultrasonic scaler and stored in 0.5% chloramine-T at 4°C and used within 1 month. Dentin disc surfaces were polished with SiC papers up to 1000-grit under running water for 1 minute to create a standardized smear layer. Dentin discs were divided equally into six groups (n=5), and each group was assigned to one of the five all-in-one adhesives or control. The tested adhesives were placed on dentin and light cured according to the manufacturer's instructions (Table 2) with an LED light-curing unit (Demetron A.1, Kerr/Sybron, Orange, CA, USA) with a curing distance of 0.5 mm operating at 1600 ± 10 mW/cm^2. The light intensity was verified every five specimens using a digital curing radiometer (Cure Rite, Dentsply Caulk, Milford, DE, USA). Ten minutes after exposure to the curing light, the cured films were rinsed with acetone to remove the oxygen-inhibited layers and air-dried, and the DC was measured and analyzed by attenuated total reflectance Fourier transform infrared spectroscopy (FTIR; Spectrum GX, PerkinElmer, Coventry, UK). Spectra of the set materials were taken (400-4000-cm^{-1} wave number range, 4-cm^{-1} resolution, 40 scans coaddition, and 2.5-μm depth of analysis at 1000 cm^{-1}). Spectra were acquired for each material before polymerization to serve as a reference. The DC of each specimen was estimated on a relative

Table 1: *Names, Codes, Manufacturers, and Composition of Tested Adhesives*

Adhesive	Manufacturer	Composition
Adper Easy Bond (AP)	3M ESPE, St Paul, MN, USA	HEMA, bis-GMA, methacrylated phosphoric esters, 1,6-hexanediol dimethacrylate, methacrylate-functionalized polyalkenoic acid, silica filler (7 nm), ethanol, water, camphorquinone, stabilizers
Clearfil S3 (S3)	Kuraray America, New York, NY, USA	MDP, HEMA, bis-GMA, hydrophobic dimethacrylate, ethyl alcohol, water, photoinitiator, colloidal silica
iBond (IB)	Heraeus Kulzer, Hanau, Germany	UDMA, 4-MET, glutaraldehyde, acetone, water, photoinitiators, stabilizer
Optibond All-in-One (OB)	Kerr Corp, Orange, CA, USA	GPDM, mono- and difunctional methacrylate monomers, water, acetone, ethyl alcohol, camphorquinone, nanosilica and sodium hexafluorosilicate fillers
Xeno IV (XB)	Dentsply Caulk, Milford, DE, USA	Mono-, di-, and trimethacrylate resins, PENTA, photoinitiators, stabilizers, cetylamine hydrofluoride, acetone, water
Adper Single Bond Plus (SB)	3M ESPE	Bis-GMA, HEMA, dimethacrylates, polyalkenoic acid copolymer, initiators, water, ethanol, silica nanofillers

Abbreviations: HEMA, 2-hydroxyethyl methacrylate; Bis-GMA, bisphenol A and glycidyl methacrylate; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; UDMA, urethane dimethacrylate; 4-MET, 4-methacryloxyethyltrimellitate; GPDM, glycerophosphate dimethacrylate; PENTA, dipentaerythritol pentaacrylate phosphate.

percentage basis with the two-band method and the tangent baseline technique.[11] The peaks of the aliphatic (C=C) bonds stretching vibrations at 1636 cm^{-1} were chosen as the analytical band, whereas the peaks of the aromatic (C=C) bonds stretching vibrations at 1607 cm^{-1}, which are not affected by the polymerization reaction, were selected as the reference band. The DC was calculated using methods commonly found in the literature[12,13] and according to the equation

$$\%DC = 1 - \frac{(AliphaticC = C/AromaticC = C)Polymer}{(AliphaticC = C/AromaticC = C)Monomer} \times 100$$

