

One-year Adhesive Bond Durability to Coronal and Radicular Dentin Under Intrapulpal Pressure Simulation

HA El-Deeb • O Badran • EH Mobarak

Clinical Relevance

A two-step self-etch adhesive maintains high bonding strength to both coronal and radicular dentin even after long-term storage under intrapulpal pressure simulation.

SUMMARY

Objective: To evaluate the microtensile bond strength (μ TBS) of different adhesives to coronal vs radicular dentin after one year of storage in artificial saliva and under intrapulpal pressure (IPP) simulation.

Methods and Materials: Roots of 36 freshly extracted premolars were sectioned 5 mm apical to the cemento-enamel junction and pulp tissue was removed. Buccal enamel and cementum were trimmed to obtain standardized flat dentin surfaces. Specimens were

divided into three groups ($n=12/\text{group}$) according to the adhesive strategies utilized: a two-step etch-and-rinse adhesive; a two-step self-etch adhesive; and a single-step self-etch adhesive. Adhesives and resin composite were applied to coronal and radicular dentin while the specimens were subjected to IPP simulation. After curing, specimens were stored in artificial saliva at 37°C in a specially constructed incubator while the IPP was maintained for either 24 hours or one year prior to testing. Bonded specimens were sectioned into sticks with a cross section of $0.8 \pm 0.01 \text{ mm}^2$ and subjected to μ TBS testing. Data were statistically analyzed using multi-way analysis of variance (ANOVA) with repeated measures; one-way ANOVA tests; and Bonferroni post hoc test ($p<0.05$). Failure modes were determined using a scanning electron microscope at 100× magnification.

Results: ANOVA results revealed a statistically significant effect for the adhesive strategy ($p<0.001$) and storage period ($p<0.001$) as well as for their interaction ($p=0.024$) on the μ TBS. However, dentin substrate and its interactions

Heba Ahmed El-Deeb, associate professor, Restorative Department, Faculty of Oral and Dental Medicine, Cairo University, Cairo, Egypt

Omar Badran, PhD student, Faculty of Oral and Dental Medicine, Restorative Department, Cairo University, Cairo, Egypt

*Enas Hussein Mobarak, EBM, professor, Restorative Department, Faculty of Oral and Dental Medicine, Cairo University, Cairo, Egypt

*Corresponding author: 14 ElAnsar str, Cairo, 12311, Egypt; e-mail: enasmobarak@hotmail.com

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revealed no significant effects. For both dentin substrates, the two-step self-etch adhesive revealed statistically significantly higher μ TBS values than did the other two adhesives after 24 hours and one year of storage. After one-year storage, a significant decrease in bond strength values of all tested adhesives occurred with both dentin substrates. Modes of failure were mainly adhesive and mixed.

Conclusions: Adhesives were not sensitive to structural differences between coronal and radicular dentin even after one year of storage under IPP simulation. However, all tested adhesive systems strategies were sensitive to storage.

INTRODUCTION

New adhesive systems have been developed in an attempt to reduce the steps and simplify the clinical bonding procedures. However, one of the challenges facing the adhesive systems' manufacturers has been, and still remains, the development of adhesive agents that adhere equally well to different tooth substrates.

Dentin is a biologic composite structure composed of apatite filler crystallites in a collagen matrix with a fluid-filled tubular structure connecting the pulp to the dentino-enamel junction. This heterogeneous and intrinsically wet substrate changes with different dentin depths and varies from location to location.¹ The use of coronal dentin is adequate as a means to obtain information about the bonding efficacy of any material. However, in the clinical situation, bonding is performed to dentin, which is located at various sites. Recent developments in preventive dentistry and periodontology have considerably increased the demand for restoration of root dentin defects such as cervical erosion, abrasion, and root caries.^{2,3} In root dentin, there is a significant reduction in the average density of dentinal tubules running in a straight course compared with coronal dentin tubules running in an "s"-shaped course.⁴ These variations in density and morphology of dentinal tubules were reported to affect the interaction between earlier versions of dentin adhesives with different dentin sites.^{5,6}

