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ISABELLE ADAD FORNAZARI

Comparative study of microshear and microtensile bond strength tests of composite repairs using universal adhesives

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Tese apresentada ao Programa de Pós-Graduação em Odontologia da Pontifícia Universidade Católica do Paraná, como parte dos requisitos para obtenção do título de Mestre em Odontologia, Área de Concentração em Clínica Odontológica Integrada (Ênfase em dentística).

Orientadora: Profª. Drª. Evelise Machado de Souza

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SUMÁRIO

Title page

To be submitted to **Dental Materials**

Comparative study of microshear and microtensile bond strength tests of composite repairs using universal adhesives

Isabelle Adad Fornazari[1]
Rafael Torres Brum[2]
Rodrigo Nunes Rached[3]
Evelise Machado de Souza[3]

[1] DDS, MSD, PhD Candidate, Graduate Program in Dentistry, School of Life Sciences, Pontifícia Universidade Católica do Paraná, Curitiba, PR, Brazil
[2] DDS, MSD, PhD, Faculty of Dentistry, School of Life Sciences, Pontifícia Universidade Católica do Paraná, Curitiba, PR, Brazil
[3] DDS, MSD, PhD, Graduate Program in Dentistry, School of Life Sciences, Pontifícia Universidade Católica do Paraná, Curitiba, PR, Brazil

Corresponding author:
Evelise Machado de Souza
Imaculada Conceição, 1155
Curitiba, PR - Brazil
80215-901
E-mail: evelise.souza@pucpr.br

Abstract

Objective. The aim of this study was to evaluate the reliability of microshear (μSBS) and microtensile (μTBS) bond strength tests on composite repairs using universal adhesives with or without the application of additional silane.

Methods. Cylindrical (μSBS) and block-shaped (μTBS) specimens were fabricated using nanofilled (F – Filtek Bulk Fill) and a nanohybrid (T- Tetric EvoCeram Bulk Fill) bulk-fill composites. The specimens were aged by thermocycling (5,000 cycles, 5–55 ºC), sandblasted, and then divided into three groups ($n = 30$) as follows: non-repaired (FC and TC), repaired with universal adhesives (FS, Scotchbond Universal; and TA, AdheSE Universal), and with the application of additional silane (FS-S and TA-S). After 48 h, the specimens were repaired using the same composite. The μSBS and μTBS specimens exhibited bonded areas of 1 mm^2 and subject to shear stress and tension until failure, at a cross-head speed of 0.5 mm/min in a universal testing machine. A Weibull analysis and Pearson correlation ($\alpha = 0.05$) were applied to the data.

Results. At 10% and 63.2% probabilities of failure, groups FS and FS-S exhibited significantly higher μSBS values when compared with TA and TA-S, respectively ($p < 0.05$). The same trend was observed for groups FS-S and TA-S when tested by μTBS at a 63.2% probability of failure. The correlation between Weibull modulus was strong negative and not significant ($p > 0.05$).

Significance. Microshear and microtensile bond strength tests used in composite repairs exhibited a material-dependent behavior according to the different Weibull parameters evaluated.

Keywords: composite repair, universal adhesive, microshear bond strength, microtensile bond strength, silane, Weibull

1. Introduction

The major reason for the replacement of composite resin restorations is secondary caries [1]. The replacement of composite resin restorations is also carried out due to material degradation, marginal staining, the loss of anatomical shape, and fractures [2,3]. Previous studies have revealed that the total replacement of the restoration results in increased cavity preparation and a more significant loss in the tooth structure, prolonged clinical time, and higher costs [2,4–6]. Composite repair has been highly recommended in daily clinical practices as a partial replacement of defective restorations due to the increased preservation of the dental structure, prolonged longevity of restoration, and the retaining of the functional tooth for a longer time-period [7–9].

The effectiveness of the repair with respect to the bond strength is dependent on the composite composition, the wettability of the bonding agent, and the surface treatment [10,11]. Chemical bonding between the exposed particles of the original composite and the organic matrix of the repair composite is achieved using a bifunctional agent referred to as silane [12,13]. The application of silane is simple, safe, and requires no additional equipment or techniques [14]. An adhesive should be applied after the silanization of the composite surface, to the improve wetting of the surface due for repair [12,15].

Different bonding strategies have been implemented for composite repairs, such as total-etching [5,16] and self-etching [17,18]. The recent use of universal adhesives allows for the selection of a more suitable strategy by the clinician, with the versatility to bond to direct and indirect restorative materials [19]. Among the commercially available universal adhesives, only a few contain silane in their compositions, which could simplify the repair technique and prevent the separate use of the silane agent [18]. However, only a few studies have been conducted on universal adhesives for composite repairs, the findings of which require further clarification [15,20,21].

In a recent systematic review, it was reported that the microshear bond strength test (μSBS) is the most commonly used method for the evaluation of the repair bond strength [22]. This can be attributed to the ease of specimen preparation [23], simpler test protocol, and lower incidence of pre-testing failures [24,25]. However, problems related to the reliability of microshear values can be attributed to cohesive failures, given that stresses are concentrated on the substrate, which results in the occurrence of failures a distance away from the interface [26]. Based on a finite element analysis, the μSBS test underestimates the true stress, given that a uniform interfacial stress distribution is assumed, which is not achieved [27]. Moreover, the microtensile bond strength (μTBS) test offers several advantages, such as a higher incidence of adhesives failures, less cohesive failures, higher values of the bond strength due to the reduced specimen size, and the testing of irregular surfaces [28,29]. Nevertheless, the μTBS test requires a higher technical demand, and may involve the damage or loss of post-fracture specimens during removal from gripping devices, complex measurements of significantly low bond strengths, and the induction of micro-cracks within the specimen due to the diamond saw sectioning [29,30].

The comparison between the microtensile and microshear tests may be critical to the elucidation of several controversial aspects with respect to the ideal evaluation method of the bond strengths of composite repairs. The aim of this study was to evaluate the reliability of microshear (μSBS) and microtensile (μTBS) bond strength tests on composite repairs using universal adhesives with or without the application of additional silane. The tested null hypotheses were as follows: (1) there is no differences in repair bond strength tested either by μSBS or μTBS, and (2) there are no differences between the repair bond strengths considering the composites, the adhesives and additional silane.

