Mestrado Integrado em Medicina Dentária

Faculdade de Medicina da Universidade de Coimbra

Comparação de duas metodologias na determinação das forças de adesão à dentina

Bond strength evaluation to dentin: comparing two test methods

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Abstract

Introduction: Modern restorative dentistry has been significantly influenced by the constant progress of dental adhesives technology. In vitro mechanical bond strength tests became of highest importance to evaluate and compare the current adhesive systems to enamel and dentin.

Purpose: The aim of this study was to compare the adhesive bond strengths and failure modes of dentin–composite interfaces measured by two different test methods: shear and microshear.

Materials and Methods: Thirty caries free human pre-molars were randomly assigned to the bond strength testing methods compared (n=15). After occlusal flat dentin surfaces were obtained and grinded with 240-, 400-, 600-grit silicon-carbide sandpaper. Adhesively composite specimens (SDR™, DentsplyDeTrey; Konstanz, Germany) were bonded to dentin prepared surfaces by using a two-step etch & rinse adhesive system (Prime&Bond®NT™, DentsplyDeTrey, Konstanz, Germany) according the technical specificities of each method. Shear and microshear bond strength tests were performed after 7 days of storage in distilled water at 37ºC.

Following specimens fracture, failure mode pattern was evaluated and data statistical analysed with t student test for a significance level of 0,05.

Results: The mean bond strength, in MPa (±SD), was 9,02 (±2,07) and 11,68 (±5,79) for the shear and microshear test, respectively. Concerning bond strengths mean values, no significant differences were found between groups. The failure mode for all the specimens was classified as adhesive and microshear test bond strength exhibited a larger standard deviation value.

Conclusions: More studies are essential to understand the specific restrictions of microshear bond test because it has an inherent more labour-intensive and sensitive technique when compared to shear bond strength test.

Keywords: shear, microshear, dentin bond strength, bonding systems

Resumo

Introdução: A Medicina Dentária actual tem sido significativamente influenciada pela constante evolução da tecnologia dos adesivos dentários. Os testes laboratoriais *in vitro* de resistência adesiva tornaram-se importantes para avaliar e comparar os atuais sistemas adesivos para o esmalte e dentina.

Objectivos: O objetivo deste estudo foi comparar as forças de adesão e os modos de fratura na interface dentina-resina avaliada por dois métodos de avaliação: cisalhamento e microcisalhamento.

Materiais e métodos: Trinta dentes pré-molares humanos íntegros foram aleatoriamente distribuídos pelos métodos de avaliação estudados para a determinação das forças de adesão (n=15). Após a obtenção de superfícies planas de dentina e da realização de processos de polimento com lixas de carboneto de silício de grão crescente 240 -, 400- e 600-, procedeu-se à execução das amostras pela utilização de um sistema adesivo do tipo condicionar e lavar de dois passos (Prime&Bond®NT™, DentsplyDeTrey, Konstanz, Alemanha) e subsequente colocação de uma resina composta (SDR™, DentsplyDeTrey; Konstanz, Alemanha) de acordo com as especificidades técnicas de cada método. Os testes de cisalhamento e microcisalhamento foram realizados após 7 dias de armazenamento dos dentes em água destilada a 37º C. Os modos de fratura da interface foram

determinados e os dados foram analisados estatisticamente com o teste t de Student para um nível de significância de 0,05.

Resultados: A força de adesão média do Prime&Bond™NT ®/ SDR™ em MPa (± SD), foi 9,02 (± 2,07) e 11,68 (± 5,79) para o teste de cisalhamento e microcisalhamento, respectivamente. Quanto aos valores médios de adesão, não foram encontradas diferenças estatisticamente significativas entre os grupos. O modo de fratura na interface foi classificado como adesivo para todas as amostras e o teste de microcisalhamento apresentou um valor de desvio padrão mais elevado.

Conclusões: É essencial que sejam realizados mais estudos para compreender as limitações específicas do teste de microcisalhamento pois, como foi possível verificar, a técnica é mais sensível e mais demorada do que a do teste de cisalhamento.

Introduction

The advent of adhesive restorative dentistry with a continued and fast development of new products leads the in vitro mechanical tests to become of a paramount importance to initial evaluation and comparison of bond strengths of adhesive systems to enamel and dentin[1-4].

Although the validity of bond strength tests to predict clinical performance of dental adhesives is questionable, recent evidence shows that some clinical results can, in some instances, be estimated based upon laboratory results. Moreover, mechanical testing of bonded interfaces has provided some valuable information in terms of identifying substrate variables and helping to define guidelines for application procedures and protocols for clinical trials[5].