Table 2: *Application Procedures of Tested Adhesives*

Adhesive	Application Procedure
Adper Easy Bond (AP)	1. Apply adhesive to enamel and dentin (two layers) and scrub for 20 s 2. Gentle air blow for 5 s 3. Light cure for 10 s
Clearfil S3 (S3)	1. Adhesive application (two layers) 2. High-pressure airstream for 5 s 3. Light polymerize for 10 s
iBond (IB)	1. Application of adhesive (two layers) for 20 s with agitation 2. Start with gentle air blow, followed by a strong air blow for at least 5 s 3. Light cure for 20 s
Optibond All-in-One (OB)	1. Adhesive application and scrubbing for 20s (two layers) 2. Gentle air blow, then medium-force air dry for 5 s 3. Light cure for 10 s
Xeno IV (XB)	1. Adhesive application and scrubbed for 20 s (two layers) 2. Gentle air blow for 5 s 3. Light cure for 10 s
Adper Single Bond Plus (SB)	1. Etch enamel and dentin, with 37% phosphoric acid, rinse with water, and blot dry 2. Adhesive applications (two to three layers) and scrub for 15 s 3. Air blow for 5 s 4. Light cure for 10 s

Microtensile Bond Strength

One hundred and forty-four noncarious, nonrestored human molars were collected and treated as mentioned previously. Occlusal enamel was removed, and the exposed dentin on the occlusal surfaces was examined under a light microscope (Nikon Measurescope UM-2, Nikon Corp, Kanagawa, Japan) for any remnants of enamel or pulp exposure. A second cut was made in the root approximately 3 mm from the cemento-enamel junction, creating two parallel surfaces. All specimens were mounted on plastic blocks.

Bond Strength to Occlusal Flat Dentin Surfaces

Ninety-six of the prepared specimens were selected randomly, and an automatic grinder/polisher with a 600-grit SiC paper disc running at 120 rpm under water cooling for 1 min (Buehler, Lake Bluff, IL, USA) was used to create a standardized smear layer on the flat occlusal dentin. Specimens were randomly divided into six experimental groups. Each group was assigned to one of the five all-in-one adhesive systems and the control. Adhesive systems were applied according to the manufacturer's instructions. Specimens were restored with hybrid resin composite (Filtek Z250, 3M ESPE, St Paul, MN, USA) using two increments each 1.5 mm thick, packed with the help of a plastic instrument, and cured for 20 seconds. Specimens were stored in double-distilled water at 37°C for 1 week to allow for bonded interface maturation before testing. Specimens of each group were divided in two equal subgroups (n=8). The first subgroup (flat TC) was subjected to 5000 thermal cycles between two water baths of 5°C and 55°C with a dwell time of 30 seconds at each temperature extreme (Thermocycler, Willytec, Munich, Germany) before μTBS testing. The second subgroup was tested right after water storage (flat non-TC).

Specimens were sectioned serially in a mesiodistal direction and perpendicular to the bonded surfaces, rotated 90 degrees and sectioned in the bucco-lingual direction to obtain 16 beams from each tooth (0.8±0.2 × 0.8±0.2 mm). The four sticks taken from the center of the restorations were selected for μTBS testing to exclude the variable of degree of approximation of the beam to the outer enamel layer. Specimens were mounted on a μTBS tester (Bisco Dental Products, Schaumburg, IL, USA) with cyanoacrylate and stressed to failure. The maximum kilograms force necessary to break the bonds in tension was recorded. The bonded surface area at the adhesive interface was calculated at the fracture site using a digital micrometer with 0.01 mm precision. Bond strength was obtained and expressed in MPa by dividing the measured force by the cross-sectional area of the bonded surfaces in centimeters.

Bond Strength to Proximal Class II Cavities

The remaining 48 teeth were sectioned parallel to the long axis of the tooth to remove proximal enamel and expose superficial dentin at the two proximal surfaces of each tooth. An occlusal slot cavity was prepared at the mesial site of the tooth with standardized dimensions of 2.5 ± 0.25 mm facio-lingually, 1.5 ± 0.25 mesio-distally, and 3.0 ± 0.25 mm occluso-gingivally, using a high-speed carbide bur (FG 245, KG Sorensen, SP, Brasil) under constant water cooling. The bur was replaced after every two preparations. Teeth were randomly divided into six experimental groups (n=8). Each group was assigned to one of the five all-in-one adhesive systems or the control adhesive.