2. Methods

2.1 – Microshear bond strength (μSBS)

A total of 150 cylindrical-shaped specimens (diameter: 4,5 mm; thickness: 5 mm) were fabricated using two bulk-fill resin composites (Filtek Bulk Fill Posterior Restorative, 3M ESPE, St Paul, MN; and Tetric Evoceram Bulk Fill, Ivoclar Vivadent, Schaan, Liechtenstein). The specimens were fabricated using a Teflon mold set between mylar strips and two glass plates. The composites were packed into the mold and light-cured for 40 s using an LED light-curing unit (Bluephase, Ivoclar Vivadent, Schaan, Liechtenstein) at a light intensity of 1200 mW/cm^2. The specimens were then subjected to accelerated aging in a thermal cycling machine (OMC300, Odeme Dental Research, Luzerna, SC, Brazil) for 5,000 cycles at 5 ºC and 55 ºC, with a dwell time of 15 s.

Thereafter, the specimens were air-abraded for 5 s using Al_2O_3 particles with sizes of 50 µm, which were a distance of 10 mm away from the surface, under a pressure of 4 bar. The surfaces of all the specimens were then etched with 37% phosphoric acid for 30 s, rinsed using an air/water spray for 60 s, and air-dried for 60 s. An adhesive tape, which had a central orifice with a diameter of 1.2 mm, was used to limit the bonding area on the composite surfaces that were to be repaired in all the specimens.

The Filtek Bulk Fill Posterior specimens were divided into three groups (n = 30) as follows: non-repaired (C), repaired using a silane-containing universal adhesive (FS-Scotchbond Universal Adhesive, 3M ESPE), and with silane application (FS-S - RelyX Ceramic Primer, 3M ESPE). The Tetric EvoCeram Bulk Fill specimens were divided (n = 30) and subjected to the same treatments using a non-silane containing universal adhesive (TA - Adhese Universal, Ivoclar Vivadent), and with the application of additional silane (TA-S). Silane was applied for 60 s and indirectly air-blasted for 10 s prior to the adhesive application. Adhesives were applied by rubbing on the composite surface for 20 s, followed by indirect air-blasting for 10 s. Light curing was carried out for 20 s using an LED light-curing unit (Bluephase, Ivoclar Vivadent, Schaan, Liechtenstein).

A silicon impression was made from a repaired specimen, to obtain a mold in which the composites were inserted in bulk to realize non-repaired control. The materials used in the study and their respective compositions are shown in Table 1.

Silicon Tygon tubes (1.1 mm diameter) were placed on the treated surfaces. The bulk-fill composites were then inserted into the tubes in one increment, and light-cured for 40 s. The specimens were stored in distilled water at 37 ºC for 48 h.

The tubes and tapes were removed carefully using a scalpel blade, to expose the cylindrical composite repair; and then analyzed using an optical microscope (Olympus BX60, Olympus Corp, Tokyo, Japan) at a magnification of 50 ×, to identify interfacial flaws, gaps, bubbles, and/or other defects. Specimens with these defects were excluded from the study.

Table 1. Universal adhesives and composites used in the study with their respective compositions and manufacturers

Materials (Manufacturer)	Resin Matrix	Filler Type
Scotchbond Universal Adhesive (3M ESPE St Paul, MN)	Bis-GMA, HEMA, 10-MDP, dimethacrylate ethanol, water, initiators, silane	Colloidal silica nanofiller, copolymer of acrylic and itaconic acids
Filtek Bulk Fill Posterior Restorative (3M ESPE St Paul, MN)	AUDMA; UDMA; DDDMA	Silica filler, zirconia filler, Aggregated zirconia/silica cluster filler, ytterbium trifluoride filler (76.5% wt / 58.5% vol)
AdheSE Universal (Ivoclar Vivadent Schaan, Liechtenstein)	HEMA, Bis-GMA, MDP Methacrylated carboxylic acid polymer, D3MA, ethanol, water, initiator	Silicon dioxide nano-filler (67% wt)
Tetric EvoCeram Bulk Fill (Ivoclar Vivadent Schaan, Liechtenstein)	Bis-GMA, UDMA, Bis-EMA	Barium aluminium silicate glass, ytterbium fluoride and spherical mixed oxide (80% wt /61% vol)

Bis-GMA: bisphenol A glycidyl methacrylate; Bis-EMA: ethoxylated bisphenol-A dimethacrylate; HEMA: hydroxyethyl methacrylate; MDP: Methacryloyloxydecyl dihydrogen phosphate; UDMA: diurethane dimethacrylate; AUDMA: aromatic urethane dimethacrylate; DDDMA: 1, 12-Dodecanediol dimethacrylate; D3MA: 1,10 Decanediol dimethacrylate

Microshear bond strength tests were carried out in a universal testing machine (EMIC 2000, Instron, Illinois Tool Works Inc, Norwood, MA) with a metal blade positioned at the repair interface, at a crosshead speed of 0.5 mm/min, until failure.

2.2 – Microtensile bond strength (μTBS)

Sixteen composite blocks (5 mm × 5 mm × 5 mm) were fabricated using two bulk-fill resin composites (Filtek Bulk Fill 3M ESPE, St Paul, MN; and Tetric Evoceram Bulk Fill Ivoclar Vivadent, Schaan, Liechtenstein). The composites were packed into a metal mold and light-cured for 40 s using an LED light-curing unit (Bluephase, Ivoclar Vivadent, Schaan, Liechtenstein). The specimens were then subjected to accelerated aging in a thermal cycling machine (OMC300, Odeme Dental Research, Luzerna, SC, Brazil) for 5,000 cycles at 5 ºC and 55 ºC, with a dwell time of 15 s. Thereafter, the specimens were air abraded for 5 s using Al_2O_3 particles with sizes of 50 μm at a distance of 10 mm away from the surface, under a pressure of 4 bar. The surfaces of all the specimens were etched using 37% phosphoric acid for 30 s, rinsed using an air/water spray for 60 s, and air-blasted for 60 s.

