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Literature Review

Analysis and Comparison of Different Bond Strength Tests

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Abstract

In Dentistry, the mechanical tests aim to evaluate the properties and predict the behavior of dental materials, mimicking the real biological conditions and providing methods to be further used. Among the existing tests, tensile and shear tests are highlighted. However, since the 90's, the so-called microtests appear as an alternative to those tests. The advantage of microtests is the reliability of the results. This paper aims to evaluate, through a literature review, the advantages of microtests over the conventional tests, as well as their indications for in vitro simulation of the bond strength between the dental surfaces.

INTRODUCTION

In Dentistry, the mechanical tests aim to evaluate the properties and predict the behavior of dental materials, mimicking the real biological conditions and providing methods to be further used [1]. Although no methodology of mechanical test is capable of simulating exactly all existing clinical variables inside the mouth, the *in vitro* studies enable to evaluate and compare the behavior of different materials at short time and are very useful to create guidelines for clinical practice, mainly if we consider the extremely difficult in executing clinical studies on resistance to fracture of materials [2,3].

With Dentistry advancement and improvement, the adhesive systems demonstrate a considerable evolution over the years. Some tests are used to evaluate the adhesiveness between different materials: tensile, microtensile, shear, and microshear.

The macrotests, very used until mid-90s, have the advantage of practicability. However, van Noort et al. [4] conducted a finite element study and found lack of uniformity of the force distribution along the adhesive interface. This resulted in an excessive stress on the substrate but not on the adhesive interface, which led to a high index of cohesive failures.

The microtests appear to improve the deficiencies of the tests used so far (tensile and shear tests), with advantages of: greater percentage of cohesive failures, low coefficient of variation, possibility of evaluating different areas of the same specimen, calculation of mean and standard deviation on one single tooth, easy SEM evaluation [5].

The microtensile test was introduced in Dentistry by Sano et al. [6] to measure the bond strength and the modulus of elasticity of the mineralized and demineralized dentin. This versatility was not possible with the available methods so far, such as conventional shear and bond strength tests. This first study was conducted to compare the bond strength of normal dentin with the variations, such as: affected and sclerotic dentin; at different areas of MOD preparation; in Class V cavities; among others.

Thus, this study aimed to evaluate through literature review, the bond strength, tensile, microtensile, shear, and microshear tests used in Dentistry, as well as their applicability on the different dental materials.

LITERATURE REVIEW

Many *in vitro* studies used mechanical resistance to fracture tests to compare the materials and evaluate the influence of the experimental variables on the adhesive interface of resin materials and tooth substrate. By the end of the 80's, the most used tests for this purpose, were the shear and bond strength tests. By the end of the 90's, these tests were questioned by the literature. van Noort et al. [4], discussed the need to control and standardize the tests and conducted a finite element analysis to evaluate the effect of the sample size and the conditions of force application and distribution during the shear bond strength tests. The results showed that the force distribution on the adhesive surface was not homogeneous, even on a uniform load. There was a stress concentration on the restoration surface but not on the interface. Also, the stress concentrated on the middle of the sample as the area was reduced.

Della Bona & van Noort [7] questioned the reliability of the shear bond strength tests. The authors executed shear and bond strength tests between ceramic and resin surfaces fixed with adhesive and resin cement. Two sample designs were used: fixed cylindrical ceramic base adhered to a resin cylinder; and the opposite configuration, i.e., fixed resin base adhered to the ceramic cylinder. The authors observed through finite element analysis that even changing the position of the samples, in the shear bond test, the stress concentration occurred always outside the adhesive interface, inside the bases, confirmed by the higher percentage of cohesive fractures on the fixed bases. The bond strength test showed that all fractures were adhesive type. The authors concluded that the bond strength test was more suitable for evaluating the adhesion between resins and ceramics.

Some authors suggest that the analysis of the bond strength microtests demand samples with up to 1 mm^2 of adhesive [8]. For macrotests, the samples should have from 3 to 4 mm^2 [2].