Adhesive systems were applied according to manufacturer's instructions, then a clear plastic strip (Mylar Matrix Strips, Patterson Dental Supply, St Paul, MN, USA) was applied around the crown of the tooth to cover the proximal surface and held in place with a metal paper clip applied on the buccal and lingual surfaces of the specimen. All cavities were restored with hybrid resin composite (Filtek Z250, 3M ESPE), using two increments, each 1.5 mm thick, and cured from the occlusal direction with an LED light-curing unit with the same curing protocol mentioned previously and subjected to thermocycling.

The apices of the roots were sealed with resin-modified glass ionomer restorative (Fuji II LC, GC America Inc, Alsip, IL, USA). The entire surface of each specimen was then covered with two coats of varnish up to 1-mm from the restoration margins. Specimens were soaked in an aqueous solution of 5% methylene blue dye for 24 hours at 37°C. Following dye exposure, the specimens were rinsed thoroughly with double-distilled water for 30 seconds and kept moist for bond strength testing.

Specimens were serially sectioned in a mesio-distal direction, starting from the tooth restoration interface perpendicular to the gingival floor, into four slices, each 0.8 ± 0.1 mm in thickness. The outer two slices were separated for microleakage testing and micromorphological analysis, and the central two slices were rotated 90 degrees and sectioned again to obtain two beams with a cross-sectional area of 0.8 × 0.8 mm. The μTBS was tested and calculated as for the flat-surface specimens.

The debonded microtensile specimens were dehydrated in a desiccator containing dehydrated silica gel at room temperature for 24 hours, mounted on aluminum stubs, sputter coated with 100 Å gold-palladium, and examined to identify the failure mode by low-vacuum scanning electron microscopy (SEM; JSM 5310LV, JEOL Inc, Tokyo, Japan) running with a working distance of 20 mm at 10 kV of accelerating voltage and 60 mA of probe current. Micrographs were collected at magnifications up to 500×. The failures were characterized as adhesive when failure occurred either between adhesive resin and dentin or between adhesive resin and composite, cohesive when failure occurred within the adhesive layer or composite, and mixed when including two different types of failures.

Microleakage Test

The two outer slabs from each tooth were polished with SiC papers of increasing fineness (600-1200 grit) to create uniform flat surfaces and ultrasonically cleaned in distilled water for 10 minutes to remove any superficial debris created during the cutting and polishing procedures. Slabs were evaluated under a digital multiaxis dimensional measurement device (Quadra-chek 200, Metronics Inc,

Table 3: *Weibull Parameters and Mean Microtensile Bond Strength (μ-TBS) to Flat Surfaces With (TC) and Without Thermocycling (Non-TC) and to Class II Gingival Floor Dentin, Degree of Conversion (DC), and Microleakage of Adper Easy Bond (AP), Clearfil S3 Bond (S3), ibond (IB), Optibond All-in-One (OB), Xeno IV (XB), and Adper Single Bond Plus (SB)*

Material	μTBS to Flat Surface		μTBS to Class II Gingival Floor			DC %	Microleakage (Dye Penetration) (mm)
	Flat Non-TC (MPa)	Flat TC (MPa)	Class II TC (MPa)	Weibull Characteristic Strength (MPa)	Weibull Modulus		
AP	43.55 (6.2)^{Ba}	36.64 (6.6)^{ABb}	3.5 (1.0)^{Cc}	4.0	7.6	82.56 (1.88)^{AB}	2.9 (0.2)^{A}
S3	39.62 (7.1)^{Ba}	33.79 (3.8)^{Ba}	23.7 (11.2)^{ABc}	20.0	7.4	84.76 (3.20)^{A}	1.6 (0.2)^{CD}
IB	26.75 (4.2)^{Ca}	21.12 (4.5)^{Ca}	12.4 (9.3)^{DEc}	12.0	2.6	62.15 (3.36)^{D}	2.3 (0.2)^{B}
OB	48.40 (8.0)^{Ba}	33.32 (7.2)^{Bb}	18.8 (7.1)^{BDc}	18.0	9.0	63.50 (2.08)^{D}	1.2 (0.1)^{D}
XB	25.88 (5.5)^{Ca}	20.17 (3.8)^{Ca}	4.9 (2.4)^{CEc}	7.0	7.4	70.99 (3.74)^{C}	1.9 (0.2)^{C}
SB	58.17 (10.1)^{Aa}	43.56 (8.4)^{Ab}	23.9 (6.2)^{Ac}	27.0	7.3	77.02 (2.34)^{B}	1.4 (0.1)^{CD}