Another issue is related to the outward fluid movement through dentinal tubules, which is one of the most critical differences between clinical and laboratory conditions.⁷ As a consequence, it is necessary to employ pulpal pressure simulation when adhesives are tested *in vitro*. On reviewing

the literature, no research has been conducted to determine the long-term bond durability to coronal and radicular dentin when intrapulpal pressure (IPP) is simulated. The null hypotheses were the following: 1) Bonding to coronal or radicular dentin has no influence on adhesive bond strength. 2) There is no difference in the microtensile bond strength among different adhesive systems of different bonding strategies. 3) Storage under IPP simulation has no effect on bond strength of different adhesives to dentin.

METHODS AND MATERIALS

Specimen Preparation

Thirty-six sound human lower premolar teeth, extracted for orthodontic reasons from young patients (14-17 years), were collected and stored in phosphate buffer solution containing 0.02% sodium azide at 4°C for not more than one month until they were used. The roots were trimmed perpendicular to the long axis of the teeth, leaving 5 mm apical to the cemento-enamel junction (CEJ). The pulp tissue was removed from the pulp chamber using a broach (Mani Inc, Utsunomya Tochgi, Japan), size 35,⁸ and then the pulp chamber was irrigated with saline solution to ensure complete cleanliness of the chamber.⁹ Each tooth segment was fixed perpendicularly from the cut root surface to the center of a circular Teflon plate (11-mm diameter and 1.5-mm thickness, with a central hole of 1-mm diameter) using a cyanoacrylate adhesive (Rocket Heavy, Dental Ventures of America Inc, Corona, CA, USA). A 19-gauge stainless-steel butterfly needle (Shan-chuan Medical Instruments. Co, Ltd, Zibo, China) was verified to penetrate the plate to reach the root canal of the tooth. A line was drawn with an indelible pen demarcating the middle of the proximal surface. Another line was drawn at the CEJ to differentiate between coronal and root dentin surfaces. The tooth segment attached to the Teflon plate was horizontally embedded in a polyester resin (Polyester resin #2121, Hsein, Taiwan) up to the level of the middle of the proximal surfaces that was demarcated, while the lingual surface was facing downward and the buccal surface was facing upward. The needle was left inserted in the root canal during embedding to guarantee a patent pathway to the pulp chamber. Buccal enamel and dentin were then trimmed parallel to the tooth long axis using a slow-speed diamond saw sectioning machine (Buehler Isomet Low Speed Saw, Lake Bluff, IL, USA) under water coolant to obtain a standardized flat dentin surface.

Table 1: Materials, Compositions, and Application Procedures		
Material (Manufacturer)	Composition	Application procedures
Adper Single Bond 2 <ul style="list-style-type: none">Two-step etch-and-rinse adhesive system3M ESPE Dental Products, St Paul, MN, USA Batch#51202	<i>Etchant:</i> 35% Phosphoric acid, colloidal silica. <i>Adhesive:</i> Bis-GMA, HEMA, dimethacrylates, ethanol, water, photoinitiator, methacrylate functional copolymers of polyacrylic and polyitaconic acids, silica nanofillers.	<i>Etching:</i> Apply for 15s, water rinsing for 10s then blot excess water with minisponge (visibly moist surface). <i>Adhesive:</i> Apply with gentle agitation for 15s, gently air-thin for 5s and light cure for 10s.
Clearfil SE Bond <ul style="list-style-type: none">Two-component two-step self-etch adhesive systemKuraray Medical Inc. Sakazu, Kurashiki, Okayama, Japan Primer: Batch #00999A Adhesive: Batch #01486A	<i>Primer:</i> MDP, HEMA, hydrophilic dimethacrylate, D,L-camphorquinone, N,N-diethanol-p-toluidine and water. <i>Bond:</i> MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, dl-Camphorquinone, N,N-Diethanol-p-toluidine and silanated colloidal silica	<i>Primer:</i> Apply onto the visibly moist prepared tooth surface, leave undisturbed for 20s and then dry with oil-free mild air flow for 5s. <i>Bond:</i> One coat application and a gentle oil-free air stream for 5s then light cure for 10s.
Adper Easy One <ul style="list-style-type: none">(One-component single-step self-etch adhesive system)(3M ESPE Dental products, Seefeld, Germany) Batch #D-82229	HEMA, Bis-GMA, methacrylated phosphoric esters, 1.6 hexanediol dimethacrylate, methacrylate functionalized polyalkenoic acid (Vitrebond copolymer), finely dispersed bonded silica filler with 7nm primary particle size, ethanol, water, initiators based on camphorquinone, stabilizers.	Apply with the disposable mini-sponge brush tip for 20s to the whole dentin surface, then air thin with oil-free mild air flow for 5s until the film no longer moves, and cure for 10s.
<i>Bis-GMA=Bis-phenol-A glycidyl methacrylate, HEMA=2-hydroxyethyl methacrylate, MDP=10-Methacryloyloxydecyl dihydrogen phosphate.</i>		