Through finite element analysis associated with shear bond test and fractographic analysis, Verluis et al. demonstrated that the cohesive fractures in dentin occurred because of the biomechanics test itself, resulting in high stress concentrations on dentin. Thus, the authors proved the hypothesis that this test was not suitable for evaluating the bond strength of biomaterials and suggested the development of new technologies to measure the bond strength on the biological interfaces.

Sirisha et al [9,10], analyzed the macro and micro bond strength tests in two literature review studies, namely Part I and II, respectively. The authors concluded that regardless of some inherent deficiencies, macrotests can still be used to evaluate the adhesion of the material to the dental structures, because they are simpler tests (Part I). According to the authors, macrotests resulted in many cohesive failures and overestimated the adhesion values, so that microtests are more reliable (Part II). Prior to the use of bond strength tests, the methodology should be standardized at most, because many factors can interfere in the results. Thus, the comparison of different study results should highlight the test type used, tooth type and substrate, substrate deepness and location, direction of the enamel prisms and dentinal tubules, presence of pulp pressure, smear layer, tooth extraction time and storage, thickness of the adhesive pellicle, dimension of the contact area, mechanical properties of the composite, treatment protocol, thermocycling protocol, type and amount of the load applied, speed and failure type (Part I). Based on all the aspects involving the adhesion tests, the authors affirm that these tests can be used in the laboratorial step to develop new adhesives, but not alone, especially by means of predicting clinical results. Some recommendations are valid when laboratorial tests are used for a higher result relevance: only adhesive or mixed failures (with little involvement of resin or dentin - $<10\%$) should be considered to calculate the bond strength; use of the Weibul statistical test with a minimum of 30 specimens with non-cohesive failures (Part II).

Tensile and microtensile tests

By conducting a meta-analysis, Munck et al. [11], collected bond strength data to identify the primary parameters that may affect the results of the bond tests and report the tendencies of adhesive performance for the different adhesive protocols currently available. The authors verified that two tests are the most used. Among which, microtensile tests have a higher discriminatory power than macrotensile tests. Such fact would explain the current popularity of microtensile tests in research field. Also, the macrotests were not capable of preventing the clinical performance of the materials tested.

Sano et al. [6], analyzed the relationship between the bond strength and the interface area by comparing the results of two adhesive systems (trimming and non-trimming) applied on third molars and submitted to pure tensile and the results of a new methodology so-called microtensile in which the samples were submitted to serial cuts so that each cut had the interface area reduced approximately from 0.25 to 11m^2 (Figue 1). The results showed an inverse relationship between them. The authors concluded that the adhesive area reduction influenced on the bond strength and reduced the cohesive failures. Thus, microtensile test would be an adequate and advantageous methodology to evaluate the adhesive interfaces.

Since most of the materials submitted to bond strength analysis had values ranging from 20 to 30 MPa, Pashley et al. [12], reported that tensile tests guided the force towards the substrate but not towards the adhesive interface, resulting in higher number of cohesive failures. The authors reported that the microtensile test would be a possible solution to evaluate the adhesion under clinically relevant conditions because it enables analyzing bond strength values of up to 70 MPa with small percentage of cohesive failures. The authors highlighted the following advantages of microtensile tests: greater percentage of adhesive failures instead of cohesive failures; possibility of measuring very high bond strength values; possibility of measuring local bond strength; possibility of calculating the mean and standard deviation of a single tooth; possibility of testing very small areas; easy examination in scanning electronic microscopy (SEM).

In other study [13], the authors highlighted that is important to understand that cohesive fractures in dentin observed more frequently in the conventional shear and bond strength tests (up to 80% of cohesive failures when the adhesive bond strength reached 25 MPa) did not mean that the adhesion between resin and tooth substrate were strong enough to surpass the cohesive strength of dentin, which is approximately 100 MPa. According to these authors, the adhesive test type provides stress concentration on localized areas of dentin that surpassed 100 MPa and resulted in cohesive failure, even when the mean calculated bond strength was approximately 25 MPa. In fact, the adhesive interface was not tested regarding the bond strength because prior to its rupture, the dentin underwent cohesive failure due to propagation of the stress concentration.