** Within a row, same lowercase superscript letters show mean values with no statistically significant difference ($p>0.05$).*
*** Within a column, same uppercase superscript letters show mean values with no statistically significant difference ($p>0.05$).*

Bedford, NH, USA) connected to a Measurescope (UM-2, Nikon) to measure the total depth of dye penetration in multiple axes. Dye penetration at the tooth-restoration interface at the gingival margin and axial wall of each slab was recorded in millimeters, and mean dye penetration of each tooth was calculated from the average of the readings of the two outer slabs.

Micromorphological Analysis

After the microleakage test, one of the outer slabs was selected randomly from each tooth and immersed in 6 N hydrochloric acid for 1 minute to demineralize the dentin, rinsed with tap water for 5 minutes, and then deproteinized by immersion in 2.5% sodium hypochlorite for 5 minutes and rinsed again with tap water for 5 minutes. Slabs were desiccated, sputter coated, and examined under SEM for morphological analysis of the bonded interface. Micrographs were collected at different magnifications up to 5000×.

Statistical Analysis

The μTBS results were analyzed with statistical software (SPSS version 20.0, SPSS Inc, Chicago IL, USA) using two-way analysis of variance (ANOVA) and a Bonferroni *post hoc* multiple comparison test (α=0.05). To estimate bonding performance, the μTBS data were also analyzed using Weibull-distribution survival analysis; the analysis included a frailty term to correlate the measurements from beams coming from the same specimen, following the protocol recommended by Eckert and Platt in 2007.[14] Specimens that spontaneously debonded were treated as left-censored at the lowest measured strength, and specimens that debonded due to cyanoacrylate failure or that did not fail prior to the end of testing were treated as right-censored at the highest measured strength. If the failure of these specimens was at a lower value, then they were treated as censored at the measured MPa. The DC and microleakage results were analyzed using one-way ANOVA followed by a Bonferroni *post hoc* multiple comparison test (α=0.05). Correlation between DC, microleakage, μTBS, thickness of the hybrid layer, and resin tag length was analyzed by calculating the Pearson correlation coefficient and regression statistics.

RESULTS

Mean values, standard deviations of DC, μ-TBS, and microleakage (dye penetration) of the five all-in-one adhesives tested and the control adhesive material in all experimental conditions are presented in Tables 3 and 4. The Weibull-distribution curve is presented in Figure 1. Typical failure patterns of the six adhesives tested are listed in Table 4 and illustrated in Figure 2a through f, and micromorphological analysis of the adhesive-dentin interfaces is presented in Figure 3a through f.

Degree of Conversion

According to the results of the present study, Clearfil S3 Bond (S3) showed the highest values (84.76±3.20) of DC; however, there was no statistically significant difference between S3 and Adper Easy Bond (AP; 82.56±1.88). Self-etch ibond (IB) and Optibond (OB) adhesives showed the lowest DC of all products tested.

Table 4: *Mode of Failure Percentage (%) of Adper Easy Bond (AP), Clearfil S3 Bond (S3), ibond (IB), Optibond All-in-One (OB), Xeno IV (XB), and Adper Single Bond Plus (SB)*

Material	Mode of Failure (%)		
	Adhesive	Cohesive	Mixed
AP	16	—	84
S3		—	100
IB	96	—	4
OB	66	13	31
XB	31	—	69
SB	79	—	21

** Adhesive failures are either between adhesive resin and dentin or between adhesive resin and composite. Cohesive failures are within adhesive layer or composite. Mixed failures includes two different types of failures.*