The blocks were divided into the same groups and subjected to the same bonding procedures as described for the specimens in the microshear bond strength tests. The non-repaired control groups were composed of blocks with different dimensions (5 mm × 5 mm × 10 mm). The composites were inserted in bulk and light-cured for 40 s at the top and bottom sides. All the blocks were set in distilled water for 48 h at 37 ºC.

Thereafter, the blocks were serially sectioned perpendicular to the interface using a diamond saw (Extec Corp., Enfield, CT, USA) with a thickness of 0.3 mm at low-speed, and then subjected to water cooling in a cutting machine (Isomet 1000, Buehler, Lake Buff, IL, USA), to obtain sticks with approximate dimensions of 1.0 mm × 1.0 mm × 10 mm. A minimum of 30 sticks were obtained for each group.

The specimens were fixed in a microtensile device (OD03d, Odeme Biotechnology Ltd., Joaçaba, SC, Brasil) using a cyanoacrylate-based glue (Slo-

Zap, Super Glue Corp., Ontario, CA). The microtensile strength test was carried out in a universal test machine (EMIC 2000, Instron, Illinois Tool Works Inc, Norwood, MA) at a speed of 0.5 mm/min. The μTBS values were calculated by the division of the applied force at the time of fracture (F) by the bonded area (mm^2), which was verified using a digital caliper (Absolute Digimatic Caliper, Mitutoyo Corp., Kawasaki, Japan).

2.3 – Failure mode analysis

The failure mode was determined using a stereomicroscope at a magnification of 50× (Olympus UC30, Olympus Corp., Tokyo, Japan), and recorded as 'adhesive failure' (adhesive interface), 'cohesive', or 'mixed failure' (more than one type). The most representative failures of each group were selected for analysis using scanning electron microscopy - SEM (Vega 3, Tescan Orsay Holding, Brno, Czech Republic).

2.4– Statistical analysis

The Weibull distribution parameters (the Weibull modulus/scale (*m*)), characteristic strength, 63.2% probability of failure (σ_θ), and 10% probability of failure (σ_{10}) were calculated using the maximum likelihood estimation method at a confidence level of 95%, to determine the reliability and durability trends of the resin composite repairs. The Pearson correlation of both methods was carried out at σ_{10}, $\sigma_{\theta,}$ and *m*. Moreover, all the tests were carried out at a significance level of 0.05 (Minitab V.18, State College, PA, USA)

3. Results

3.1 Microshear bond strength (μSBS)

The μSBS results are presented in Table 3. The Weibull modulus varied from 2.90 (TA) to 4.97 (FS-S), and no differences were observed between the non-repaired and repaired groups within the same resin composite.

At 10% and 63.2% probabilities of failure, a significant difference was found between the groups in which silane was applied prior to the universal adhesives, and TA-S exhibited a lower bond strength than FS-S ($p < 0.05$). The same trend was observed in the comparison between groups FS and TA ($p < 0.05$).

The Weibull plot of the μSBS specimens (Fig. 1) reveals that TA and TA-S were the least reliable treatments for composite repair when compared with the other groups, as indicated by the steepness and location of the lines.

Table 2 – Microshear bond strength of the evaluated groups (MPa)

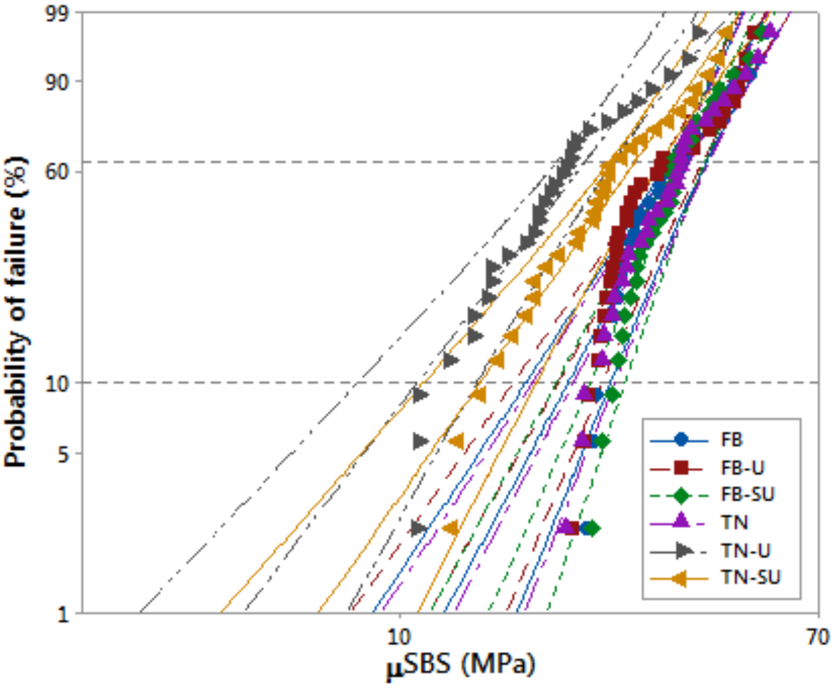
Groups	*m*	σ_{10}[1]	σ_{θ}[2]
FC	4.11 [3.16 – 5.34]^{ab}	21.98 [18.07 – 26.73]^{ab}	38.01 [34.65 – 41.70]^{a}
FS	3.92 [2.99 – 5.14]^{ab}	20.96 [17.01 – 25.82]^{ab}	37.18 [33.75 – 40.96]^{a}
FS-S	4.97 [3.84 – 6.43]^{a}	24.39 [20.78 – 28.61]^{a}	38.36 [35.53 – 41.41]^{a}
TC	4.20 [3.23 – 5.47]^{ab}	22.53 [18.61 – 27.29]^{ab}	38.49 [35.37 – 42.13]^{a}
TA	2.90 [2.23 – 3.78]^{b}	10.90 [08.26 – 14.38]^{c}	23.66 [20.75 – 26.96]^{b}
TA-S	3.14 [2.39 – 4.13]^{ab}	14.58 [11.22 – 18.95]^{bc}	29.84 [26.45 – 33.67]^{b}

m= Weibull modulus/scale parameter and 95% interval;

[1] Estimation and 95% interval at 10% probability of failure (PF10); groups with the same letter are statistically not different

[2] Estimation and 95% interval at characteristic strength (63.2% probability of failure); groups with the same letter are statistically not different

Fig. 1– Weibull plot for μSBS. Dotted lines represent 95% confidence bounds for each group.