Macorra & Higueras [14] assessed the influence of the adhesion area variation on the bias of microtensile tests. The study showed that it was not possible to construct specimens with the same adhesion area and variations inside the same study and among the studies is a known covariance in microtensile researches. The authors verified that areas smaller than 1mm^2 exhibited greater result variations, without known cause. A hypothesis would be the Poisson effect on the adhesive layer, accounting for increasing the stress on the external surface; or the adhesive layer thickness is the predominant factor. The authors recommended that if the adhesion area is about 1mm^2, this value can be used as adhesion area and it resulted in clinically understandable outcomes.

Scherrer et al. [15], conducted a systematic review and analyzed the results of laboratorial bond strength studies on six dentinal adhesive systems submitted to conventional mechanical tests (shear and tensile) and microtests (microtensile and microshear). The authors observed that the bond strength values were significantly smaller in the microtensile and microshear bond strength tests, with a significantly smaller number of cohesive fractures than those of conventional tests.

According to Abdalla [16], the technique of specimen preparation is highly sensible and requires careful handling during bonding and cutting to avoid premature loss. Thus, the author recommended to modify the technique by using an adhesive tape to mark the interface area at 1.0mm2, which would avoid the specimen cutting (Figure 2). The results demonstrated that this technique achieved higher bond strength values than those of the conventional technique, with smaller number of cohesive failures.

The design of the specimens was analyzed by Sadek et al. [3]. The authors compare the hourglass shape (trimming technique), which demands the weariness of the adhesive interface on the specimen cuts to be analyzed, to the stick shape (non-trimming technique), in which the specimens achieve the stick shape during the phase of the specimen cutting, not requiring the further weariness of the adhesive interface. By evaluating the parameters of the adhesive area (0.25 mm^2 or 1.0 mm^2), specimens design ("stick" or "hourglass"), and tooth substrate (dentin or enamel), the authors observed that the stick-shape specimens had the mean bond strength values higher than that of hourglass-shape specimens with low percentage of premature failures. This difference occurred more expressively for the enamel than the dentin. The author concluded that the hourglass-shape specimens (trimming technique) were submitted to a higher stress on the adhesive interface during the weariness ("narrowing"), which resulted in cracks seen on SEM, thus reducing the bond strength. The non-trimming technique should be chosen, mainly for enamel, and the cross-sectional area of the specimens should range from 0.5 to 1.0mm^2.

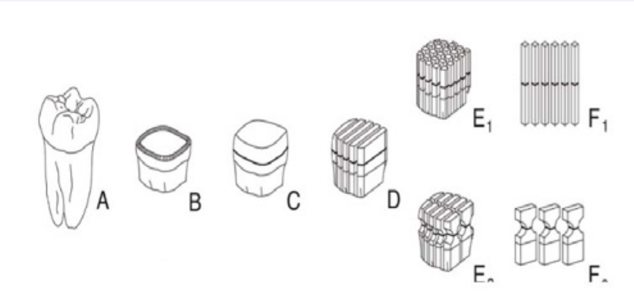

Figure 1 Trimming (E1/F1) and non-trimming technique (E2/F2).

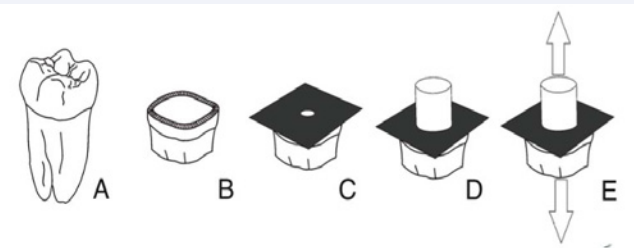

Figure 2 Schematic representation of the microtraction technique proposed by Abdalla. Using an adhesive tape to mark the interface area at 1.0mm^2.