By definition, the ideal bond strength test should be easy, with low technique-sensitivity, reproducible, reliable and relatively fast and inexpensive[6]. The preference for conventional shear and tensile tests is justified because they have some of those qualities[5]. The bond strength can be measured using a macro or micro test set-up, basically depending upon the size of the bond area[6]. However, conventional shear and tensile tests have been criticized for using relatively larger bonded surfaces, over which stress distribution is likely to be uneven in relation to the density of intrinsic faults, possibly acting as stress raisers[3].

The most commonly used technique for bond strength assessment is the shear bond strength, mainly because of their relative simplicity when compared to tensile bond strength tests[6]. Shear bond strength test reached high popularity between manufactures and other research centres because their easiness specimen preparation, simple test protocol and relatively fast execution[6]. However, problems related to the validity of obtained measurements started to arise as cohesive failures in the substrate were frequently observed with new adhesives that yield improved bond strengths[4].

Different studies have shown that macro shear tests are significantly influenced by the variability on specimen geometry and experimental setup[7-9], bonded area[10], preparation tools[11], operator experience[12] and other variable factors[13,14]. The ability to load multiple small specimens to one tooth in either a microtensile or microshear approach has become a useful and the most current methodologies for bond strength assessment[15].

Microtensile bond strength test allows measurements of the tensile bond strength on very small surfaces[4,10,16]. The bond area tested of 1 mm^2 or less is much smaller compared to that of the "macro" tests and involves the application of a loading force perpendicular to the adhesive interface. This kind of test gave rise to evaluate regional variations within dentin or other substrates and had the advantage of producing many specimens from the same tooth[16], which allows easier sample collection, the ability to compare between a variety of substrates and areas in the same tooth[2] and offers more uniform stress distribution[3].

Microshear bond strength test has become popular as an alternative to the conventional shear bond strength test[2]. Theoretically, this test combines the facility of manipulation with the ability to test several specimens per tooth[3,6]. This methodology involves the application of a loading force parallel to adhesive interface by means of a chisel–shaped rod or a wire loop from a universal testing machine[17,18]. Likewise microtensile, microshear test uses small areas, thus allowing a regional mapping or depth profiling of different substrates as well as preparing multiple specimens from the same tooth[3,4].

Despite the disagreement between laboratory data and clinical outcomes[19], those experimental trials still represents the most often routine procedures for bonding evaluation[4,8,16].

The objective of this study was to compare the adhesive bond strengths and failure modes at the dentin–composite interface measured by two different test methods: shear and microshear. The null hypothesis was that there were no differences between both methods.

Materials and methods

Thirty caries free, unrestored extracted human premolars were collected and stored for up to 8 weeks after extraction. The teeth were cleaned from debris, vertically embedded in a self-cure acrylic resin (Orthocryl, Dentaurum) using phenolic rings leaving the coronal part outside of the cylinder. Occlusal surfaces were cut and grounded perpendicular to the long axis of the tooth, under water-cooling (Accutom 50, Struers, Ballerup, Denmark), until a flat dentin surface without any residual enamel was obtained, which was confirmed under microscope magnification at 40x (Leica M320 F12, Switzerland). Following, dentin surface were further wet-ground with 240-, 400- and 600-grit silicon-carbide sandpaper in a circular motion for 60 seconds to create an uniform smear layer. Finally, the teeth were thoroughly rinsed with water.

The prepared premolars were randomly divided in 2 groups, according bonding test methodology (n=15) and subjected to a bonding procedure strictly according to the manufacturer's instructions with a two-step etch and rinse adhesive (Prime&Bond®NT™, DentsplyDeTrey, Konstanz, Germany) and a flowable "low-shrinkage" composite resin (SDR™, DentsplyDeTrey; Konstanz, Germany) (Table I), according to the specific methodology inherent to each bond strength test method, either shear (Group 1) or microshear (Group 2).

Table I: Materials studied, manufacturers, composition and batch numbers.

