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RESEARCH AND EDUCATION

Comparative evaluation of 3 microbond strength tests using 4 adhesive systems: Mechanical, finite element, and failure analysis

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ABSTRACT

Statement of problem. Bond strength (BS) values from in vitro studies are useful when dentists are selecting an adhesive system, but there is no ideal measuring method.

Purpose. The purpose of this in vitro study was to investigate the influence of the evaluation method in the BS between dentin and composite resin.

Material and methods. Molars with exposed superficial dentin (N=240) were divided into 3 groups according to the test: microtensile (μTBS), microshear (μSBS), and micropush-out (μPBS). Each one was subdivided into 4 groups according to the adhesive system: total etch, 3- and 2-step; and self-etch, 2- and 1-step). For the μPBS test, a conical cavity was prepared and restored with composite resin. An occlusal slice (1.5 mm in thickness) was obtained from each tooth. For the μSBS test, a composite resin cylinder (1 mm in diameter) was built on the dentin surface of each tooth. For the μTBS test, a 2-increment composite resin cylinder was built on the dentin surface, and beams with a sectional area of 0.5 mm^2 were obtained. Each subgroup was divided into 2 (n=10) as the specimens were tested after 7 days and 1 year of water storage. The specimens were submitted to load, and the failure recorded in units of megapascals. Original BS values from the μTBS and μSBS tests were normalized for the area from μPBS specimens. Original and normalized results were submitted to a 3-way ANOVA (α=.05). The correlation among mechanical results, stress distribution, and failure pattern was investigated.

Results. Significant differences ($P<.05$) were found among the adhesive systems and methods within both the original and normalized data but not between the storage times ($P>.05$). Within the 7 days of storage, the original BS values from μTBS were significantly higher ($P<.001$) than those from μPBS and μSBS. After 1 year, μSBS presented significantly lower results ($P<.001$). However, after the normalization for area, the BS values of the μTBS and μPBS tests were similar, and both were higher ($P<.001$) than that of μSBS in both storage times. In the μSBS and μTBS specimens, cohesive and adhesive failures were observed, whereas μPBS presented 100% of adhesive failures. The failure modes were compatible with the stress distribution.

Conclusions. The storage time did not affect the results, but differences were found among the adhesives and methods. For comparisons of bond strength from tests with different bonding areas, the normalization for area seemed essential. The microshear bond test should not be used for bond strength evaluation, and the microtensile test needs improvement to enable reliable results regarding stress concentration and failure mode. The micropush-out test may be considered more reliable than the microtensile in the bond strength investigation, as demonstrated by the uniform stress concentration and adhesive failure pattern. (J Prosthet Dent 2018;119:166-174)

Any restoration using tooth-colored materials requires stable adhesion to tooth substrate.[1] This is particularly challenging to dentin because of its high organic content, wet tubular microstructure, and the presence of a smear layer.[2-5] Dental adhesive systems attempt to provide higher and more durable bond strength (BS) values,[6-14]

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Clinical Implications

Providing more accurate bond strength results from methods which simulate clinical conditions more faithfully may help dentists make appropriate choices regarding the adhesive system to use.

but despite the high BS achieved, various factors may influence the clinical performance of the system.[3,15-24] Earlier tensile and shear test methods used large bonding areas, but higher BS and incidence of adhesive failures were observed for smaller areas.[11,25-30] However, studies using the microshear (μSBS) and microtensile (μTBS) methods demonstrated that BS values may depend on the method used, and thus the reported values should not be directly compared.[31-35] Although in vitro results are considered limited and do not necessarily reflect the clinical behavior of dental materials, they can indicate the performance of a material. Materials with higher BS may be considered the best choice for clinical use. However, different results from tests using different equipment for strength-based studies[36,37] combined with concerns that in vitro BS assessments are poor predictors of clinical success[32-34,38-41] have led to the development of new BS evaluation methods.[42-43]

The micropush-out (μPBS) test has been used to determine the BS of cemented endodontic posts but not often to evaluate the BS of composite resin to dentin.[44-48] The search for improved adhesives has led researchers to compare results from specimens with different bonded areas, but a reliable comparison would be matched after the normalization for area among the experimental groups.[30] Thus, the BS of 4 different adhesive systems was investigated using 3 evaluation methods after 2 periods of water storage, and original and normalized data were compared. Using mechanical tests, finite element analysis, and failure analysis, the purposes of this study were to compare analytically the BS among the adhesive systems and the influence of the method on the results; to determine the need for the normalization of area when the BS from specimens with different dimensions and cross-sections were compared; to evaluate the correlation between the stress distribution and the failure analysis as parameters to validate the analytical results; and to investigate the influence of water storage on BS. The null hypotheses were that the composition and handling of adhesive systems, the method of evaluation, and the water storage would not influence the BS between composite resin and dentin.