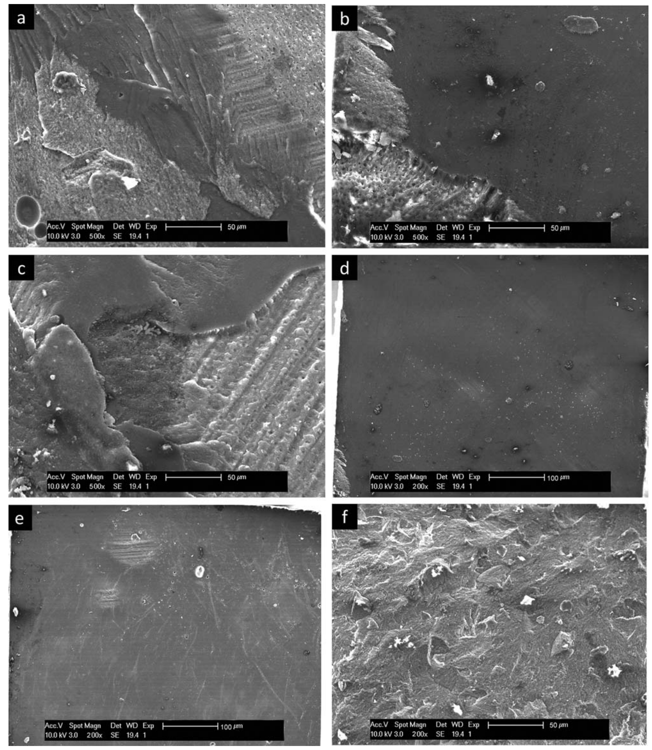

Figure 2. *Representative examples of failure mode as seen by scanning electron microscopy (SEM) on the bottom dentin side (class II restorations). (a): Optibond All-in-One (OB). (b): Adper Easy bond (AP). (c): Clearfil S3 Bond (S3). (d): ibond (IB). (e): Xeno IV (XE). (f) Adper Single Bond Plus (SB).*

Microtensile Bond Strength

According to the results, when bonded to flat surfaces (flat non-TC), the control group Adper Single Bond Plus (SB) showed the highest bond strength (58.17±10.1) among all adhesives tested, while the lowest values were recorded for IB (26.75±4.2) and Xeno IV (25.88±5.5). When specimens were subjected to thermocycling (flat TC), all bond strength values were negatively affected; however, this reduction was statistically significant for SB, OB, and AP only. There was a moderate correlation between DC and μTBS reduction after thermocycling (Pearson coefficient=0.5116).

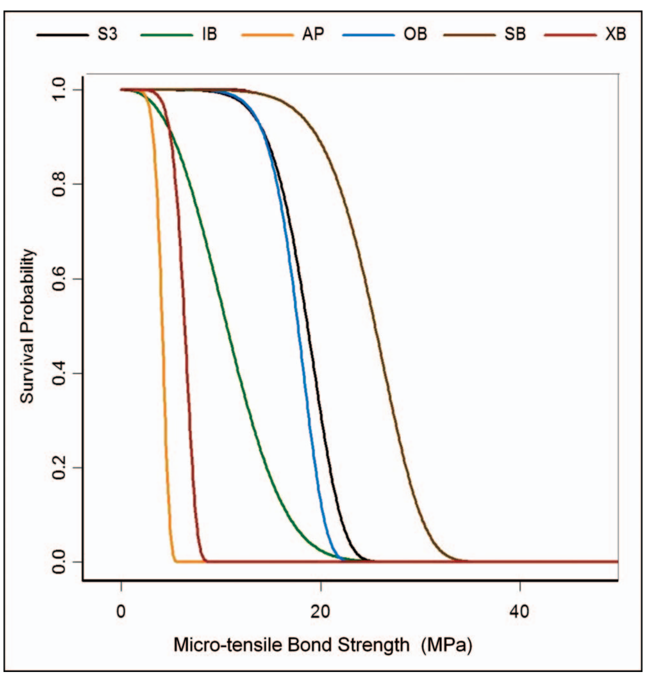

Figure 1. *Weibull-estimated survival probability of microtensile bond strengths (μ-TBS) to class II gingival floor dentin of Adper Easy Bond (AP), Clearfil S3 Bond (S3), ibond (IB), Optibond All-in-One (OB), Xeno IV (XB), and Adper Single Bond Plus (SB).*

Comparing μTBS to the gingival floor of the class II cavity (class II TC) with that of flat surfaces after thermocycling (flat TC), all bond strength values for class II TC were significantly lower than those for the flat TC group for all adhesives tested. Control group SB and S3 showed the highest μTBS (23.9±6.2 and 23.7±11.2, respectively), while μTBS values reported for AP (3.5±1.0) and XB (4.9±2.4) were very low, with no statistically significant difference. High standard deviations, a low Weibull modulus, and a high number of pretesting failures were recorded for both products.