The dentin surfaces were hand finished with wet 600-grit silicon carbide (SiC) abrasive paper for 20 seconds to obtain a clinically relevant uniform smear layer. The drawn line identifying the CEJ was regained to differentiate between coronal and root surface dentin. The teeth segments (n=36) were connected to the IPP assembly during bonding and storage following the same procedures described by Mobarak.¹⁰

Restorative Procedures

Prepared teeth segments with flattened dentin surfaces (coronal and radicular) were divided into three subgroups (n=12) according to the adhesive system strategies evaluated: a two-step etch-and-rinse adhesive system (Adper Single Bond 2, SB, 3M ESPE Dental Products, St Paul, MN, USA); a two-component two-step self-etch adhesive system (Clearfil SE Bond, SE, Kuraray Medical Inc, Okayama, Japan); and a one-component single-step self-etch adhesive (Adper Easy One, AE, 3M ESPE Dental Products, Seefeld, Germany). Each adhesive system was applied to moist dentin surfaces according to its manufacturers' instructions, as described in Table 1. Resin composite (Valux Plus, 3M ESPE Dental Products) of shade A3.5 was applied in two increments of 1.5 mm each, building up two blocks of resin composite (approximately 3 mm in height and 3 mm in length), to the prepared

coronal and radicular dentin, where a matrix band was placed to separate surfaces. Each composite increment was polymerized for 40 seconds using a Bluephase C5 light curing unit (Ivoclar Vivadent, Schaan, Liechtenstein) with an intensity of ≥ 500 mW/cm². Light intensity was checked using an LED radiometer (Kerr Dental Specialties, Orange, CA, USA). The specimens were then immersed in artificial saliva either for 24 hours (n=6) or one year at 37°C in a specially constructed large incubator to accommodate the IPP assembly. The artificial saliva was prepared according to Pashley and others¹¹ and was changed weekly.⁸

Microtensile Bond Strength Testing

Before specimen sectioning, the coronal composite build-up was color-coded to guarantee the differentiation of the sticks after sectioning. Each bonded tooth was sectioned in the X and Y axes to obtain sticks of 0.8 ± 0.01 mm² for the microtensile bond strength (μ TBS) test. From each specimen, sticks of similar length and remaining dentin thickness (four for coronal and four for radicular) were selected; thus, a total of 24 sticks of each experimental variable were tested. Each stick was fixed to the modified ACTA microtensile strength jig¹² with a cyanoacrylate adhesive (Rocket Heavy) and stressed in tension using a universal testing machine (Lloyd Instruments Ltd, Ametek Company, Bognor Regis,

Table 2: Microtensile Bond Strength (μ TBS) Values mean (standard deviation) in MPa of the Tested Adhesive Systems^a