Composite	Manufacturer	Composition	Filler	Batch n.
SDR™ Microhybrid	Dentsply DeTrey	Modified UDMA EBPADMA TEGDMA	Ba-Al-F-B-Si-glass Sr-Al-F-Si-glass (68 wt %., 45 vol %)	1201231
Adhesive	**Manufacturer**	**Chemical Composition**	**Instructions**	**Batch n.**
Prime&Bond ®NT™ 2-Step Etch & Rinse Adhesive	Dentsply DeTrey	Di-and trimethacrylate resins PENTA Photoinitiators Stabilizers Nanofillers Acetone	Apply 36% phosphoric acid for 15 seconds; spray and rinse with water for 15 seconds; blot dry conditioned areas; apply adhesive and leave the surface wet for 20 seconds; gently dry for at least 5 seconds; polymerize for 10 seconds; apply a second layer of adhesive.	1201231

UDMA: urethane dimethacrylate; EBPADMA: ethoxylated bisphenol A dimethacrylate; BISGMA: bisphenol A-glycidyl methacrylate; PENTA: dipenta- erythritol penta acrylate monophosphate

Group 1 - Shear Bond Strength (SBS) protocol: each sample was etched for 15 seconds with 36% phosphoric acid (Conditioner 36, Dentsply DeTrey) and immediately rinsed with water for 15 seconds, removing excess water with a cotton pellet and leaving a moist dentin surface. The adhesive (Prime&Bond®NT™, DentsplyDeTrey, Konstanz, Germany) was applied using a micro brush and left undisturbed for 20 seconds. Then a 5 second gentle air blast was applied promoting solvent evaporation, followed by a 10 second light-curing exposure (Bluephase®, Ivoclar Vivadent, Lichenstein). A second layer of adhesive was then applied and cured immediately in similar way. For delimiting bonded area, a size #9 gelatine capsules with 4.9 mm^2 cross-sectional area were used (Torpac Inc., Fairfield, NJ, USA), filled with composite resin (SDR™, DentsplyDeTrey; Konstanz, Germany), applied to the treated dentin surface and light-cured for 40 seconds using a LED device (Bluephase®, Ivoclar Vivadent, Lichenstein) (Figure 1). Then, the samples were stored in distilled water at 37ºC for 7 days.

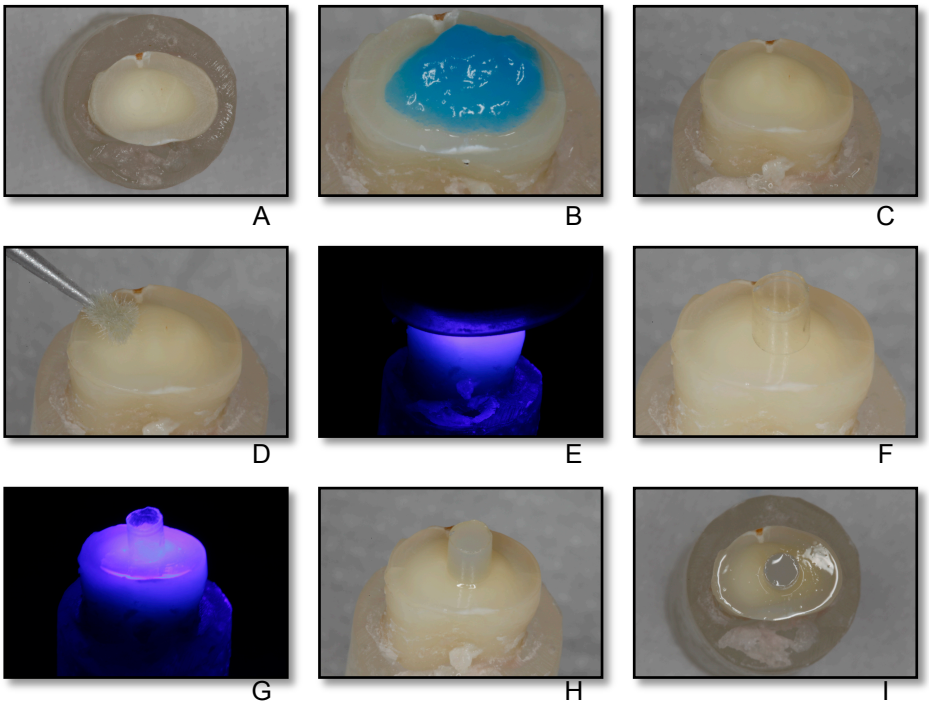
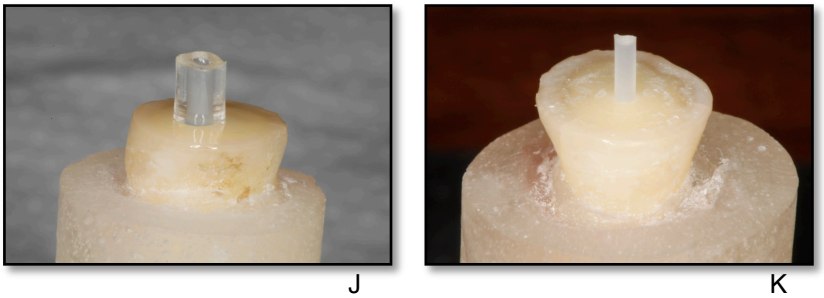


