

Evaluating resin-enamel bonds by microshear and microtensile bond strength tests: effects of composite resin

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ABSTRACT

Objectives: The aims of this study were to evaluate the effect of resin composite (Filtek Z250 and Filtek Flow Z350) and adhesive system [(Solobond Plus, Futurabond NR (VOCO) and Adper Single Bond (3M ESPE)] on the microtensile (μ TBS) and microshear bond strength (μ SBS) tests on enamel, and to correlate the bond strength means between them. **Material and Methods:** Thirty-six extracted human molars were sectioned to obtain two tooth halves: one for μ TBS and the other one for μ SBS. Adhesive systems and resin composites were applied to the enamel ground surfaces and light-cured. After storage (37°C/24 h) specimens were stressed (0.5 mm/min). Fracture modes were analyzed under scanning electron microscopy. The data were analyzed using two-way ANOVA and Tukey's test ($\alpha=0.05$). **Results:** The correlation between tests was estimated with Pearson's product-moment correlation statistics ($\alpha=0.05$). For both tests only the main factor resin composite was statistically significant ($p<0.05$). The correlation test detected a positive ($r=0.91$) and significant ($p=0.01$) correlation between the tests. **Conclusions:** The results were more influenced by the resin type than by the adhesives. Both microbond tests seem to be positive and linearly correlated and can therefore lead to similar conclusions.

Key words: Enamel. Adhesives. Resins.

INTRODUCTION

Various conventional mechanical test methods, such as shear, tensile, and flexural tests, have been used to assess dental adhesion. Tensile and shear tests are the most commonly used¹. However, problems related to the validity of the measurements obtained began to arise as cohesive failures in the substrate were frequently observed with new adhesives that yielded improved bond strengths. According to some researchers³⁰, the explanation for this fact was that stresses were mostly concentrated in the substrate, thus causing it to fail prematurely, before failure at the interface itself. Another point that has drawn fundamental criticism concerns the non-uniform nature of stress distributions along tested interfaces⁷.

To improve stress distribution and the range of

bond strength values, shear and tensile tests were almost completely replaced by the microtensile bond strength (μ TBS) test, introduced by Sano, et al.²¹ (1994). The main characteristic of this test is the reduced specimen size, which provides a higher bond strength mean²². More recently, some authors^{25,26} have advocated a new test method using specimens with reduced dimensions, as a substitute for the conventional shear test: the so-called "microshear" bond strength (μ SBS) test.

A better stress distribution can be accomplished in smaller specimens, tested either under μ TBS or μ SBS testing, since the number of voids and stress-raising factors is lower than the ones that possibly occur in larger areas, such those used for shear or tensile bond strength tests¹⁵. The μ SBS test would allow small areas to be tested, and it has the same advantages as the μ TBS, without the need

for sectioning procedures to obtain specimens, as these laboratory procedures themselves may induce early micro-cracking within the specimen^{8,20,25-27}. Whether or not this has any effect on the overall conclusions reached, these methods deserve further investigations, since few studies have compared them^{12,16}.

The data from bond strength studies may depend largely on experimental factors such as the type of composite, stress rate, sample size and geometry, and the actual test method^{16,18,29}. With regard to resin composite, some authors use flowable^{10,17,23} instead of microhybrid composites^{6,10} to fill the Tygon tube in μ SBS testing due to their easy placement. However, to the best of our knowledge no study has so far addressed the effects of this variation in method on the number of cohesive failures and bond strength values. Therefore, the objective of the present investigation was two-fold: 1) to evaluate the effect of the type of resin composite and adhesive system on the bond strength values obtained both in μ SBS and μ TBS tests; and 2) to correlate the bond strength means gathered from these mechanical tests to verify whether they are able to reach similar conclusions.