Otani et al. [17], compared the bond strength to Y-ZTP ceramics through tensile, microtensile, shear, microshear, push out, and micro push out tests. The ceramics were divided into groups with silanization and Al_2O_3 sand blasting + silanization. The authors concluded that regardless of the surface treatment type, the microtensile and microshear tests had the greatest values than the macrotests. The tensile tests showed the greatest variability of the results. The method of silica coverage through sandblasting increased the bond strength for all tests.

Shear and microshear tests

According to Placido et al. [18], the shear bond strength test is still very used mainly due to the process simpler than that of the tensile tests. Tensile tests show the difficult to place the specimen, which may alter the result of the fracture load distribution.

Al-Salehi & Burke [19], analyzed 50 studies on bond strength to dentin. According to the authors, 80% of the cases applied shear bond strength tests to evaluate the adhesion. Moreover, the molars were the most used teeth and the waiting time of the tests were mostly after 24 hours. Failures occurred in 42% of the studies. The authors concluded that the studies on adhesion are poor standardized and suggested that better standardization would improve the studies. Burke et al. [20], evaluated 102 studies on bond strength to dentin. According to the authors, 46% of the cases used shear bond tests.

DeHoff et al. [21], analyzed through finite element three different protocols to standardize the shear bond strength tests and evaluate the values, thickness of the adhesive agents, and the conditions of the load distribution on the adhesion area. The authors found a great effect of the stress concentration for all components close to the load application point. The maximum shear stress generally occurred 0.3 mm below the point of force application and then decreased in all directions. According to the authors, approximately 0.5 mm below, the forces are relatively uniform. The authors concluded that no evidence demonstrated that the bond strength is applicable for the clinical performance and the knowledge of the failure points of the test would help in evaluating the possible interferences on the clinical analysis.

Plácido et al. [18], analyzed the shear and microshear tests through finite element. The authors demonstrated that the forces applied on both tests were uneven and may vary according to the specimens' shape, force configuration, and material properties. The forces may not be necessarily adhesive. According to the study, the nominal values did not represent the values applied on the tests. Thus, the authors found that although the shear bond forces were applied, the fractures may have occurred due to tensile stress prior to fracture, which were higher in the microshear tests.

Although shear and microshear tests are standardized by ISO TR 11405, the process may vary, which may alter the possible results. According to the studies of DeHoff et al. [21], the use of chisel causes considerable stress peaks on the area of force application. However, the use of a wire loop better distributes the forces on the adhesive interface.

Aiming to evaluate the cytotoxicity and bond strength among four adhesive system generations, da Silva et al. [22], conducted the microshear test with 0.2mm steel wire surrounding the adhesive area, instead of the conventional beveled blade. The authors based this study on that of Foong et al. [18], in which they verified that the steel wire was the method with higher reliability of results than that of the beveled blade, with a variation coefficient 50% smaller. The authors concluded that the steel wire was capable of distribute the shear bond stress more uniformly, because it surrounds half of the circumference of the composite resin stick and it is easier to place on the adhesive interface.

Heintze et al. [23], evaluate the variability of the results of the bond strength tests (tensile, microtensile, shear, and microshear) on the adhesive systems by correlating this variability to the clinical parameters on the marginal degradation of the restorations. The authors concluded that the high variability of the results of the bond strength tests highlights the need to establish levels of clinical acceptance for each test.

DISCUSSION

Over the last years, the microtensile test has been employed in studies on bond strength of materials because allegedly it enables to solve some problems of the conventional shear and tensile tests, mainly about the reduction of cohesive failures that makes difficult to measure the real values. Accordingly, the literature evidently shows that cohesive failures are significantly smaller after microtensile, mainly when compared with shear bond tests. This aspect is extremely important when comparing the bond strength of the materials because cohesive fractures occur prematurely, before the occurrence of the maximum force required to rupture the adhesive interface.