Figure 1: Sequential representation of the sample preparation inherent to shear bond strength test method: dentin etching with 36% phosphoric acid (A, B, C); applying adhesive system Prime&Bond®NT™ (D); adhesive light-curing (E); application of a second adhesive layer followed by a size #9 gelatine capsule positioning and light curing (F, G); capsule filled with SDR™ (H, I).

Group 2 - Microshear Bond Strength (μSBS) protocol: the dentin surfaces of the premolars were treated with the same adhesive as in group 1. For bonded area delimitation, a tygon tube (Tygon Tubing, TYG-030, Saint Gobain Performance Plastic; Maime Lakes, FL, USA) with internal cross-sectional area of 0.5 mm^2 was used and filled with the same composite and followed the procedures as in group 1. However, after storage period, while gelatin capsules of group 1 dissolved in the water storage, plastic tygon tubes must be removed by gently cutting the tube into two hemi-cylinders using surgical blade (Figure 2) under an optical microscope at x40 magnification (Leica M320 F12, Switzerland). Composite cylinders samples with evident interfacial defects were excluded.



J K

Figure 2: Specimens for microshear bond strength test. Plastic mold filled with SDR™ (J). Composite cylinder bonded to dentin surface after plastic mold removal (K).

After storage period all the samples were tested in shear mode using a universal testing machine (Model AG-I, Shimadzu Corporation, Kyoto, Japan). The compression load resulting in the shear bond strength was performed parallel and near of the adhesive interface. The shear force was applied by a chisel-shaped rod at a crosshead speed of 1mm/minute, up to bond disruption (Figure 3). Shear bond strength values, expressed in MegaPascals (MPa), were calculated by dividing peak break force (N) by the cross-sectional area of the bonded interface of each group.

The bond strengths values of each specimen was registered in an excel file and statistical analysis was conducted with t student test at a significance level of 0.05. The debonded specimens were observed for failure modes characterization by a single operator under a optical microscope at 40x magnification (Leica M320 F12, Switzerland) and classified in either cohesive failures in dentin, cohesive failures in resin, adhesive failures or mixed failures.

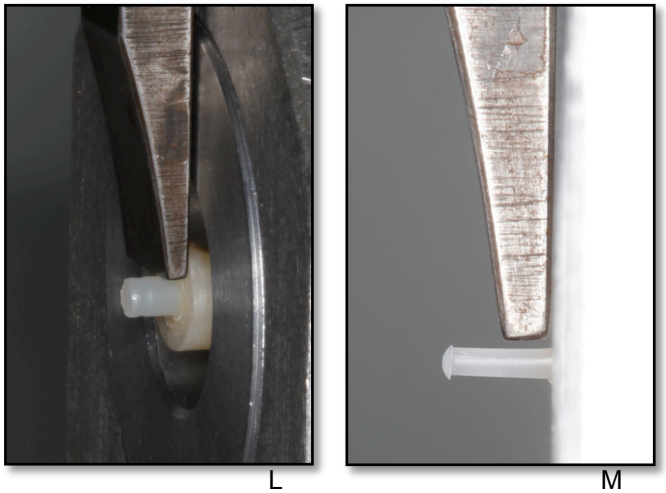


Figure 3: Samples positioned for either shear (L) or microshear (M) test.

Results

The mean bond strength, in MPa (±SD), was 9,02 (±2,07) and 11,68 (±5,79) for the shear and micro-shear test, respectively (table II and figure 4). Concerning adhesion mean values, no significant differences were found between groups (p=0.138), confirming the null hypothesis of this study.

Table II: Results

Method	Bond strength (MPa) mean ± SD	n	Failure mode			
			Adh	CohD	CohR	Mix
SBS	9,02 ± 2,07	**15**	**15**	**0**	**0**	**0**
MSBS	11,68 ± 5,79	**13**	**13**	**0**	**0**	**0**

n: total number of samples tested

Adh: adhesive failure; CohD: Cohesive failure in dentin; CohR: cohesive failure in resin; Mix: mixed failure

However, group 2 exhibited larger standard deviation value and some pre-testing failures, which will deserve some considerations in discussion.