Regarding failure patterns (Table 4), in flat dentin surfaces, most of the failures were recorded as mixed adhesive and/or cohesive regardless of adhesive. However, in the class II cavity dentin gingival floor (class II TC), only S3 showed 100% mixed-type failure (Figure 2c). The rest of the adhesives showed both mixed and adhesive failures with the IB and control group SB failing adhesively at a high percentage (96% and 79%, respectively).

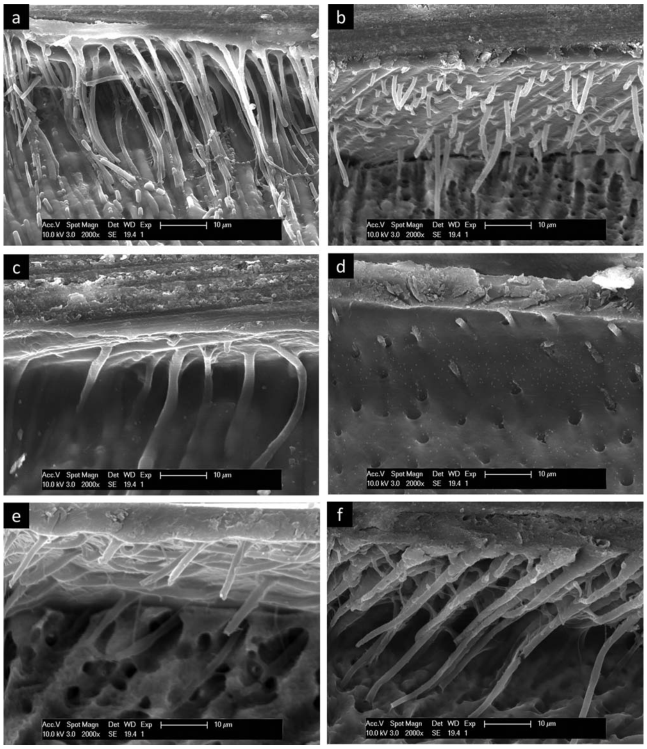

Figure 3. *Representative examples of bonded interface analysis of demineralized/deproteinized samples (class II bottom dentin) seen by scanning electron microscopy (SEM). (a): Optibond All-in-One (OB). (b): Adper Easy Bond (AP). (c): Clearfil S3 Bond (S3). (d) ibond (IB). (e): Xeno IV (XE). (f): Adper Single Bond Plus (SB).*

Microleakage

AP showed the highest ($p<0.05$) dye penetration (2.9 ± 0.2 mm) compared with all other materials, while the least dye penetration ($p<0.05$) was exhibited by OB (1.2 ± 0.1 mm). However, there was no statistically significant difference between OB, control group SB (1.4 ± 0.1), and S3 (1.6 ± 0.2). Correlation analysis revealed a strong reverse relation between dye penetration and μTBS values (Pearson coefficient=0.642, $p<0.01$).

Micromorphological analysis of the adhesive-dentin interfaces of the control group SB (Figure 3f) showed a much thicker hybrid layer (over 8 μm) and long resin extensions in the lateral branches of the dentinal tubules when compared to all-in-one products, which in most of the cases exhibited thin hybrid layers not exceeding 1 μm and short resin tags not exceeding 5 μm except for OB (Figure 3a), which showed a resin infiltration similar to that of the control group. Gap formation and separation between the adhesive layer and the composite was identified in most of the samples. Correlation statistics revealed no direct relation between the μTBS and the thickness of the hybrid layer or the length of the resin tags for any of the adhesives.