MATERIAL AND METHODS

After approval by the ethics committee of the Federal University of Uberlândia, 240 intact human third molars were extracted, cleaned, and stored in 0.2% thymol solution[47] at room temperature for up to 3 months.[49] The teeth were fixed in acrylic resin blocks, the occlusal third was cut under water cooling using a low-speed, 4-inch × 0.12-mm diamond saw (EXTEC) attached to a cutting machine (Isomet 1000; Buehler).[47] The teeth were divided into 3 groups (sample size, 80 per group) according to the BS tests: μPBS, μSBS, and μTBS. Each group was then subdivided into 4 groups according to the adhesive system (sample size, 20 per group): SBM: total-etch 3-step (Adper Scotchbond Multipurpose; 3M ESPE); SGB: total-etch 2-step (Adper Single Bond; 3M ESPE); ADH: self-etching 2-step (AdheSE; Ivoclar Vivadent AG); and PLP: self-etching 1-step (Adper Prompt L-Pop; 3M ESPE). The 20 specimens from each group were then divided into 2 subgroups according to the storage time (7 days and 12 months) in distilled water at room temperature.

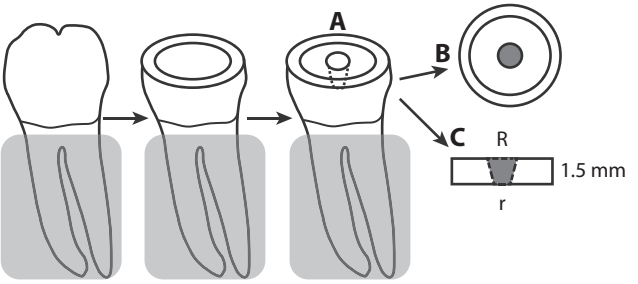

Figure 1. Schematic illustration of tooth preparation (A) and characteristics of specimen for micropush-out bond strength test: slice occlusal (B) and side (C) views. Radius: R=1.0 ±0.2 mm and r=0.8 ±0.2 mm.

Composite resin (Filtek Z350 XT; 3M ESPE) was bonded to all the specimens after dentin surface treatment as follows: 32% phosphoric acid etching for 15 seconds, air-water spray, and soft paper drying, followed by the adhesive application and polymerization. All materials were used according to the manufacturer's instructions and, when applicable, photopolymerized for 20 seconds at 1 mm from the surface using a 600-mW/cm^2 halogen light source (Optilight; Gnatus). Parameters used in the tests were Emic DL 2000 machine (Instron Brasil), 50-N compression load cell at a speed of 0.5 mm/min,[9] and BS values in megapascals.

For the μPBS test, a central and conical cavity with a depth of 2 mm was prepared in each tooth, using a high-speed, tapered, diamond rotary instrument (#703 KG; Sorensen) under water cooling. Each rotary instrument was used for 10 preparations. After dentin treatment, the cavities were restored in a single resin increment, and an occlusal slice approximately 2 mm thick was obtained from each tooth using a low-speed diamond saw under water cooling.[47] The thickness was standardized manually with wet 600-grit silicon carbide paper[9] to a final thickness of 1.5 ±0.2 mm, which was checked with digital

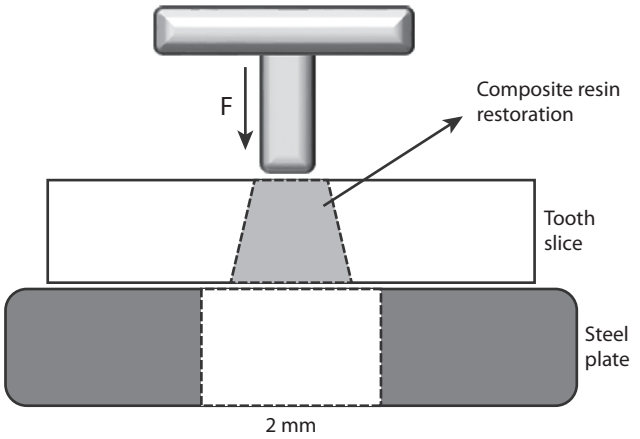

Figure 2. Schematic illustration for micropush-out bond strength testing.

calipers (Super Caliper, Series 500; Mitutoyo). The 1.0 ±0.2 mm of the larger radius (R) and the 0.8 ±0.2 mm of the smaller radius (r) were measured using stereomicroscopy at ×40 magnification (Fig. 1).

Specimens were positioned over a steel plate with a 2-mm central hole fixed on the testing machine with the smaller radius up and the load applied with a steel cylinder-shaped rod (diameter, 0.6 mm) with the specimens immersed in water during the test (Fig. 2). The μPBS values were obtained using the formula $A=\pi h(R+r)$,[50] where A is the bonding area (8.3 mm^2), π is the 3.14 constant, h is the thickness in millimeters, R is the larger radius, and r is the smaller radius (in millimeters).