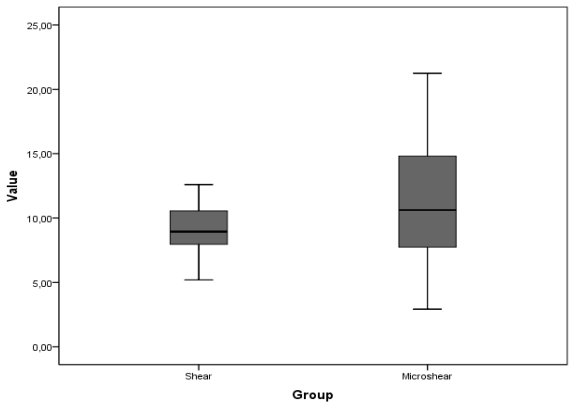


Figure 4: Results

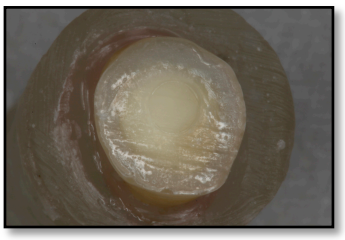


Figure 5: Adhesive failure

Concerning the failure mode all the specimens were classified as adhesive failures apart the studied group (table II and figure 5).

Discussion

Long term clinical performance of adhesives formulations are not routinely feasible, due to time, labour and costs associated. Besides, in many cases, when data becomes available one particular adhesive has already been replaced or modified. Although some restrictions are commonly mentioned to in vitro research, there are so many other valuable reasons to promote an extensive laboratory research in the adhesion field, namely for bond strength measurements. Some notes for guidance regarding in vitro investigations have been published[19-21]. Ideally a sound correlation between in vitro and in vivo findings should be previously established. However, the reliability and validity of bond strength determinations on dentine bonding have been questioned, and laboratory tests have generally been considered to be unreliable[8,22-24] and not predictive of clinical behaviour[25].

A crucial factor in evaluating the usefulness of a specific bond strength test is a thorough awareness of the force distribution and stress patterns involved, which in turn influence the mode of failure[26]. The large spread of results of bond testing protocols have been mainly explained by Finite Element Analysis [4,8,9,16,27-29] where stress distribution can be highly inhomogeneous resulting in stress peaks initiating in dentin or composite rather than in adhesive interface[6]. In addition, almost every possible testing variables like specimen geometry, experimental setup[7-9] , bonded area, preparation tool or operator experience may significantly influence the results[10-14].

Macro bond strength test methods using surface areas larger than 3 mm^2 deliver lower bond strength values and failure mode frequently occurs cohesively in dentin, which does not provide reliable information with regard to the adhesive strength of the bond.[30]

The capability to differentiate local conditions of bonding over the same substrate is definitely one of the advantages of tests using small-sized specimens, such as in microshear and microtensile approaches[31]. Both of these techniques also allow gathering of multiple measurements from one tooth and a more uniform stress distribution, thus supporting the assessment of the actual interfacial bond strength in comparison with conventional shear and tensile tests[3,10].

According to scientific research it would be expected to obtain a higher microshear mean bond strength compared to shear results, however this was not

observed in the present study. A recently review published by Scherrer et al[32] collected bond strength data obtained for six adhesive systems measured by four different test methods (shear, tensile, microshear and microtensile) allowing extensive analysis of results with respect to average bond strength, coefficient of variation and mode of failure related to each method. Concerning PBNT adhesive the overall mean shear and microshear bond strength founded were 17,7 MPa and 20,8 MPa, respectively. Nevertheless, for microshear evaluation only one study was available[33], conversely to the other 3 adhesive systems (Clearfil SE Bond, Single Bond, Scotchbond Multi Purpose Plus) for which at least two microshear studies were reported. For these last materials, the microshear bond strengths were about 1.2 to 3 times higher than shear values[32], which is in the range of the present study where microshear bond strength data showed a mean value 1.3 times higher than the shear method, although no significant differences were found between means.