DISCUSSION

Five all-in-one bonding agents and one etch-and-rinse adhesive were evaluated. The results of the present study led to rejection of all the null hypotheses. Thermocycling and cavity configuration had a significant effect on the μTBS. The DC was correlated with a decrease of μTBS after thermocycling, and there was a strong inverse relation between microleakage and μTBS in class II gingival floor dentin.

In all three different testing conditions, etch-and-rinse control (SB) showed better bonding efficiency than most of the all-in-one adhesives tested. The results of the present study fit well with previous data showing that one-bottle self-etch adhesives are inferior in bonding efficiency to etch-and-rinse products.[15] Several explanations—such as weakened adhesion of the restorative resin composite to the adhesive layer due to a high-oxygen inhibition layer and high acidity,[16] incomplete wetting and insufficient thickness of the adhesive layer, difficulty evaporating residual solvents, and phase separation between hydrophilic and hydrophobic ingredients and the resulting sensitivity to hydrolysis—have been seen as contributing to lower bonding performance of one-bottle all-in-one self-etch adhesives to dentin as compared to the three-step etch-and-rinse and two-step self-etch adhesives.[7,17-19]

Today's adhesives are complex mixtures of functional and cross-linking monomers, curing initiators, inhibitors or stabilizers, solvents, and often silica fillers. The performance of the all-in-one adhesives tested varied, depending on the testing conditions. Differences in their composition and application mode seem to be the key reasons for the different performance of the adhesives tested.[3]

Many studies investigating the performance of self-etch adhesives appear in the literature. However, most of the studies used no thermal or mechanical stresses and do not take into account polymerization stresses and cavity configuration.[20]

In the current study, μTBS was tested and compared on flat dentin surfaces with (flat TC) and without thermocycling (flat non-TC). Then μTBS was evaluated in class II composite resin restorations gingival floor dentin (class II TC) and compared with the bond strength on the flat dentin surfaces (flat TC). In both cases, samples were subjected to thermocycling under similar conditions. To ensure minimal variations in polymerization shrinkage stresses, standard class II cavities were made, and the same composite resin was used in all groups (flat surfaces and class II cavities). Orientation of the slices and the size of the sticks were standardized so that the bonded areas tested were identical.

According to the results, thermocycling and cavity configuration affected the bonding efficacy of all adhesives tested, even the control two-step etch-and-rinse adhesive (SB). The μTBS values were further significantly decreased in class II restorations after thermocycling for all adhesives tested. The possibility of improper drying of etched dentin should be considered because of the narrower dimensions of the class II cavities prepared in this study. High polymerization contraction stresses, along with high difficulty in removing residual solvents adequately from these hydrophilic adhesives, may be the main reason for this inferior bonding performance in class II restorations.[10]

All-in-one adhesive S3 showed the highest bonding efficiency of all the all-in-one self-etch products tested. S3 exhibited the least reduction in bond strength after thermocycling. It is interesting to notice that S3 on flat surfaces showed inferior bond strength compared to control group SB, but when evaluated on class II restorations after thermocycling, no significant statistical difference was recorded in μTBS values between S3 and control group SB. The findings of our study are in accordance with previous studies comparing S3 with other all-in-one adhesives that reported good bond strength to enamel and dentin even under cyclic loading and mechanical stress.[7,30,31] S3 adhesive is characterized by mild acidity and containing 10-methacryloxydecyl dihydrogen phosphate (10-MDP) in its composition as a functional monomer. This specific molecular composition is capable of interacting with residual hydroxyapatite within the hybrid layer, forming a stable MDP-Ca salt deposition and a strong nanolayer at the adhesive interface.[33] This chemical interaction acting synergistically with superior infiltration into the decalcified substrate, the mild acidity of S3, the homogeneity of the adhesive, and the lack of phase separation might be responsible for enhanced bond stability over time.[15,35]

Low bond strengths were identified for the remaining all-in-one adhesives, especially in class II restorations after thermocycling. An interesting finding was that the modes of failure for all self-etch adhesives tested were mostly mixed on flat surfaces, but in class II gingival floor dentin, an increase in adhesive mode of failure was identified, which might be related to low bond strength.