Storage Periods	Coronal dentin			P-Value
	Adper Single Bond 2	Clearfil SE Bond	Adper Easy One	
24 hours	30.7 (5.2) A [Ptf/tnt=1/24]	39.9 (9.1) B [Ptf/tnt=0/24]	25.7 (3.4) A [Ptf/tnt=2/24]	<0.01
1 year	13.0 (2.6) A [Ptf/tnt=8/24]	29.4 (3.9) B [Ptf/tnt=2/24]	12.1 (3.3) A [Ptf/tnt=9/24]	<0.001
p-value	<0.0001	0.001	0.037	

^a [ptf/tnt=pretest failure/total number of tested sticks]. Within rows, for each dentin substrate, means with different capital letters are statistically significantly different ($p > 0.05$, Bonferroni test).

West Sussex, UK) at a cross-head speed of 0.5 mm/min until failure. The tensile force at failure was recorded and converted to tensile stress in MPa units using computer software (Nexygen-MT, Lloyd Instruments). Sticks that failed before testing were counted as zero MPa.⁸

The mean and standard deviation (SD) of each group were calculated. Comparison between groups was performed using the multi-way analysis of variance (ANOVA) with repeated measures where μ TBS was the dependent variable and the dentin site, adhesive strategies, and storage periods were the independent variables. The interactions between each of the two independent variables as well as the interaction among the three variables were also tested. A Bonferroni post hoc multiple-comparison test was used when indicated. A *t*-test was used to compare the bond strength values of 24-hour and one-year μ TBS mean values for each adhesive system with each dentin site. A *p*-value of <0.05 was considered statistically significant. Data were analyzed using SPSS for Windows (Statistical Package for Social Sciences, release 15 for MS Windows, 2006, SPSS Inc, Chicago, IL, USA).

Failure Mode Analysis

The fractured dentin side of all tested sticks was inspected under scanning electron microscopy (SEM) (Scanning Electron Microscope 515; Philips, Eindhoven, The Netherlands) to determine the mode of failure. The failure mode was allocated to either type 1: adhesive failure at dentin side; type 2: cohesive failure in the adhesive layer; type 3: mixed failure (adhesive at dentin side/cohesive in the adhesive layer); or type 4: mixed failure (adhesive at dentin side/cohesive in the adhesive layer/cohesive in resin composite). The frequency of each mode of failure was expressed as a percentage value.¹³

SEM Observation of the Bonded Coronal and Radicular Dentin Interfaces

An additional two sticks from each tested category were randomly selected for evaluation of the inter-

facial morphology using SEM (515; Philips). Sticks were polished using SiC paper of increasing grit size (1000, 1200, 2500, and 4000), rinsed with water for 30 seconds, etched with 10% phosphoric acid for 10 seconds; and deproteinized in 5% sodium hypochlorite for five minutes. After rinsing with distilled water, sticks were left to air-dry in a dessicator; they were then mounted on aluminum stubs and sputter-coated with gold to be examined using SEM at different magnifications.

RESULTS

Multi-way ANOVA with repeated measures revealed a significant effect for the adhesive strategies ($p < 0.001$) and storage periods ($p < 0.001$) as well as for their interactions ($p = 0.024$) on the μ TBS. However, dentin site and its interactions revealed no significant effects. The descriptive statistics, means, and SDs for the μ TBS (MPa) of all tested categories are presented in Table 2. The one-way ANOVA test also indicated that there was a significant difference among the adhesive systems with both dentin substrates when tested after 24 hours and after one year of storage (Table 2). The two-step self-etch adhesive (SE) revealed the highest mean bond strength compared with the two-step etch-and-rinse adhesive (SB) and the single-step self-etch adhesive (AE). The Bonferroni post hoc test revealed that SB and AE were significantly lower than SE at 24 hours as well as at one year. With regard to the effect of storage period, the *t*-test revealed a significant decrease in bond strength values of all tested adhesive systems to coronal and radicular dentin after one year of storage (Table 2).