In the same study, all methods implied on bond strength assessment have shown to produce high ranges of coefficient of variation of analysed results, whereas the shear test displayed the highest coefficient of variation between 24 and 45%[32]. This may be due to the lack of valid standardized test protocols for bond strength testing, which implies that for the same product a broad range of bond strength related values could be found on published data[34]. Besides, FEA demonstrated that shear bond strength tests generates a non-uniform stress distribution, as high tensile stresses are produced by the bending moment at load application, which can be responsible for fracture initiation that may not necessarily be focused at the true interface[4,8,9,16,28]. In addition, Placido et al.[4], concluded that this behaviour can even be more factual when microshear bond strength tests are implied. The authors claims that the use of a non-soluble mold for composite resin build-up sample with a classic diameter of 0.7 mm, in combination with a relatively thicker adhesive layer can lead to the introduction of flaws that may yet result in different stress concentrations at the interface with considerable bending and non-uniform shear loading conditions. Furthermore, it is impossible to confine the adhesive to the area tested, as required by ISO-standard No. 11405[35], which cannot be accomplished in microshear method[5].

Another source of variability relies in the choice of testing assembly. Several different tools can be used to apply the shear force, including wire loops, notched or a chisel-shaped rod. In particular, the use of a wire loop for shear bond-strength tests appeared to concentrate stress more near the interface rather than a chisel-shaped rod who rather causes severe stress concentration at the load application area[28].

Both, shear and microshear bond strength tests showed relatively low values in our study as in Bradna et al. research, which reported a mean shear bond strength and standard deviation of 12,0 ± 3,1 MPa for PBNT[36]. These lower values can be partially explained by two main reasons. The first is related to the depth dentin profile available for adhesion and the second concerns the low elastic modulus of the resin composite used. Several studies showed that bond strength to deep dentin were lower when compared with the achieved at more superficial dentin[37-42]. Deep dentin has a greater number and larger diameter of dentinal tubules per unit area with a concomitant decrease of intertubular dentin and increased peritubular mineralized dentine. Higher permeability, greater water content and lower amount of intertubular dentin is progressively more overt as dentin depth approaches, which may explain the greater difficulty in obtaining effective adhesion[23,37,41]. The composite E-modulus evidenced a positive correlation with bond strength, in which stiffer composites leads to higher bond strength values[5]. Different studies showed a relatively weak but statistically significant correlation between dentin shear or tensile bond strength and composite flexural properties[43,44]. The SDR™, a microhybrid composite defined as a stress decreasing resin is a flowable material with an E-modulus around 9 GPa, which is lower than highest viscosity composite resins, but not necessarily lower when compared to other flowable composite resins[45].

Also, a strong correlation was found between the mean bond strength and the failure mode in which the higher the bond strength, the higher the rate of cohesive failures[13]. Nevertheless, such cohesive failures are rarely seen clinically. In the shear tests, whenever high bond strengths are accomplished it is often not possible to differentiate between the strength of an adhesive and that of the composite or dentin because of the increased likelihood of cohesive failure within the dentin[4,16,28,46]. In this study, all specimens observed were classified as adhesive failures, independent of test method employed, that can be explained not only by the lower bond strength values obtained, but also due to the reduced bonded area accomplished with the use of either the gelatine capsules with 4.9 mm^2 cross-sectional area for SBS and a tygon tube with internal cross-sectional area of 0.5 mm^2 for microshear bond strength.

In this study, concerning adhesion mean values, no significant differences were found between groups. Nevertheless, the standard deviation differences and the pre-testing failures occurred in microshear test justifies some reflections. We must recognize that the reduced bond area makes the technique extremely sensitive converting the operator to a considerable influence on the test result[34], widely varying

for each product.

Another limitation or difficulty in microshear test could be related to the necessity to apply huge forces with scalpel blade, or other instrument, to remove the tygon tube from the composite sample, which can introduce potential defects or stress in the bonding interface. However, there is greater control over the dimensions of the adhesive interface with the tygon tubing[6]. To reduce the technique sensivity and standard deviation influence it would be necessary to improve the number of samples for each group and/or using water-soluble molds to trim the microshear composite samples.

The concerns resulting from the present study are in agreement with some literature, which consider this method as controversial, labor-intensive, and technique-sensitive[32]. Conversely, one author supported that the preparation of specimens for microshear bond test is much simpler[6].

Conclusions

In our study, regarding bond strengths mean values, no significant differences were found between shear and microshear bond strength tests. Nevertheless, microshear bond strength test presented larger standard deviation value and some pre-testing failures. No less important is the fact that all specimens were classified as adhesive failures.

Within the limitations of this in vitro study, it can be concluded that microshear bond test is more labour-intensive and technically sensitive than shear bond strength test. However, more laboratory and clinical studies are necessary to understand the specific restrictions of these techniques. It seems that as a first step, there is a real need for a consensus regarding some standardizing of test protocols.

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