3.2 Microtensile bond strength (μTBS)

The μTBS results are presented in Table 4. The Weibull modulus ranged from 3.46 (FC) to 4.62 (TA-S); however, no significant differences were observed between the groups, which indicates that the adhesive treatments for repair yielded similar bond strengths when compared with the non-repaired groups.

At 10% probability of failure, no statistically significant differences were found between all the groups ($p > 0.05$). At the characteristic strength, the FS-S group with the application of additional silane application exhibited a statistically superior bond strength than those compared with its counterpart TA-S and FS ($p < 0.05$), whereas no significant differences were found to FC ($p > 0.05$).

The Weibull plot of the μTBS specimens depicted in Fig. 2 reveals the reliability of each group, as indicated by the steepness of the lines, which was in good agreement with the characteristic strength values.

Table 3 – Microtensile bond strength of evaluated groups (MPa)

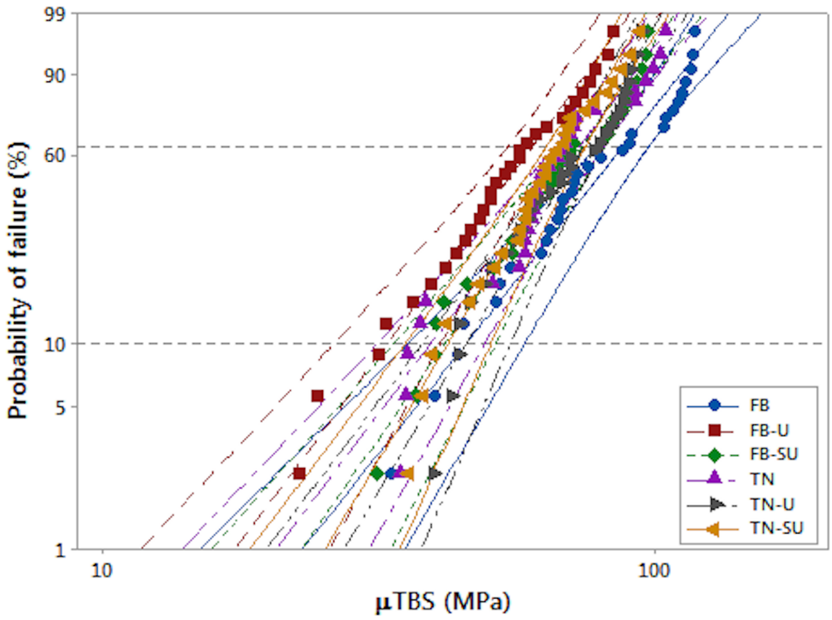
Groups	*m*	σ_{10}[1]	σ_{θ}[2]
FC	3.46 [2.60. – 4.60]^{a}	45.69 [35.80 – 58.30]^{a}	87.57 [78.51 – 97.68]^{a}
FS	3.73 [2.81 – 4.95]^{a}	32.84 [26.26 – 41.08]^{a}	60.00 [54.23 – 66.38]^{c}
FS-S	3.95 [2.97 – 5.26]^{a}	41.77 [33.73 – 51.73]^{a}	73.85 [67.12 – 81.26]^{a}
TC	3.66 [2.78 – 4.80]^{a}	39.43 [31.51 – 49.35]^{a}	72.97 [65.79 – 80.93]^{abc}
TA	4.62 [3.46 – 6.17]^{a}	45.71 [38.02 – 54.95]^{a}	74.38 [68.54 – 80.71]^{ab}
TA-S	4.59 [3.49 – 6.04]^{a}	42.37 [35.43 – 50.68]^{a}	69.15[63.69 – 75.09]^{bc}

m= Weibull modulus/scale parameter and 95% interval;

[1] Estimation and 95% interval at 10% probability of failure (PF10); groups with the same letter are statistically not different

[2] Estimation and 95% interval at characteristic strength (63.2% probability of failure); groups with the same letter are statistically not different

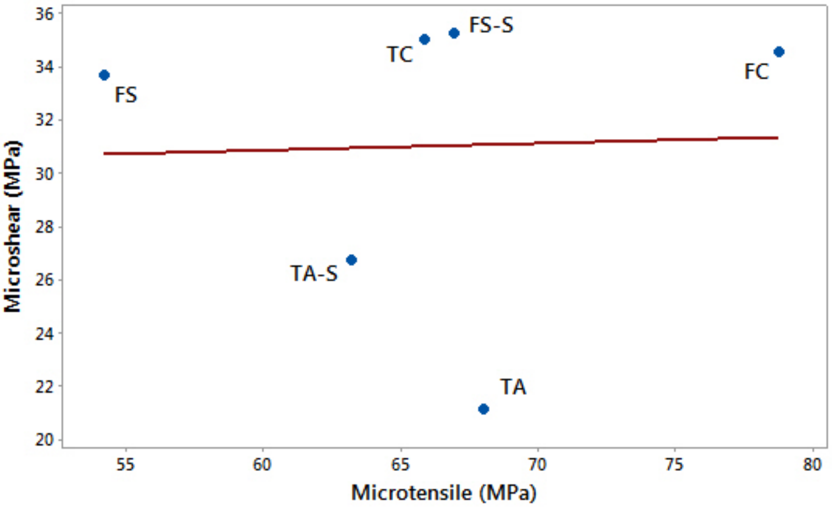
Fig. 2 – Weibull plot for μTBS. Dotted lines represent 95% confidence bounds for each group.