Regarding the failure modes, Figure 1 shows the percentages of the recorded failure modes. For both dentin sites, after 24 hours of storage, fractured specimens for all adhesive system showed mainly a type 1 mode of failure. After one-year storage, for both dentin sites, SB fractured specimens showed predominantly a type 3 mode of failure; SE fractured specimens showed mainly types 1 and 3 modes of failure, while for the AE fractured specimens, failure

Table 2: Microtensile Bond Strength (μ TBS) Values (standard deviation) in MPa of the Tested Adhesive Systems ^a (ext.)				
Storage Periods	Radicular dentin			P-Value
	Adper Single Bond 2	Clearfil SE Bond	Adper Easy One	
24 hours	28.1 (4.5) A [Ptf/tnt=0/24]	43.3 (8.5) B [Ptf/tnt=0/24]	22.3 (3.0) A [Ptf/tnt=5/24]	<0.001
1 year	10.1 (2.3) A [Ptf/tnt=7/24]	32.7 (3.9) B [Ptf/tnt=2/24]	15.6 (3.7) A [Ptf/tnt=7/24]	<0.001
p-value	0.028	0.01	<0.01	

mode types 1 and 4 were mainly recorded. Representative SEM micrographs for the predominant failure modes recorded with tested adhesive systems bonded to either coronal or radicular dentin are presented in Figure 2.

Figure 3 shows the SEM images of the bonded coronal and radicular interfaces. Characteristic pictures for SB (etch-and-rinse) specimens were captured in which resin tags with conical swellings were detected. For the self-etch adhesives (SE and AE) specimens, SEM images showed uniform homogeneous hybrid layer and adhesive layer thickness.

DISCUSSION

Results of the present study indicate that the first null hypothesis should not be rejected, as there was no difference between coronal and radicular dentin bond strength values at 24 hours or at one year of storage. Earlier studies,^{14,15} indicated opposite results where lower bond strength values of root dentin were recorded compared to those of coronal dentin surfaces. They attributed this difference in bonding to a decrease in number and diameter of dentinal tubules as well as permeability in root dentin relative to those of coronal dentin which might reduce the hydrophilic resin infiltration capacity of adhesives.

On the contrary to previous findings and in agreement with part of our findings, Pazinato and others¹⁶ did not find any influence of tubule orientation or dentin site on adhesive bond strength to dentin. It is important to emphasize that all earlier studies did not test coronal and radicular dentin bond strength under simulated IPP and were done over short-term periods. In the present study, bonding and storage of the specimens were done under IPP simulation. Although IPP simulation in other studies has influenced dentin bond strength,^{17,18} the difference in dentin site and dentinal tubule orientation did not show a significant effect in the present study even after one year of storage under IPP simulation.

Regardless of dentin sites, dentin bond strengths of tested adhesive systems were significantly different leading to the rejection of the second null hypothesis. Long-term storage under IPP simulation significantly decreased bond strengths of all adhesives which suggested the rejection of the third null hypothesis.

Many authors^{16,19,20} confirmed that dentin bond strength is adhesive dependent. After 24 hours and one year, SE (the two-step self-etch adhesive system)

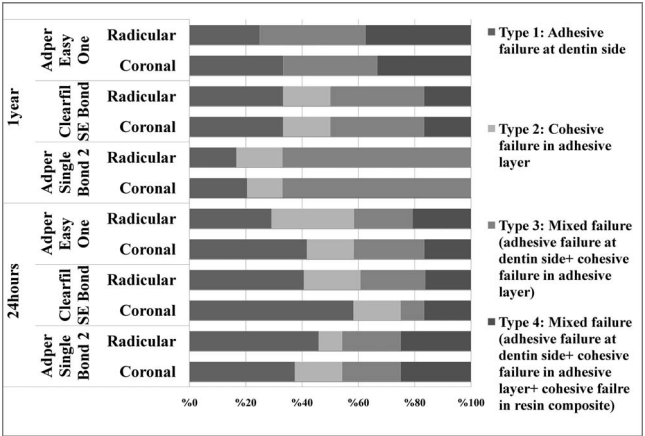


Figure 1. Percentage failure modes of the tested specimens.