For the μSBS test, a silicone cylinder (Fabrimed Comercial Ltda, Brazil) 1 mm in diameter and length (0.78 mm^2 of bonding area) was positioned on the treated dentin surface of each tooth and filled in 1 increment of composite resin. After polymerization, the silicone cylinder was sectioned with a surgical blade and removed, and the specimens were stored and then loaded (Fig. 3).

For the μTBS test, a 4-mm-high composite resin block was built in 3 increments on the dentin surface, and 8 central beams with approximately 0.5 mm^2 of bonding area[11] were obtained from each tooth/resin block and stored together. Each specimen was fixed with cyanoacrylate resin adhesive (Cola Universal; Loctite) in a device attached to the EMIC machine and submitted to tensile load (Fig. 4). The mean values from the 3 specimens that presented adhesive or mixed failures were considered for the analysis.

Original BS values recorded from the 3 mechanical tests were submitted to 3-way ANOVA (α=.05) and the Tukey honestly significant difference (HSD) test. To enable a reliable comparison among specimens with different cross-sectional areas, normalization for area is recommended.[30] The BS values from the μPBS groups were submitted to Weibull analysis[51] to calculate the

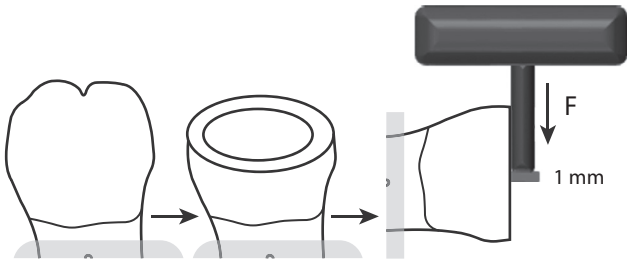

Figure 3. Schematic illustration for microshear bond strength specimen preparation and testing.

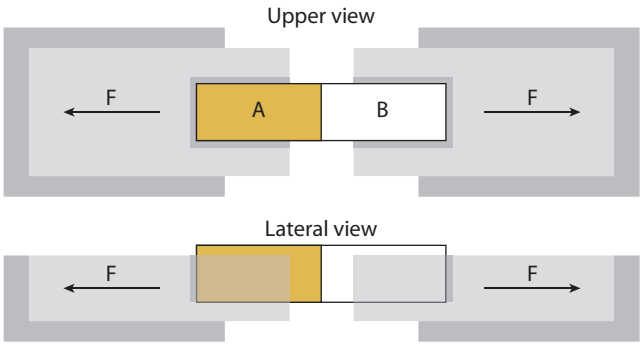

Figure 4. Schematic illustration for microtensile bond strength testing. Composite resin (A), Dentin (B).

Weibull modulus (m) at the 5% confidence level, which was used to calculate the normalized BS from the corresponding μTBS and μSBS groups. Thus, the normalized bond strength from the SBM microtensile and microshear groups were obtained using the m value from the SBM micropush-out group. The μTBS specimens' normalization for area to the μPBS was made using formula (1), and the μSBS specimens' normalization for area was made using formula (2), as follows[30,52]:

(1) $\sigma\mu_{Tn}=\sigma\mu_T\ (A\mu_T/A\mu_P)^{1/m}$, where $\sigma\mu_{Tn}$ is the μTBS strength value normalized for the μPBS specimen bonding area, $\sigma\mu_T$ is the original μTBS BS value, $A\mu_T$ is the bonding area from μTBS specimens (0.5 mm^2), $A\mu_P$ is the bonding area from μPBS specimens (8.3 mm^2), and m is the Weibull modulus from the respective μPBS group.

(2) $\tau\mu_{Sn}=\tau\mu_S\ (A\mu_S/A\mu_P)^{1/m}$, where $\tau\mu_{Sn}$ is the μSBS strength value normalized for the μPBS specimen bonding area, $\tau\mu_S$ is the original μSBS BS value, $A\mu_S$ is the bonding area from μSBS specimens (0.78 mm^2), $A\mu_P$ is the bonding area from μPBS specimens (8.3 mm^2), and m is the Weibull modulus from the respective μPBS group.

The normalized BS values were submitted to 3-way ANOVA (α=.05) and Tukey HSD test, and the tested specimens were evaluated using stereomicroscopy at ×40 to ×100 magnifications to determine the failure mode: adhesive (failure at bonding interface), mixed (failure at