3.3 Correlation of microshear and microtensile bond strength tests

Both tests had a weak negative correlation at σ_{10} (R = -0,379 p = 0,458), and a very weak positive correlation at σ_{θ} (R = 0,082, p = 0,878). The correlation for *m* (Fig. 3) was strong negative (R = -0,721, p = 0,106).

Fig. 3 – Correlation analysis per tests for Weibull modulus (*m*)



3.4 – Failure modes

The frequency of failure modes of the μSBS and μTBS groups are presented in Fig. 4 and SEM images of representative failures are shown in Figs. 5 and 6.

Most of the μSBS fractures were mixed for FS-S and TA, and equally distributed between mixed and cohesive for FS and TA-S. TA and TA-S exhibited the same frequency of adhesive and mixed failures modes for μTBS, whereas FS and FS-S exhibited more mixed failures. The groups submitted to μTBS exhibited more frequency of adhesive failures compared with μSBS, especially TA group.

Fig. 4 – Failure mode frequency (%) for the evaluated groups paired by bond strength test.

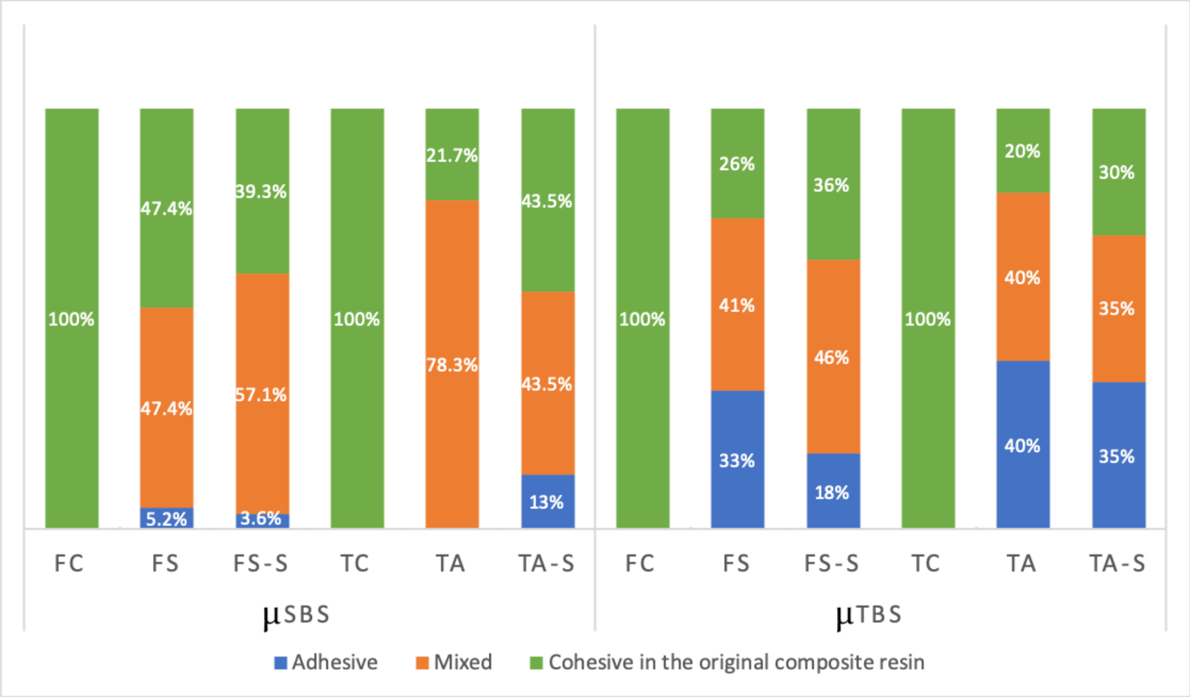


Fig. 5 – Representative SEM micrographs of μSBS test: (1) adhesive, (2) cohesive, (3) mixed; A – Adhesive; C – Composite

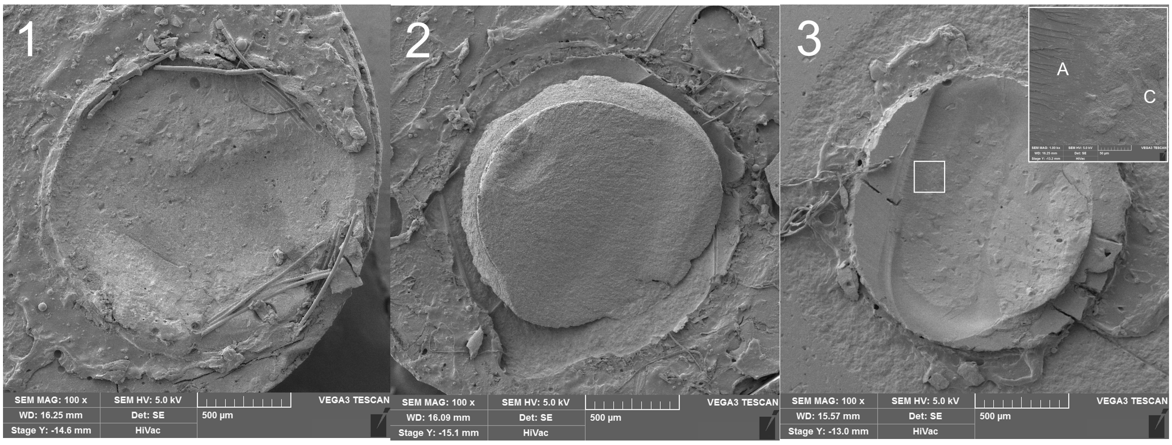
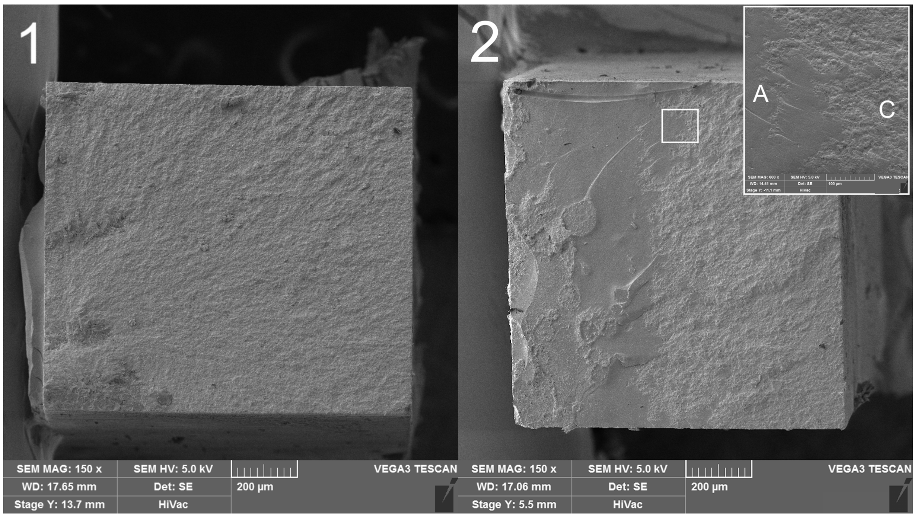