MATERIAL AND METHODS

Thirty-six extracted, caries-free human molars were used in this study. The teeth were collected after obtaining the patients' informed consent. The Research Ethics Committee of the University of São Paulo reviewed and approved this study under Protocol Number 193/06. Teeth were disinfected²⁴, cleaned by removing all debris and calculus, and stored in distilled water for 48 h. The teeth were decoronated and the crowns were sectioned transversally in a buccolingual direction, using a diamond disc at slow speed (Isomet, Buehler, Lake Bluff, IL, USA) in order to obtain two tooth halves. One half was used for the μ TBS test and the other half was used for the μ SBS test. The enamel surfaces were ground on wet # 180 and 600 Si-C papers (60 s).

A three-step etch-and-rinse system Solobond Plus (VOCO, Cuxhaven, LS, DEU), a two-step etch-and-rinse system Adper Single Bond 2 (3M ESPE, St. Paul, MN, USA) and a one-step self-etch adhesive Futurabond NR (VOCO, Cuxhaven, LS, DEU) were used in the present investigation together with the microhybrid composite Filtek Z250 (3M ESPE) and the flowable composite Filtek Flow Z350 (3M ESPE). The composition, application mode and batch number of each material are shown in Figure 1.

Microtensile bond strength test

Thirty-six tooth halves were randomly divided into six groups of equal size according to the

combination of the main factors Adhesive and Composite. After conditioning procedures, adhesives were applied and light polymerized using a quartz-tungsten halogen Optilux 501 unit (Kerr Corp., Orange, CA, USA) set at 600 mW/cm². Resin composite build-ups were constructed in two 20 mm-thick increments, which were individually light polymerized for 40 s. All bonding procedures were carried out by a single operator at a room temperature of 24°C. For placement of Filtek Flow Z350, an individual matrix was placed around the enamel slices.

After storage in distilled water (37°C/24 h), the specimens were sectioned longitudinally perpendicular to the adhesive interface by means of a diamond saw in an ISOMET machine, producing 0.9-mm-thick rectangular slabs. Next, each slab was visually analyzed to select the areas with the flattest interfaces and individually cut into sticks with cross sectional areas of about 0.8 mm. The cross-sectional area of each specimen was measured with a digital caliper (Absolute Digimatic, Mitutoyo, Tokyo, TYO, JPN) to the nearest 0.01 mm and recorded for measuring the bond strength. Cyanoacrylate adhesive (Super Bonder Gel, Loctite, Henkel Ltda, São Paulo, SP, BRA) was used to fix the specimens to a microtensile testing device used in a universal testing machine (Instron model 5565, Canton, MA, USA) at 0.5 mm/min. The number of prematurely debonded sticks *per* tooth was recorded.

Microshear bond strength test

The other 36 tooth halves were randomly divided into six groups of equal size according to the combination of the main factors Adhesive and Composite. After enamel conditioning, the adhesives were applied and light polymerized using the Optilux 501 unit, set at 600 mW/cm².

A plastic tube 54-HL (TYGON® Medical Tubing, Saint Gobain, Akron, OH, USA) with an approximate internal diameter of 0.7 mm and height of 0.4 mm was placed on the enamel surface. Resin composite was injected into the tube; a clear cellophane sheet was placed over the resin composite, pressed gently and photo-irradiated (40 s/600 mW/cm²).

Specimens were then stored in distilled water (37°C/24 h). The plastic tubes were removed with a sharp blade, and the specimens were checked with a light stereomicroscope at 10× magnification to discard any with any evident air bubbles or gaps at the interface. After this, the specimens were fixed to a modified testing device²⁶ with a cyanoacrylate adhesive and placed in the Instron testing machine for μ SBS testing. A 0.20-mm-diameter stainless steel orthodontic wire (Morelli Ortodontia, São Paulo, SP, BRA) was looped around the resin cylinder, making contact through half

Figure 1- Composition, mode of application and batch number of the adhesives used