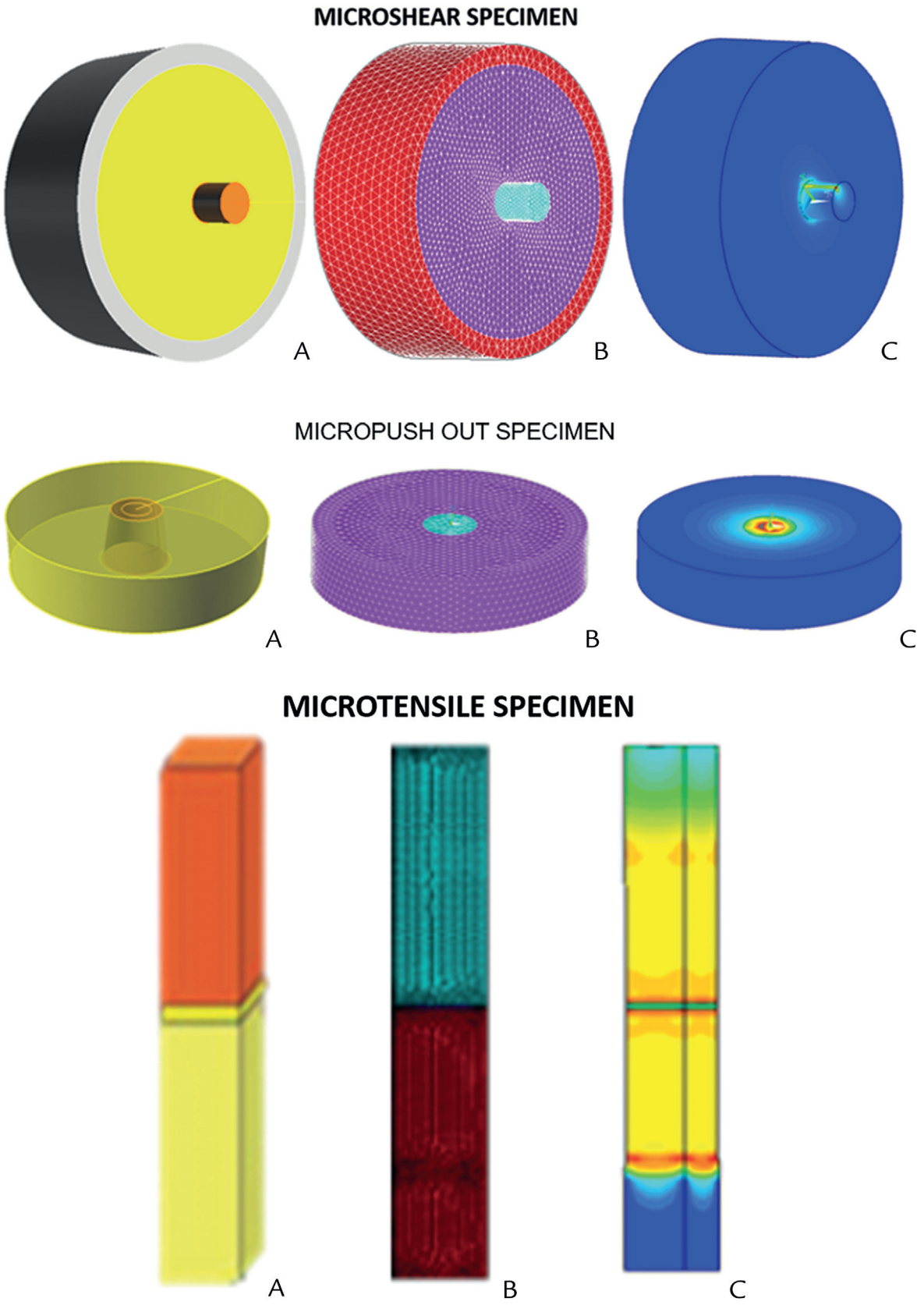

Figure 5. Three-dimensional finite element analysis from tested specimens: model (A), meshing (B), and corresponding stress distribution (C).

Table 1. Mechanical properties of structures used for finite element analysis

Structure/Material	Elastic Modulus (GPa)	Poisson Ratio
Dentin[54]	18.0	0.31
Composite resin[55]	16.6	0.24
Enamel[56]	46.8	0.30

bonding interface with fragments of dentin or composite resin), and cohesive (failure outside the bonding interface). Specimens with cohesive failures were considered for failure mode comparison only. Representative 3-dimensional (3D) finite element analysis models were developed by computer-aided design and computer-aided engineering (CAD-CAE) software association and constructed to simulate μSBS, μPBS, and μTBS specimens (Fig. 5). The geometry of each model was created by CAD software (Rhinoceros 3D 4.0; McNeel North America), simulating the dimensions of the tested specimens. The data obtained were exported to CAE software (Ansys v9.0; ANSYS). The models were meshed with a higher-order 3D 20-node structural solid element (SOLID186), which is defined by 20 nodes having 3 degrees of freedom per node: translations in the nodal *x*, *y*, and *z* directions.[53] Table 1 shows the mechanical properties of all structures,[54-56] which were considered elastic, linear, homogeneous, and isotropic. The boundary conditions (static pressure and displacement restriction) of each model were simulated according to experimental tests. The qualitative stress distribution analyses were recorded using von Mises and Sy criteria.[47]