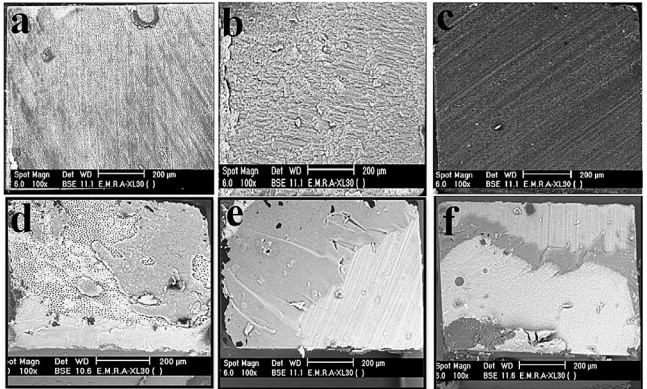


Figure 2. Representative scanning electron photomicrographs showing the predominant failure modes of Adper Single Bond 2, Clearfil SE Bond and Adper Easy One/coronal dentin fractured specimens (a, b and c, respectively). While (d, e and f) are the representative SEMs of the predominant failure modes of Adper Single Bond 2, Clearfil SE Bond and Adper Easy One/radicular dentin fractured specimens, respectively.

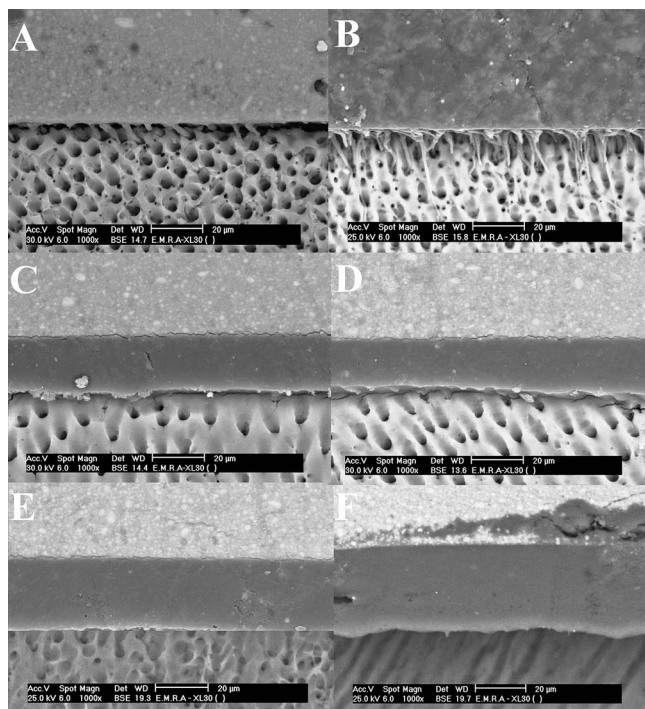


Figure 3. Representative scanning electron photomicrographs showing the bonded interfaces of Adper Single Bond 2, Clearfil SE Bond and Adper Easy One/coronal dentin specimens (A, C and E, respectively) and Adper Single Bond 2, Clearfil SE Bond and Adper Easy One/radicular dentin specimens (B, D and F), respectively.

showed the highest significant bond strength values in comparison to the other two adhesive systems, which in turn were comparable with no statistically significant difference.

The lower bond strength of the two-step etch-and-rinse adhesive (SB) compared to the two-step self-etch adhesive (SE) was in accordance with others^{2,9,21,22} despite that bonding was done in their studies to coronal dentin at different locations. Bonding to parallel-cut dentin implies that the numbers of resin tags, which have been postulated to contribute to 25% of the recorded bond strength values, are reduced.²³ Also, the smear layer removal with etch-and-rinse adhesive systems increases dentin hydraulic conductance allowing the outward flow of the fluid within the dentinal tubule to the surface of the dentin. This renders the etch-and-rinse adhesive systems to be very sensitive to IPP simulation. The debonded specimens of SB, showed predominately adhesive failure at the dentin side for both coronal and root dentin groups which support that IPP simulation caused excessive moisture on the adherent substrate.