Adhesive systems	Composition	Application mode	Batch number
Adper Single Bond 2 (3M ESPE)	1. Scotchbond – 35% phosphoric acid 2. Adhesive – Bis-GMA, HEMA, dimethacrylates, polyalkenoic acid copolymer, initiators, water and ethanol	a, b, c, d, e, h	7KK
SoloBond Plus (VOCO)	1. Etching agent – Vococid 36% phosphoric acid 2. Primer - Maleic acid, hydrophilic methacrylates, polyfunctional monomers, acetone, water 3. Bonding agent - HEMA, polyfunctional monomers	a1, b1, c, d1, e, f, i	621667
Futurabond NR (VOCO)	1. BIS-GMA, HEMA, phosphate methacrylates, BHT, ethanol, fluorides and organic acids	f1, f2, g, h	
Filtek Z250 (3M ESPE)	1. Bis-GMA, UDMA and Bis-EMA, filler. Additional contents: stabilizers, catalysts and pigments. Particle size (average diameter: 0.6[0.01-3.5]) and approximately 83% wt filler load	±2 mm light cured for 40 s at 600 mW/cm ²	7WN
Filtek Flow Z350 (3M ESPE)	2. Methacrylate resin monomers Bis-GMA, TEGDMA and Bis-EMA; dimethacrylate polymer; silica (75 nm) and zirconia (5-10 nm) nanofiller; approximately 65% wt filler load.	±2 mm light cured for 40 s at 600 mW/cm ²	6AR

a- acid-etching (15 s); a1- acid-etching (30 s); b- rinsing (15 s); b1- rinsing (20 s); c- air-drying (30 s); d- two coats of adhesive were lightly applied (15 s); d1- one coat of primer was lightly applied(30 s); e- air-dry for 10 s at 20 cm; f- one coat of adhesive was applied (15 s); f1- mix one drop of liquid A and one drop of liquid B(5 s); f2- one coat of adhesive was lightly applied (20 s); g- air dry for s at 20 cm; h- light-polymerization (10 s - 600 mW/cm²); i- light polymerization (20 s -600 mW/cm²)
Bis-GMA: bisphenol A diglycidyl methacrylate; HEMA: 2-hydroxyethyl methacrylate; BHT: butylated hydroxy toluene; UDMA: urethane dimethacrylate; TEGDMA: Triethylene glycol dimethacrylate; Bis-EMA: Bisphenol A polyethylene glycol diether dimethacrylate.

of its circumference, and was gently held flush against the resin-enamel interface. A shear force was applied to each specimen at a crosshead speed of 0.5 mm/min until failure occurred. All fractured specimens after μ TBS and μ SBS tests were mounted on aluminum stubs, sputter-coated and observed under a scanning electron microscope (JEOL 5600 LVj, JEOL Ltd., Tokyo, TYO, JPN). The bond failure modes were evaluated and classified as one of three types: mixed (adhesive + cohesive failure of the neighboring substrates), cohesive (failure exclusively within enamel or resin composite) and adhesive (failure exclusively in adhesive interface).

Analysis of etching pattern under SEM

The effect of conditioning with 35% phosphoric acid and self-etching adhesive on the ground enamel surface was observed. Teeth (n=2 for each treatment) were cut into two halves longitudinally in a mesial/distal direction. A deep lingual slit was prepared with a diamond bur to facilitate subsequent fracture of the etched surfaces. Phosphoric acid-etched enamel was rinsed with water spray for 15 s. Enamel etched with self-etch adhesive was rinsed

with ethanol and acetone to remove the monomers. After that, the same specimens were fractured to provide a sagittal view of the etched enamel. After 12 h in a desiccator, they were mounted on aluminum stubs for SEM analysis.

Statistical analysis

The mean bond strength of all specimens from the same hemi-tooth was averaged for statistical purposes. The cohesive failures and the pre-testing failures were not included in the statistical analysis. The bond strength mean for every tested group was expressed as the average of the six teeth used per group. The data from μ TBS and μ SBS were subjected to a two-way analysis of variance (Adhesive vs. Composite Resin) and Tukey’s test for pair-wise comparison ($\alpha=0.05$).