Fig. 6 – Representative SEM micrographs of μTBS test: (1) cohesive, (2) mixed; A – Adhesive; C – Composite



4. Discussion

In this study, microshear and microtensile bond strength tests were compared for the evaluation of composite repair using silane-containing and silane-free universal adhesives. The first hypothesis was rejected, given that the μSBS and μTBS tests yielded different results for the same groups. The second hypothesis was also rejected, given that different associations of composites and universal adhesives resulted in different repair bond strengths.

The most commonly used coupling agent for ceramic and composite repairs is the silane functional monomer γ-methacryloxypropyltrimethoxysilane (γ-MTPS) [31,32]. Most of the previous studies revealed that the application of silane increases the repair bond strength when compared with the application of adhesives [33–35]. However, the effectiveness of silane is directly related to the surface treatment, which is responsible for the exposure of the filler particles on the composite surface [36,37]. Studies have revealed that sandblasting [15,38,39] and tribochemical [33,40,41] treatment are more effective, and lead to irregular surface morphologies with higher particle exposure areas. All the tested specimens in this study were sandblasted, which may have contributed to increased mechanical retention in the composite surface and the filler particle exposure. Therefore, the repair bond strength with both universal adhesives associated and additional silane application was similar to the non-repaired specimens, with the exception of TA-S vs TC at the characteristic strength tested by the μSBS.

The application of the silane-containing universal adhesive resulted in a lower bond strength at the characteristic strength (σ_θ) in comparison with the group with additional silane application and the non-repaired group, when tested by μTBS. This can be attributed to the chemical stability of the silane incorporated in the Scotchbond Universal adhesive. Although the manufacturer claimed that silane is stable in a solution with alcohol, filler, and a moderately acidic pH [42], recent studies revealed that the low pH of Scotchbond Universal (2.7) may promote hydrolysis and dehydration condensation, thus resulting in the chemical instability of silane [43,44]. Therefore, the application of silane is advisable for composite repair, even when a silane-containing universal adhesive is used [45].

A Weibull analysis provides information related to the performance of an adhesive material, instead of depending on the mean bond strength and standard deviation [46]. It is characterized by two principal parameters, namely, the Weibull modulus and the Weibull stress value required to cause a failure, which can be used to evaluate the performance of a bond at a constant percentage level. The characteristic strength (σ_θ) is the strength value at a 63.2% probability of failure, and it is a location parameter. In particular, a high characteristic strength shifts the data to the right, whereas a low characteristic strength shifts the data to the left [47]. The probability of failure at 10% reflects early failures in clinical situations [48]. Based on the results, μTBS was able to distinguish more differences at the characteristic strength, while μSBS distinguish better at 10% probability of failure.

The Weibull modulus (*m*) reflects the variability and reliability of the results [46,49]. A high variability in bond strength is translated into low *m* values, which indicates a low reliability of the characteristic bond strength due to the presence of critical flaws [49]. In this study, both composites exhibited an inverse behavior in terms of modulus, and this was in the strong inverse correlation found between the tests.

There is a lack of consensus with respect to the frequency of failure modes in studies on repair bond strengths. In several μSBS studies, no or minimum cohesive failures were reported for repaired specimens [18,50]; which is in contrast to other studies, wherein a higher percentage of cohesive [51,52] or adhesive failures were reported [33,53]. Such differences may be due to the different methods employed, or they may result from the critical stress distribution of the μSBS test. The μSBS test results exhibited fewer adhesive failures. Conversely, μTBS exhibited a higher frequency of adhesive failures and lower frequency of cohesive failures. It has been reported that the high frequency of cohesive failures in μSBS is because the majority of the resultant stresses are concentrated in the substrate, thus resulting in premature failure prior to the adhesive failure at the interface [54]. This is dissimilar to μTBS, which exhibits a better stress distribution during loading, thus resulting in fewer cohesive failures [28]. The results of a finite element analysis revealed that complex stresses occur at the interface, in which high tensile stresses are generated due to the bending moment during the μSBS test [55].

The limitations of the study can be attributed to the variation in the specimen geometries (cylindrical vs squared) according to each bond strength test. This variation may have an influence on the stress concentration at the interface, and therefore the nominal bond strength values. However, the μTBS test was more reliable for the evaluation of the bond strength, and it is therefore suitable for the evaluation of composite repairs. Additional mechanical approaches such as finite element analyses and fracture toughness tests could lead to a better understanding of the interfacial behavior of composite repairs.

5. Conclusion

Microshear and microtensile bond strength tests used in composite repairs exhibited a material-dependent behavior according to the different Weibull parameters evaluated.