Conversely, the self-etch adhesive system is expected to be less influenced by IPP simulation.

The etching effects of self-etch adhesives depend on the concentration and pH of their acidic monomers. The mild etching effect imposed by the self-etching primer (pH≈2.0) of SE results in residual mineral crystals within the hybrid layer and maintains smear plugs blocking the tubule orifices. This fact, combined with the use of a separate, relatively hydrophobic, solvent-free adhesive layer placed over the hydrophilic primer, significantly reduces the rate of fluid flow through the interface even in the presence of IPP.¹⁷ This was confirmed by Hashimoto and others²⁴ who reported that although the smear layer and smear plugs do not provide an impermeable or hermetic seal of the dentinal tubules, they account for up to 86% of the total resistance to fluid movement across dentin.²⁵ Another reason for the highest bond strength values of the two-step self-etch SE adhesive system could be due to the presence of an unsaturated methacrylate phosphate ester 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) as an acidic monomer together with HEMA which is believed to improve the wetting of SE adhesive to moist dentin.^{17,26} A molecule like 10-MDP has high affinity to chemically bond to the calcium in the hydroxyapatite which could have played a part in recording high bond strength.²⁷

To explain the inadequate performance of the single-step self-etch adhesive, compared with the two-step self-etch adhesive (SE), some major differences should be elicited. Single-step self-etch adhesives were found to contain high concentrations of HEMA which induces the formation of a HEMA-rich oxygen-inhibition layer that may enhance the osmotic process of water movement.²⁸ Moreover, they contain mainly hydrophilic monomers which may cause reduction in polymerization due to their dilution with water flow from the bonded dentin.²⁹ The intrinsic hydrophilicity renders them more sensitive to water contamination, even though they preserve the smear layer. Moreover, the dense distribution of polar hydrophilic domains within these adhesives increases sites for water binding and transport.³⁰

Mode of failure of the single-step self-etch adhesive specimens supported the microtensile bond strength results as they revealed a higher percentage of cohesive failure in the adhesive layer with both coronal and radicular dentin surfaces. As previously observed by Belli and others,³⁰ HEMA-containing single-step self-etch adhesives have shown clear evidences of water uptake and droplet accumulation at the adhesive/composite interface.

After one year of storage in saliva immersion at 37°C and under IPP simulation, interfacial bond strengths of all adhesive systems were significantly affected. Abdalla and others¹⁷ and El-Deeb and others⁸ reported a significant decrease in the two-step self-etch adhesive (SE) bond strength after six-month storage under IPP simulation. They referred this reduction to slow water sorption of the adhesive which could affect its mechanical properties and thus its bond strength. On the contrary, another study³⁰ reported no statistically significant difference between adhesive dentin bond strength values after one-year storage under IPP simulation. The application of simulated IPP in that study was restricted to the storage period while in the present study IPP was applied during the bonding procedure and over the storage period. Although bond strength of SE significantly decreased after storage, it maintained the highest value compared to the other adhesive systems. The formed calcium-phosphate salts along with only a limited surface-decalcification effect was referred to be more stable to hydrolytic degradation.²⁷

Present study findings emphasized that current bonding strategies could surpass circumstances of regional variability, dentin site, as well as intrinsic and extrinsic moisture that present in the oral cavity. However, improving adhesive systems to provide successful and durable restorations is still required.

CONCLUSIONS

Bond strengths of tested adhesives were not sensitive to structural differences between coronal and radicular dentin even after one-year storage and under IPP simulation. However, all tested adhesive systems were sensitive to storage.

Human Subjects Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies. The approval code for this study was 112/2011. This study was conducted at the Faculty of Oral and Dental Medicine, Cairo University.

Conflict of Interest

The authors of this manuscript 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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