The correlation between the two bond strength test methods was analyzed by simple linear regression. The strength of the association between these two properties was estimated with Pearson’s product-moment correlation statistics ($\alpha=0.05$). The correlation was tested in two different ways. First, the mean bond strength value of each tooth

half was paired (total of 36 means) and the values were tested by the Pearson correlation test at $\alpha=0.05$. In the second approach only the overall means for each group were paired and tested using the same test (total of 6 means).

RESULTS

The fracture pattern mode (%) and the number of pre-testing failures observed in this experiment are shown in Table 1. The overall number of cohesive failures was higher with Filtek Flow Z350

(~60%) when compared with Filtek Z250 (~48%). A higher overall number of pre-testing failures was obtained with the μ TBS test (14.2% or 68 out of 484) when compared to the μ SBS test (4.4% or 4 out of 93). Representative figures of each fracture mode observed in the present investigation for each test can be seen in Figure 2.

The mean cross-sectional area of the μ TBS specimens ranged from 0.67 to 0.75 mm², with a mean value of 0.71 ± 0.1 mm², while the bonding area of the μ SBS specimens was 0.44 mm². For the μ TBS, two-way ANOVA detected that the

Table 1- Fracture pattern mode (%) and percentage of pre-testing failures according to each experimental condition

MICROTENSILE BOND STRENGTH						
Composite resin	Adhesive system	Cohesive in resin	Cohesive in enamel	Mixed	Adhesive	Pre-testing failures
Filtek Z250	Solobond Plus	46.8 (36)	2.6 (2)	44.2 (34)	1.3 (1)	5.2 (4)
	Single Bond	41.8 (33)	6.3 (5)	35.4 (28)	0 (0)	16.5 (12)
	Futurabond NR	49.4 (39)	11.4 (9)	29.1 (23)	1.3 (1)	8.9 (7)
Filtek Flow Z350	Solobond Plus	62.7 (47)	1.1 (1)	23.2(20)	0 (0)	20.9 (18)
	Single Bond	54.1 (46)	2.4 (2)	17.7 (15)	0 (0)	25.9 (22)
	Futurabond NR	55.1 (49)	0 (0)	25.8 (23)	1.1 (1)	18 (16)

MICROSHEAR BOND STRENGTH						
Composite resin	Adhesive system	Cohesive in resin	Cohesive in enamel	Mixed	Adhesive	Pre-testing failures
Filtek Z250	Solobond Plus	71.4 (10)	0 (0)	28.6 (4)	0 (0)	0 (0)
	Single Bond	40 (6)	0 (0)	60 (9)	0 (0)	0 (0)
	Futurabond NR	31.3 (5)	0 (0)	62.5 (10)	0 (0)	6.25 (1)
Filtek Flow Z350	Solobond Plus	70.6 (12)	0 (0)	23.5 (4)	0 (0)	5.9 (1)
	Single Bond	45.5 (5)	0 (0)	45.5 (5)	0 (0)	9 (1)
	Futurabond NR	50 (10)	0 (0)	45 (9)	0 (0)	5 (1)