RESULTS

After exploratory analysis using the PROLAB/SAS (SAS Institute Inc) statistical program and the square root transformation, the original BS values were submitted to 3-way ANOVA factorial analysis (adhesive × storage time × evaluation method) and Tukey HSD test (α=.05). The results are shown in Table 2. The storage time did not influence the results (P=.634), but the interactions adhesive × evaluation method (P<.001) and adhesive × method × storage time showed significant differences (P=.003). Significant differences were observed among adhesives and evaluation method within each storage time. When adhesives were compared, significant lower BS values (P<.001) were detected for the μSBS groups in both storage times. For the evaluation methods, significantly higher values (P<.001) were observed for the μTBS groups in the 7-day storage time, except when they were compared with ADH system from the μPBS specimens. After 1 year of storage, significantly higher values

Table 2. Results of original bond strength analysis

		Method					
Storage Time	Adhesive	μPBS	CV (%)	μSBS	CV (%)	μTBS	CV (%)
7 d	SGB	12.90 ±4.01^{Ba}	31	11.20 ±6.05^{Bab}	54	32.00 ±15.06^{Aa}	47
	SBM	15.80 ±3.61^{Ba}	23	19.60 ±10.45^{Ba}	53	40.60 ±13.33^{Aa}	33
	PLP	15.10 ±5.47^{Ba}	36	10.90 ±7.82^{Bab}	72	34.40 ±18.57^{Aa}	54
	ADH	25.20 ±9.65^{Aa}	38	7.70 ±3.89^{Bb}	51	48.20 ±13.08^{Aa}	27
12 mo	SGB	15.00 ±4.08^{Aa}	27	7.10 ±4.23^{Bb}	60	29.40 ±14.02^{Aa}	48
	SBM	20.60 ±5.21^{ABa}	25	14.90 ±5.17^{Ba}	35	37.60 ±12.71^{Aa}	34
	PLP	15.10 ±3.96^{Aa}	26	7.50 ±4.90^{Bb}	65	29.40 ±15.14^{Aa}	51
	ADH	20.90 ±6.44^{ABa}	31	12.30 ±7.10^{Bab}	58	45.00 ±18.60^{Aa}	41

μPBS, micropush-out test; μSBS, microshear test; μTBS, microtensile test; ADH, self-etching 2-step (AdheSE; Ivoclar Vivadent AG); CV, coefficient of variation; PLP, self-etching 1-step (Adper Prompt L-Pop; 3M ESPE); SBM, total-etch 3-step (Adper Scotchbond Multipurpose; 3M ESPE); SGB, total-etch 2-step (Adper Single Bond; 3M ESPE). Data show original mean ±SD bond strength and CV (%) values. Values in MPa followed by different letters (uppercase in rows comparing method and lowercase in columns comparing adhesives within method) indicate significant differences (P<.05).

Table 3. Results of normalized bond strength analysis

		Method								
Storage Time	Adhesive	μPBS	CV	Rk	μSBS	CV	Rk	μTBS	CV	Rk
7 d	SGB	12.90 ±4.01^{Ab}	31	3rd	5.80 ±3.16^{Bab}	54	2nd	14.60 ±7.49^{Aab}	51	3rd
	SBM	15.80 ±3.61^{ABab}	23	2nd	12.30 ±6.65^{Ba}	54	1st	23.10 ±7.75^{Aa}	34	1st
	PLP	15.10 ±5.47^{Aab}	36	2nd	4.80 ±3.36^{Bb}	70	3rd	12.90 ±6.92^{Ab}	54	2nd
	ADH	25.20 ±9.65^{Aa}	38	1st	3.20 ±1.62^{Bb}	51	3rd	17.30 ±4.64^{Aab}	27	3rd
12 mo	SGB	15.00 ±4.08^{Aa}	27	1st	3.60 ±1.96^{Ba}	54	1st	12.60 ±5.78^{Aa}	46	1st
	SBM	20.60 ±5.21^{Aa}	25	1st	8.60 ±3.17^{Ba}	37	1st	19.70 ±7.00^{Aa}	36	1st
	PLP	15.10 ±3.96^{Aa}	26	1st	4.30 ±3.06^{Ba}	71	1st	15.80 ±7.83^{Aa}	50	1st
	ADH	20.90 ±6.44^{Aa}	31	1st	6.00 ±3.43^{Ba}	57	1st	19.80 ±8.1^{8Aa}	41	1st

μPBS, micropush-out test; μSBS, microshear test; μTBS, microtensile test; ADH, self-etching 2-step (AdheSE; Ivoclar Vivadent AG); CV, coefficient of variation; PLP, self-etching 1-step (Adper Prompt L-Pop; 3M ESPE); Rk, ranking of adhesives by multiple comparisons test; SBM, total-etch 3-step (Adper Scotchbond Multipurpose; 3M ESPE); SGB, total-etch 2-step (Adper Single Bond; 3M ESPE). Data show mean ±SD and CV. Values in MPa followed by different letters (uppercase in rows comparing method and lowercase in columns comparing adhesives within method) indicate significant differences (P<.05).