IB and XB provided the lowest mean bond strength among all adhesives tested regardless of thermocycling. Our study confirmed the poor results of these products seen in previous studies.[39,40] Previous reports show high pretesting failures reaching up to 51.1% for IB, which was related to poor collagen infiltration. IB is an acetone-containing non–2-hydroxyethyl methacrylate adhesive that has been associated with severe phase separation, leading to porosities or blisters occurring at the bonding interface.[15,44,45]

A low DC of dental adhesives has been associated with monomer elution and possible continuous demineralization of dentin, low bond strength values, increased permeability, and phase separation.[48-50] Incomplete polymerization of the adhesive can accelerate the water-degradation effects, leading to bonding deterioration.[51-53]

In most of the studies, degree of conversion evaluation was performed in specimens polymerized on a glass coverslip or an inert surface and not in contact with the bonding structure. In the present study, cured adhesive films fixed on dentin were rinsed with acetone to remove the O_2-inhibition layer from the surface of the adhesive film before FTIR measurements. The DC was evaluated after applying each adhesive on a dentin substrate and not on a glass coverslip or directly onto the instrument's minicrystal, closer to the clinical situation. The quantitative measurement of DC inside the hybrid layer can provide some information to explain current adhesive performance. Self-etch adhesives show a better DC when placed on dentin than on an inert surface.[15]

Differences in DC among the materials tested were observed. A number of reasons might be responsible, such as the resin and filler composition, the initiating system, filler loading, and type of solvent. Acetone-containing adhesives, such as OB and IB tested in the present study, may achieve lower DC compared to the ethanol-containing adhesives.[54,55] High volume of water or solvents makes the viscosity low and leads to a decrease in conversion and an increase in O_2 inhibition.[8]

In the present study, a moderate correlation between DC and bond strength was identified. In the literature, contradictory results have been reported by studies evaluating the effect of DC on bond strength. Two studies evaluating the correlation between μTBS and DC of adhesive systems failed to find any correlation.[49,53] Another study demonstrated that increased DC is related to an increase in the quality of the polymer network and thus less nanoleakage and higher μTBS.[56] Bond strength is multifactorial, and other factors, such as the composition of the material, are involved in the performance of the adhesives.

Although a negative correlation between dye penetration and μTBS was identified in many studies,[58] a moderate correlation was found between dye penetration and bond strength on the class II gingival floor dentin in the current study. This finding can be attributed to measuring the two parameters using the same sample, which may allow for correlation that is more consistent. AP showed the highest dye penetration in comparison to all other materials and at the same time the lowest bond strength values. The least dye penetration was exhibited by OB, but there was no significant difference between OB, S3, and control group SB. Those three adhesives presented good bonding performance as well.

Micromorphological evaluation of the dentin-adhesive material interface of the samples under SEM with high-magnification imaging showed no direct relation between the depth of infiltration in dentin and the thickness of the hybrid layer or the length of the resin tags for any of the adhesives. An interesting finding was that AP and XB, which exhibited gaps and separation between the adhesive and the resin composite, showed a high number of pretesting failures in class II restorations and a high number of adhesive mode failures. On the contrary, S3 presented a very thin hybrid layer and very few resin tags but no interfacial gaps, and this may explain the results of microleakage resistance and bonding efficiency of S3, which were comparable with the etch-and-rinse control adhesive.

It seems that many factors, such as degree of infiltration of the resin monomers into the collagen, porosity due to blisters at the bonding interface, and hydrolytic degradation of the resin components in the hybrid layer, are related to the integrity of the bonding process.[60]

CONCLUSIONS

Within the limits of this study, the following conclusions can be drawn:

1. The control etch-and-rinse adhesive showed better bonding efficiency and dye penetration resistance in all testing conditions than the all-in-one self-etch adhesives tested.
2. The μ-TBS of single-bottle adhesives varies significantly due to wide variations in their chemical composition.
3. DC, thermocycling, and cavity configuration had a significant effect on the bond strength of both etch-and-rinse and self-etch products.
4. A good correlation exists between dye penetration and bond strength when the tested adhesive systems are bonded to the class II gingival dentin floor.

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Human Subjects Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the University of Sharjah. The approval code for this study is 111010.

Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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