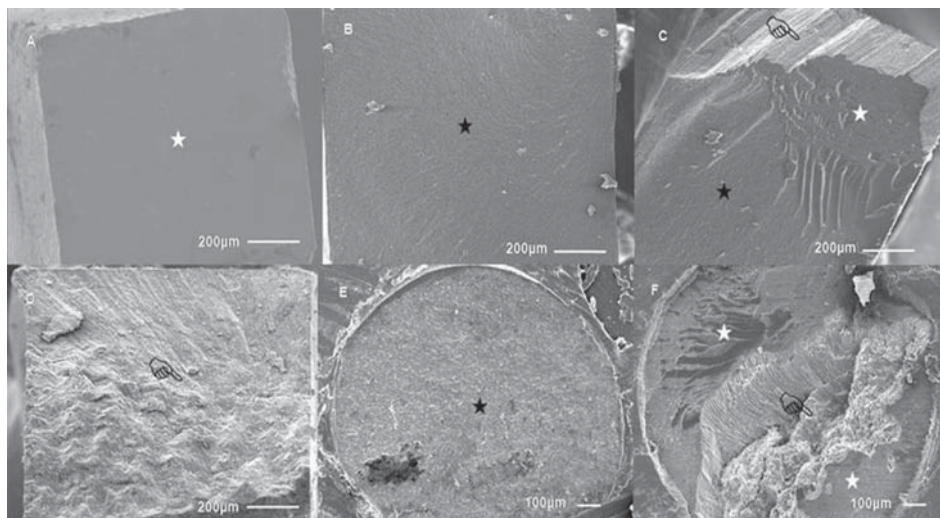


Figure 2- Representative figures of the failure modes observed in the microtensile (200x) (A-D) and microshear (140x) (E-F) tests. In (A), one can see an adhesive fracture pattern (white star). (B) and (E) represents cohesive resin failure. Black star shows resin composite in the same surface of both fractured specimens. (C) and (F) are mixed failures. Enamel is represented by the pointer, adhesive by the white star and resin composite by the black star. (C) represents a cohesive failure in enamel

Cross-product interaction ($p=0.44$) as well as the main factor Adhesive system ($p=0.58$) were not statistically significant. Only the main factor resin composite was statistically significant ($p=0.00001$). The means and standard deviations (MPa) of this test are shown in Table 2. It can be observed that higher μ TBS values were obtained with the microhybrid Filtek Z250 composite. With regard to the μ SBS, only the main factor resin composite ($p=0.0001$) was significant and the means and standard deviations (MPa) can be seen in Table 2.

Similarly to the μ TBS test, higher bond strength values were observed with Filtek Z250.

Pearson's correlation test detected a positive ($r=0.9106$) and significant ($p=0.0116$) correlation between the two bond strength tests, only when the means of each experimental group were paired. When the bond strength values from the same tooth were paired, the correlation was weak ($r=0.4749$), however highly significant ($p=0.003$) (Figure 3).

SEM micrographs of the etching pattern produced by 35% phosphoric acid and by the self-etch

Table 2- Microtensile (μ TBS) and microshear bond strength (μ SBS) values and, respective standard deviations (MPa)

Adhesive systems	Resin composite (*)	μ TBS	μ SBS
Solobond Plus	Filtek Z250 (A)	34.7±2.4	26.9±6.6
Single Bond		33.2±9.8	33.9±4.2
Futurabond NR		33.6±7.1	27.3±6.8
Solobond Plus	Filtek Flow Z350 (B)	22.4±5.3	15.6±2.2
Single Bond		23.4±5.3	14.6±4.6
Futurabond NR		28.0±6.3	20.5±4.0

(*) Groups with different letters are statistically different for tests (Tukey's test, $p<0.05$).

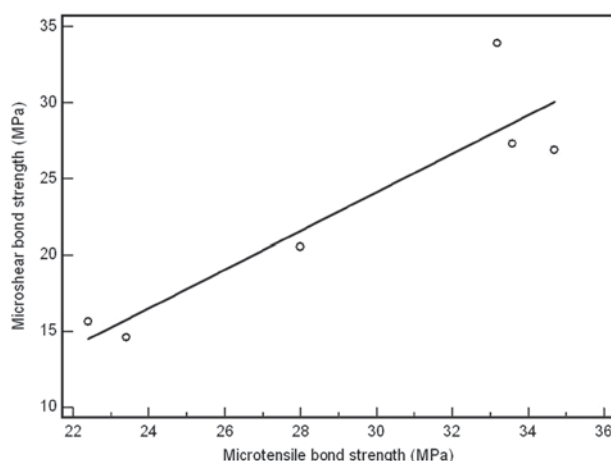


Figure 3- Linear relationship between microshear (MPa) and microtensile bond strength means (MPa)