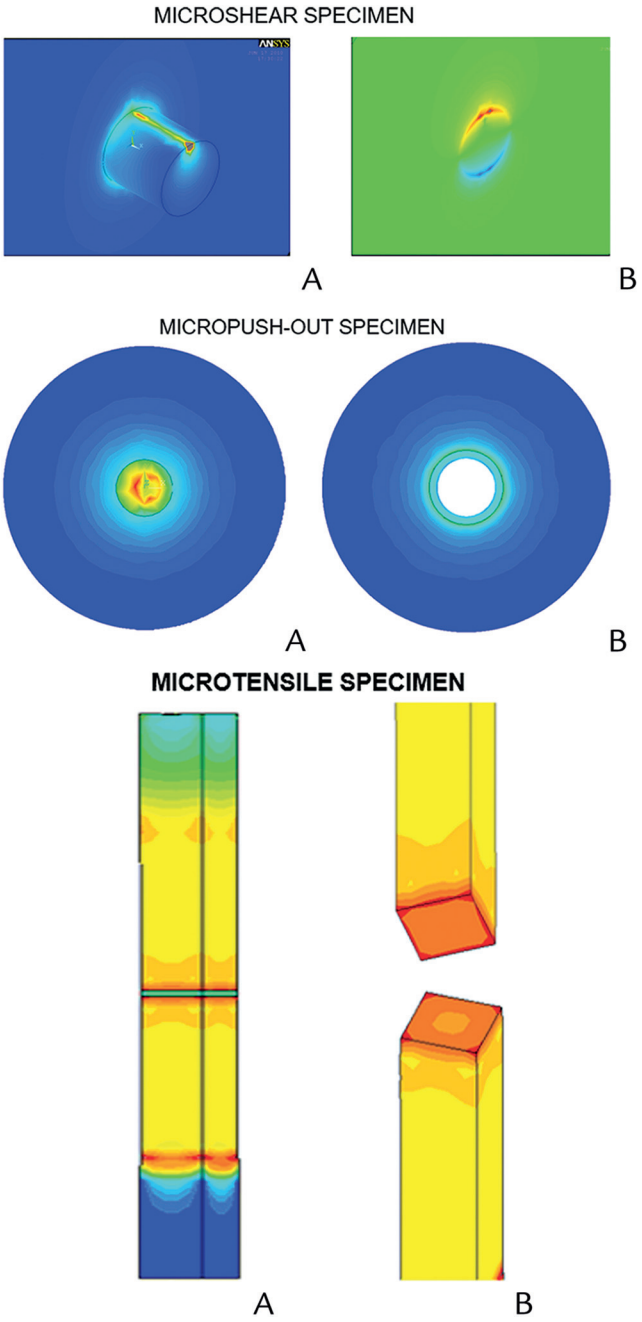

Figure 6. Qualitative stress distribution in dentin and resin substrate from tested specimens: von Mises (A) and Sy criteria (B). Sy analysis indicated for microshear specimen: areas of tensile (red) and compression (blue) stress; for micropush-out specimen: shear stress in bonding area (light green); for microtensile specimen: high tensile stress in bonding area and extending outside (yellow).

(*P*<.001) were found for the μTBS in relation to those of the μSBS specimens. The coefficient of variation from the μPBS test was lower than that of the others in both storage times.

Mean BS values from the normalized data were log transformed and submitted to ANOVA/Tukey HSD tests, and the results, including the ranking of the adhesives by the multiple comparisons test, are shown in Table 3. No significant differences were found for the storage time (*P*=.999). Significant differences (*P*=.024) among the adhesive systems were detected within each method for the 7-day storage time. Regarding the methods, results from the μTBS and μPBS groups were similar and significantly higher than those from the μSBS groups in both storage times (*P*<.001). The coefficient of variation was lower for the μPBS, intermediate for the μTBS, and higher for the μSBS.

Finite element analysis results are shown in Figure 6. The von Mises analysis showed stress concentration at the load point in the resin substrate from μPBS and μSBS groups and along the μTBS specimens but did not identify the stress distribution in the bonding area. The Sy analysis showed distinct areas of tensile and compression stress in the μSBS dentin substrate. In the μTBS specimens, areas of high tensile stress were observed in the bonding area and extended into both the dentin and the resin substrate. In the μPBS specimens, the shear stress seemed to be uniformly distributed in the bonding area.

The stereomicroscopy analysis of the μPBS specimens showed 100% adhesive failures, and some specimens showed microchippings in the cavity angle. Adhesive (70%) and mixed failures (30%) were observed for the μSBS specimens. For the μTBS specimens, 78% of the failures were adhesive, and 22% were mixed. Nearly 25% of all μTBS specimens failed prematurely during specimen preparation and testing, and 30% presented cohesive failure. Scanning electron microscopy images from μPBS representative specimens after 1 year of water storage are shown in Figure 7.