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ANEXOS

Análise estatística

Programa Minitab V18

Microshear

Distribution Analysis: FB

Variable: FB

Censoring

Censoring Information	Count
Uncensored value	30

Estimation Method: Maximum Likelihood

Distribution: Weibull

Parameter Estimates

		Standard	95,0% Normal CI	
Parameter	Estimate	Error	Lower	Upper
Shape	4,10666	0,549681	3,15904	5,33855
Scale	38,0166	1,79659	34,6535	41,7061

Log-Likelihood = -108,299

Goodness-of-Fit

Table of Percentiles

		Standard	95,0% Normal CI	
Percent	Percentile	Error	Lower	Upper
10	21,9780	2,19384	18,0726	26,7273
63,2	38,0135	1,79658	34,6505	41,7030

Distribution Analysis: FB-U

Variable: FB-U

Censoring

Censoring Information	Count
Uncensored value	30

Estimation Method: Maximum Likelihood

Distribution: Weibull

Parameter Estimates

```
                                   Standard       95,0% Normal CI
Parameter        Estimate          Error          Lower          Upper
Shape            3,92440           0,540980       2,99526        5,14176
Scale            37,1856           1,83807        33,7520        40,9684
```

Log-Likelihood = -109,188

Table of Percentiles

```
                                   Standard       95,0% Normal CI
Percent          Percentile        Error          Lower          Upper
     10          20,9574           2,23096        17,0108        25,8196
   63,2          37,1825           1,83806        33,7489        40,9653
```

Distribution Analysis: FB-SU

Variable: FB-SU

Censoring

```
Censoring Information        Count
Uncensored value                30
```

Estimation Method: Maximum Likelihood
Distribution: Weibull

Parameter Estimates

```
                                   Standard       95,0% Normal CI
Parameter        Estimate          Error          Lower          Upper
Shape            4,96689           0,653387       3,83804        6,42775
Scale            38,3639           1,49847        35,5365        41,4162
```

Log-Likelihood = -103,341

Table of Percentiles

```
                                   Standard       95,0% Normal CI
Percent          Percentile        Error          Lower          Upper
     10          24,3868           1,98905        20,7840        28,6142
   63,2          38,3613           1,49848        35,5340        41,4137
```

Distribution Analysis: TN

Variable: TN

Censoring

Censoring Information	Count
Uncensored value	30

Estimation Method: Maximum Likelihood

Distribution: Weibull

Parameter Estimates

Parameter	Estimate	Standard Error	95,0% Normal CI Lower	95,0% Normal CI Upper
Shape	4,20300	0,566339	3,22748	5,47339
Scale	38,4931	1,77455	35,1676	42,1332

Log-Likelihood = -108,294

Table of Percentiles

Percent	Percentile	Standard Error	95,0% Normal CI Lower	95,0% Normal CI Upper
10	22,5348	2,20308	18,6053	27,2942
63,2	38,4901	1,77455	35,1646	42,1302

Distribution Analysis: TN-U

Variable: TN-U

Censoring

Censoring Information	Count
Uncensored value	30

Estimation Method: Maximum Likelihood

Distribution: Weibull

Parameter Estimates

Parameter	Estimate	Standard Error	95,0% Normal CI Lower	95,0% Normal CI Upper
Shape	2,90380	0,391279	2,22982	3,78150
Scale	23,6582	1,57872	20,7578	26,9639

Log-Likelihood = -103,124

Table of Percentiles

Percent	Percentile	Standard Error	95,0% Normal CI Lower	95,0% Normal CI Upper
10	10,8997	1,54247	8,25959	14,3838
63,2	23,6556	1,57866	20,7553	26,9611

Distribution Analysis: TN-SU

Variable: TN-SU

Censoring

Censoring Information	Count
Uncensored value	30

Estimation Method: Maximum Likelihood
Distribution: Weibull

Parameter Estimates

Parameter	Estimate	Standard Error	95,0% Normal CI Lower	95,0% Normal CI Upper
Shape	3,14044	0,439048	2,38775	4,13041
Scale	29,8484	1,83742	26,4559	33,6759

Log-Likelihood = -108,705

Table of Percentiles

Percent	Percentile	Standard Error	95,0% Normal CI Lower	95,0% Normal CI Upper
10	14,5786	1,94943	11,2175	18,9469
63,2	29,8453	1,83737	26,4529	33,6727

Microtensile

Distribution Analysis: FB

Variable: FB

Censoring

Censoring Information	Count
Uncensored value	30

Estimation Method: Maximum Likelihood
Distribution: Weibull

Parameter Estimates

Parameter	Estimate	Standard Error	95,0% Normal CI Lower	95,0% Normal CI Upper
Shape	3,45810	0,503714	2,59926	4,60072
Scale	87,5787	4,88149	78,5153	97,6885

Log-Likelihood = -139,233

Table of Percentiles

Percent	Percentile	Standard Error	95,0% Normal CI Lower	95,0% Normal CI Upper
10	45,6857	5,68410	35,7994	58,3022
63,2	87,5704	4,88142	78,5072	97,6800

Distribution Analysis: FB-U

Variable: FB-U

Censoring

Censoring Information	Count
Uncensored value	30

Estimation Method: Maximum Likelihood

Distribution: Weibull

Parameter Estimates

Parameter	Estimate	Standard Error	95,0% Normal CI Lower	95,0% Normal CI Upper
Shape	3,73367	0,537818	2,81530	4,95163
Scale	60,0067	3,09368	54,2395	66,3871

Log-Likelihood = -125,977

Table of Percentiles

Percent	Percentile	Standard Error	95,0% Normal CI Lower	95,0% Normal CI Upper
10	32,8428	3,74926	26,2585	41,0782
63,2	60,0014	3,09365	54,2343	66,3818

Distribution Analysis: FB-SU

Variable: FB-SU

Censoring

Censoring Information	Count

Censoring Information	Count
Uncensored value	30

Estimation Method: Maximum Likelihood

Distribution: Weibull

Parameter Estimates

Parameter	Estimate	Standard Error	95,0% Normal CI Lower	95,0% Normal CI Upper
Shape	3,94910	0,577045	2,96565	5,25867
Scale	73,8551	3,60132	67,1235	81,2619