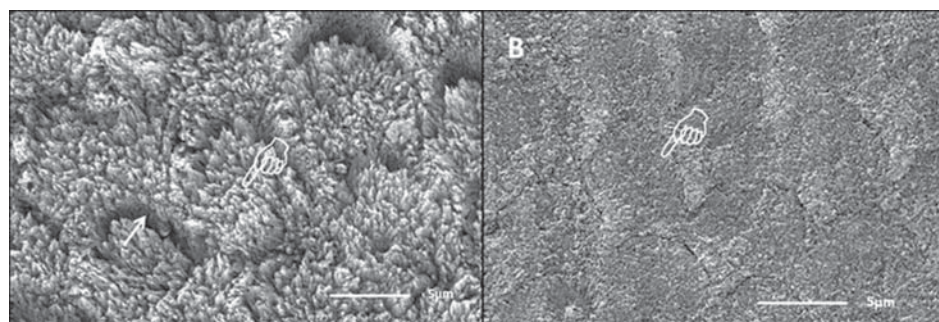


Figure 4- Scanning eletron microscopy micrographs of the 35% phosphoric acid etching (A) and the self-etch Futurabond NR (B) patterns. Selective demineralization of enamel prism periphery can be observed (white arrow, type 2 pattern). Prism cores were also superficially demineralized increasing the bonding area (pointer). (B) Demineralization created by self-etch adhesive resembles the type 2 pattern, prism peripheries were demineralized more deeply than prism cores. A shallower demineralization is observed in comparison to that produced by the phosphoric acid treatment

Futurabond NR are depicted in Figure 4. Although the enamel crystallite dissolution pattern produced by the self-etch system is similar to that produced by phosphoric acid, as both treatments created selective etching of prism cores and peripheries, there is contrast, as the demineralization produced by the former is less defined and shallower than the one produced by phosphoric acid.

DISCUSSION

At present, with the improvements in adhesive system bond strength to enamel and dentin, new micro bond tests need to be developed^{22,25}. However, each test method has both advantages and limitations. In view of this fact, the selection and use of the most feasible test method appears to be an important parameter when conducting a laboratory evaluation¹².

According to the results of the present study, both μ TBS and μ SBS tests showed that the bond strength of the self-etch adhesive system Futurabond NR to ground enamel was as high as the bond strengths of the two etch-and-rinse adhesive systems tested. However, this is not consensual in the literature^{4,11,14}. Although no data were found in the literature with regard to bond tests using Solobond Plus and Futurabond NR to ground enamel, there are some studies that have used similar bonding approaches, which corroborate the present findings. This means that provided that the enamel is abraded, some strong and intermediately strong self-etch systems may reach bond strength values as high as those obtained with phosphoric acid based adhesives^{4,11}.

The analysis of the self-etch adhesive etching pattern improves the understanding of the present findings. Since enamel bonding is primarily based on the micromechanical interlocking of a low-viscosity resin through the formation of tag-like resin extensions into the enamel microporosities, it seems that, apart from the mechanical properties of the adhesive itself, the extent, depth and pattern of etching should influence the performance of the evaluated adhesive¹⁹. Although the etching pattern provided by Futurabond NR, an intermediate strong self-etch system (pH=1.4), was not as deep and evident as that provided by phosphoric acid etching, selective enamel etching could be observed, which may have played a role in the good performance of this adhesive.

However, more important than the adhesives was the type of composite used. Filtek Z250 achieved higher bond strength values in both tests indicating that the resin type might influence the results of the bond test. By reducing the filler content², low viscosity composites were developed to achieve a better sealing of the cavity margins²⁸. In some

clinical and laboratory scenarios the high flowability of this composite is favorable. This explains why some authors have chosen this type of composite to fill the Tygon tube used in μ SBS testing^{10,17,23} instead of the conventional microhybrid composite⁶. This reduction in the filler load aroused some concerns regarding lower mechanical properties and higher polymerization shrinkage when compared with traditional hybrid composites². The elastic modulus of this composite was also significantly reduced (30-50%) when compared with traditional hybrid composites³.

This fact might explain the high number of cohesive failures (~60%) and the lower resin-enamel bond strength means measured in both bond test methods with composite Filtek Flow Z350. In addition, the higher polymerization shrinkage of the low-viscosity composite could potentially create more stress in the interface areas than traditional composites during composite polymerization. Flowable composites do not support higher stresses, particularly when subjected to the μ SBS bond strength test. According to a recent finite element strength analyses¹⁸, the use of flowable composites in μ SBS testing concentrates more stress around the adhesive interface than the use of high filled composites.