DISCUSSION

Current adhesive systems yield high BS values to dentin. However, in vitro results do not seem to comply with clinical testing. In vitro results can vary depending on the adhesive system composition and manipulation and the evaluation method, and long-term BS may be affected by water.[15,21,23,24,31] An adequate evaluation method should consist of high BS values, uniform stress distribution, and adhesive failures. As there is no ideal in vitro measuring method, the current study investigated and compared 3 tests: μTBS, μSBS, and μPBS. The approximate bonding area from the μTBS specimens was 0.5 mm^2, the μSBS was 0.78 mm^2, and the μPBS was 8.3 mm^2. As there is an inverse relationship between the BS and the bonded area,[11] comparisons of results obtained from specimens with different bonding areas would not be appropriate and might lead to misunderstandings. Thus, in order to enable a suitable comparison, the bonding areas from the μTBS and μSBS specimens were normalized for the μPBS

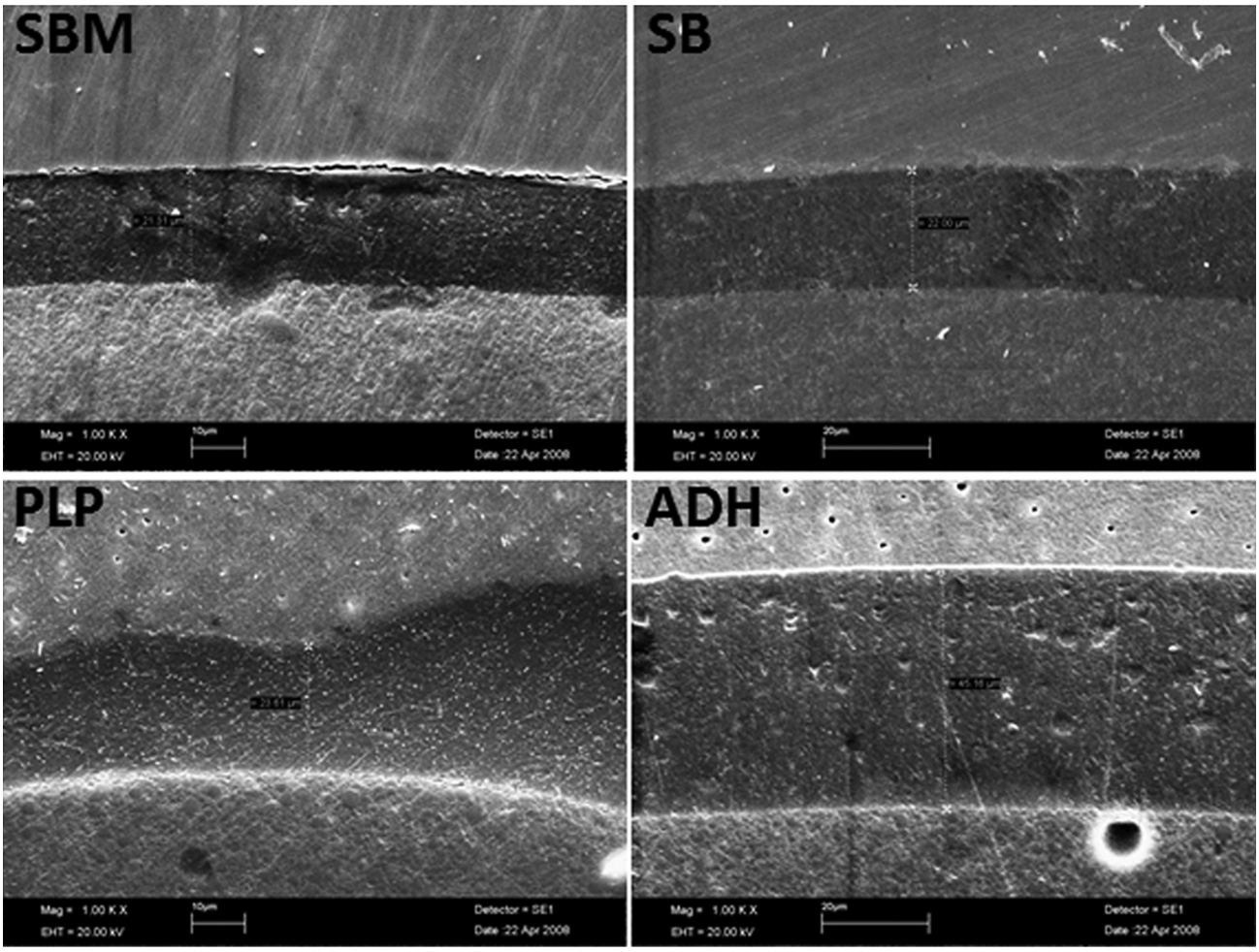

Figure 7. Scanning electron microscopy images from untested micropush-out bond strength specimens showing bonding interface after 1-year water storage. ADH, Adhese; PLP, Prompt L-Pop; SB, single bond; SBM, Scotchbond multipurpose. (Original magnification ×1000).

specimen area. Hence, all the bonding areas were similar, and the discussion is based on the data from the normalization for area (Table 3). The null hypotheses for the influence of the adhesive system and evaluation method on BS were rejected because significant differences were found for both. The null hypothesis for water storage was partially accepted as no differences were detected in the period of 1 year.