Log-Likelihood = -130,875

Table of Percentiles

Percent	Percentile	Standard Error	95,0% Normal CI Lower	95,0% Normal CI Upper
10	41,7735	4,55555	33,7345	51,7282
63,2	73,8490	3,60131	67,1174	81,2558

Distribution Analysis: TN

Variable: TN

Censoring

Censoring Information	Count
Uncensored value	30

Estimation Method: Maximum Likelihood

Distribution: Weibull

Parameter Estimates

Parameter	Estimate	Standard Error	95,0% Normal CI Lower	95,0% Normal CI Upper
Shape	3,65575	0,509192	2,78239	4,80327
Scale	72,9788	3,85553	65,8002	80,9406

Log-Likelihood = -131,753

Table of Percentiles

Percent	Percentile	Standard Error	95,0% Normal CI Lower	95,0% Normal CI Upper
10	39,4329	4,51283	31,5097	49,3484
63,2	72,9722	3,85548	65,7937	80,9340

Distribution Analysis: TN-U

Variable: TN-U

Censoring

Censoring Information	Count
Uncensored value	30

Estimation Method: Maximum Likelihood

Distribution: Weibull

Parameter Estimates

Parameter	Estimate	Standard Error	95,0% Normal CI Lower	95,0% Normal CI Upper
Shape	4,62182	0,682926	3,45970	6,17430
Scale	74,3827	3,09993	68,5484	80,7134

Log-Likelihood = -127,096

Table of Percentiles

Percent	Percentile	Standard Error	95,0% Normal CI Lower	95,0% Normal CI Upper
10	45,7102	4,29382	38,0238	54,9505
63,2	74,3774	3,09995	68,5431	80,7082

Distribution Analysis: TN-SU

Variable: TN-SU

Censoring

Censoring Information	Count
Uncensored value	30

Estimation Method: Maximum Likelihood

Distribution: Weibull

Parameter Estimates

Parameter	Estimate	Standard Error	95,0% Normal CI Lower	95,0% Normal CI Upper
Shape	4,59398	0,643426	3,49117	6,04515
Scale	69,1576	2,90555	63,6910	75,0934

Log-Likelihood = -124,329

Table of Percentiles

Percent	Percentile	Standard Error	95,0% Normal CI Lower	95,0% Normal CI Upper

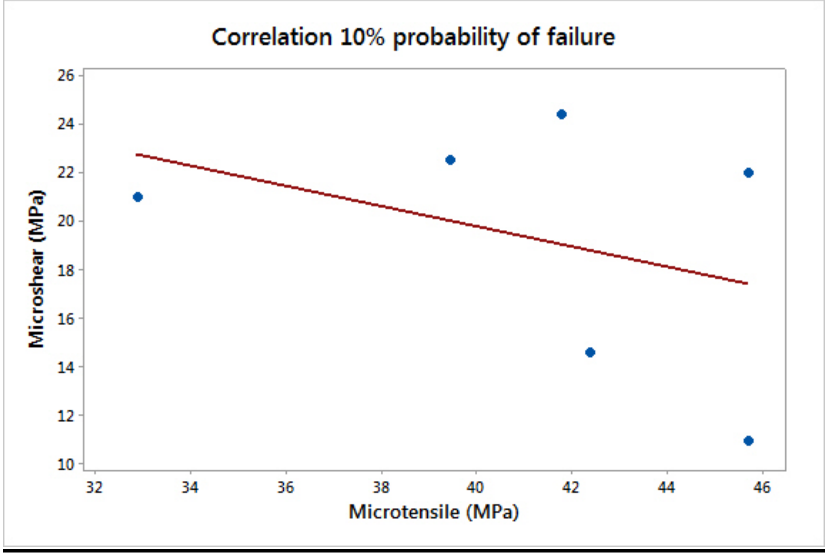
10	42,3741	3,87023	35,4288	50,6809
63,2	69,1527	2,90557	63,6861	75,0886

Correlação de Pearson

10% probability of failure:

Correlação: 10 - SBS; 10 - TBS

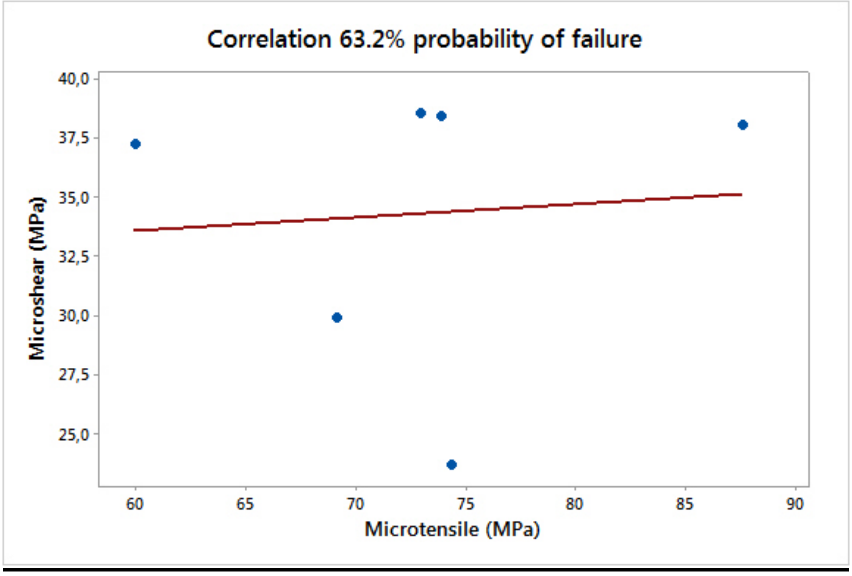
Correlação de Pearson	-0,379
Valor-P	0,458



63.2% probability of failure:

Correlação: 63 SBS; 63 TBS

Correlação de Pearson	0,082
Valor-P	0,878



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Introduction

This must be presented in a structured format, covering the following subjects, although actual subheadings should not be included:
- succinct statements of the issue in question;
- the essence of existing knowledge and understanding pertinent to the issue (reference);

- the aims and objectives of the research being reported relating the research to dentistry, where not obvious.

Materials and methods

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