Most studies that use the μ TBS test usually report premature failures^{8,23,20} during specimen preparation and this finding was also observed in the present study. In comparison with μ SBS testing, the μ TBS test showed a higher overall percentage of pre-testing failures. This result indicates that the rather aggressive slicing procedure used in specimen preparation for μ TBS testing might induce several structural enamel defects and cracks, leading to premature failures. It can be assumed that this might happen due to the intrinsic brittleness of the enamel tissue in the reduced surface areas used in the μ TBS specimens. This could be an advantage of the μ SBS over the μ TBS test since it does not require any slicing of the enamel substrate prior to testing.

In a previous scanning electron microscopy investigation of unloaded specimens prepared for μ TBS testing⁸, all the specimens revealed the presence of structural defects, which would possibly influence the bond strength values when loaded. The authors pointed out that these defects were more frequent in enamel than in dentin specimens. It was then speculated that the brittleness and low elasticity of enamel may render these specimens intrinsically more prone to defects and failure. This observation is in agreement with other studies that found that enamel specimens can easily crack during the action of trimming^{20,27}.

μ TBS values were higher than the μ SBS values for the same experimental condition. This can be attributed to the differences in the geometry of the

test. Despite this finding, the overall conclusion reached by both tests was the same when the adhesives were tested in enamel. This was confirmed by the positive and significant correlation found between these two bond strength tests in the present investigation. It is noteworthy to mention that this situation might be different in dentin, since no cracking of the substrate is expected to occur when μ TBS test is performed and higher bond strength values are to be measured⁸. If a bond strength test is capable of measuring higher values, an increase in the sensitivity of the test makes it capable of detecting subtle differences between groups.

The ideal scenario would be to choose a test that produces only interfacial failures and high bond strengths values¹³. However, other factors should be considered before choosing the bond test method. For instance, determination of bond strengths to erosion/abrasion cavities in tooth substrates or box-like cavities should be better achieved with the μ TBS test¹⁵. However, the μ SBS test should be preferred for testing a flat tooth surface on which a cut tube can be placed. Moreover, in the case of measuring bond strengths in complex preparations with high C-factors, only a μ TBS test, which can isolate the required area, can be chosen. It is useful for measuring regional variations in resin bond strength to endodontic posts or the inside of root canals¹⁵. In all other situations where a flat or an almost flat surface can be obtained, the μ SBS test could be used.

It may be hypothesized that the selection of the bond strength test seems to be much more dependent on the experimental design of the study. Researchers need to understand the limitations of their test technique and must be sure to document how they conducted the test¹³ because it is imperative to understand all conditions that act on the test protocol in *in vitro* testing⁹. Some authors claim that shear stress is considered more representative of the clinical situation⁵. Nevertheless, it has been pointed out that the stress distribution in this test is non-uniform^{7,30}. While this shear stress is considerably lower than the stresses occurring in the shear test arrangement, it is by no means suggested that tensile testing is ideal. In the case of μ TBS test, although irregular stress distributions due to geometry are avoided, the interfacial stress may not be uniformly tensile due to the changes in elastic modulus of the bonding components²⁹.

Both the μ TBS and the μ SBS tests can be used to test dental adhesives, provided that the findings are cautiously interpreted, since differing methods of load application clearly lead to differing stress distributions²³. Tensile and shear loadings seem to be equally good for laboratory testing of the

adhesive quality of resin systems¹⁶.

CONCLUSIONS

Within the limitations of the present investigation, it may be concluded that a microhybrid resin composite should preferably be used for μ TBS and μ SBS testing due to the reduced number of cohesive failures, and due to the number of high bond strength values obtained, which makes the test more sensitive for measuring differences among experimental conditions. Moreover, both methods seem to be positive and linearly correlated, and can therefore lead to similar conclusions.

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