The improved BS from microtests has been attributed to the small bonding areas with fewer structural defects to initiate failure.[11,25,36] In the present study, comparisons among original results from different bonding areas (Table 2) showed significantly higher BS values for the μTBS test. However, after normalization for area and despite its higher bonding area, the BS from the μPBS test was similar to that of μTBS and significantly higher than that of μSBS. The coefficient of variation from the μPBS was lower than those of the μTBS and μSBS tests, and the μPBS specimen preparation was easier and closer to a clinical situation.

Significant differences in BS among the adhesive systems within each method were detected for the 7-day storage time (Table 3) and, as supported by other studies, may be explained by their different composition, bonding approaches, and adhesion mechanisms.[3,13,15,23,24] Although other studies reported significant decreases in BS after relatively short periods,[7,8,11] the 1-year storage time from the present study was probably not long enough to allow deterioration, or the deterioration occurred at a level that did not affect the results.

The μPBS test materials were placed in a class-I-shaped cavity with high cavity configuration (*C*-factor), therefore additional polymerization stress in the bonding interface[7] would be expected, thus decreasing the BS and making the bonding more vulnerable to water degradation.[22] Nevertheless, differences in BS were not observed after 1 year of water storage. Probably, the small amount of composite resin was not able to induce significant stress in the interfacial bonding. The adequate storage time and its effects on bond durability needs further investigation.

The adhesive ranking presented in Table 3 was intended to help readers in their choice, but the variability with the method in the 7-day storage time and the similarity in the 1-year storage time demonstrate a lack of reliable evidence for the decision. The decision should also consider results from clinical studies and operators misusing that might select materials with poor bonding.

Stress analysis predicts the stress distribution in the tested specimens according to their properties and the way the load is applied. The Sy stress analysis indicated different stress distribution among the evaluation methods and showed a correlation with the failure patterns. In the μPBS test, the shear stress distribution was uniform in the bonding area (Fig. 6), and all failures were adhesive[42] by displacement of the restoration from the cavity. In the μTBS specimens, a high level of tensile stress was concentrated outside the bonding area thus resulting in cohesive failures. In the μSBS specimens, tensile stress on the load side and compression on the opposite side resulted in adhesive and mixed failures. Although specimens with mixed and cohesive failures were not representative of a mechanism compatible with the load applied, the BS results from μSBS mixed failures were included in the analytical comparison and served to show the influence of the method on the evaluation and for comparison with the other tests.

Associating mechanical, finite element, and failure analyses indicated that the μSBS test should not be used to investigate interfacial BS and the μTBS test needs improvements. Despite different bonding areas, the normalization of area showed similar BS between μPBS and μTBS, in agreement with another study in which the BS increased with the increase in bonding area.[48]

Overall, besides presenting BS values similar to those of the μTBS test, the results provided by the μPBS test such as domain, coefficient of variation, stress distribution, failure pattern, and closer simulation of clinical conditions seemed more reliable than the other methods. However, different from the μTBS test, which allows multiple specimens, the pilot study using the μPBS test showed premature dentin fracture in the specimens with 2 or more cavities.

Results from the current study should be interpreted carefully. Comparison with other studies would not be appropriate, and the study's following limitations should be considered: neither mechanical nor thermal stressing was simulated in the bonding area; the specimens were submitted to static load; storage was at room temperature and the storage time seemed too short to affect the bond durability; and the teeth were obtained from individuals of different ages and oral cavity conditions, resulting in differences inherent to the dentin from each specimen. Testing dentin with identical characteristics is so far impossible and the search for a valid in vitro test seems endless. Future studies aiming to diminish operator influence and using a protocol that provides only adhesive failures could define the best method.

CONCLUSIONS

Within the limitations of this in vitro testing, the following conclusions were drawn:

1. Storage time did not affect the results, but differences were found among the adhesives and methods.
2. Normalization for area seemed essential in the comparison among specimens with different bonding areas.
3. Finite element and failure analysis indicated that the μSBS test should not be used for BS evaluation.
4. The μTBS test needs improvement to enable reliable results regarding stress concentration and failure pattern.
5. The μPBS test may be considered more reliable than the μTBS test in the BS investigation, as demonstrated by the uniform stress concentration and adhesive failure pattern.

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