This was highlighted by Schreiner et al. [24], who clearly show that the microtensile test enabled the detection of a difference in the bond strength of one of the adhesive systems, which was not possible to be identified by the shear bond test. Thus, the microtensile test is more reliable to evaluate the bond strength of adhesive interfaces, mainly when the values are higher than 25 MPa. This affirmation is in line with the literature review conducted by Munck et al., who concluded that microtensile tests are more realistic for a clinical extrapolation. On the other hand, Sirisha et al reported after a large literature review that the laboratorial tests alone are not capable of reaching results that can be transitioned to clinical practice.

According to El Zohairy et al. [25], the bond strength values expressed in MPa are calculated from the force applied at the moment of rupture on the interface area (force/area). Thus, it is expected that by reducing the area (denominator) the final bond strength value will be smaller, even if the same force is applied. Accordingly, one should carefully analyze the results of microtensile studies with higher bond strength values that exhibit this as a method advantage over the shear and tensile tests, which always have a higher area. The study of the adhesion area influence on the bond strength results showed that areas smaller than 1mm^2 had greater result variations without known cause. Accordingly, these authors recommended that if the adhesion area is around 1mm^2, this value is acceptable as adhesion area, resulting in clinically understandable outcomes [14].

A problem inherent to the construction of the specimens for microtensile tests is the bond strength smaller than 5 Mpa. In these cases, the cutting stress causes greater number of premature failures and consequently makes difficult to obtain sticks and bond strength values. Thus, for these situations, the microtensile test would not be the most effective method for this analysis [13].

Some authors [25,26] suggested to change the fixation of the sticks, which preferably should be by the ends to avoid the induction of the lateral components of the force that would generate stress concentration and result in premature fracture of the interface.

According to Abdalla [16], the preparation technique of the specimens is highly sensible and requires careful handling during fixation and cutting to avoid premature loss. Thus, the author recommended to change the technique employing the adhesive tape to mark the 1.0mm^2 area and avoid the need of trimming the specimens. The results demonstrated higher bond strength values than that of conventional tensile tests and lower occurrence of cohesive failures.

The literature recommended that the studies comparing these methodologies include in the results the values of the force applied at the moment of the rupture, not only the tension (MPa). Thus, the comparison of the values would be more valid [27].

The literature shows consensus on the high rates of cohesive failure during tensile and shear bond tests, probably because the force is concentrated on the substrate, resulting in cohesive failure of dentin, before that of the adhesive system [4,7]. Notwithstanding, Placido et al. [13], stated that the cohesive failures would indicate the improvement of the adhesive systems. Because of that, Davidson [28] et al., suggested that the adhesive systems should not be improved anymore.

Even with process variations, the shear bond tests are still the most used, according to the literature, mainly due to the easy execution. DeHoff, et al. [21], suggested that the studies with finite elements would help in identifying the failures of force application during the tests.

According to Heintze et al. [23], the literature lacks consensus on the levels of individual acceptance for each bond strength test and the results should be correlated with other *in vitro* tests, such as marginal degradation. Also, the adhesiveness should be compared before and after aging to obtain a correlation between the results and clinical acceptance parameters.

Before executing any force test, one should ideally standardize and understand how the nominal bond strength is related to the stress distribution generated during the test and the clinical performance. Sudsangiam & van Noort [29] stated that “the belief that the bond strength results will be valid and consistent is inherent to the standardization process”. These same authors affirmed that until the relationship between the bond strength test and the clinical performance is fully understood, one should adopt the following steps: (1) adoption of terms and definitions universally accepted, (2) standardized reports of handling and construction of the specimens, (3) inclusion of positive and negative controls during the test, (4) standardized reports of set-up and mechanical tests, and (5) complete reports or access to complete data set.

The careful documentation and communication of how macro or microtests are conducted would help in understanding the strong and weak points of the different methods and decrease the knowledge gap between the laboratorial and the clinical practice. The new methodologies of preparation and fixation of the specimens are very promising, but further studies are necessary before drawing conclusions. Regardless of the type and size, the bond strength tests are useful tools to evaluate new adhesive protocols and investigate the experimental variables. The tests based on the adhesive force are still limited. Thus, due to the inherent limitations, if one aims to measure the bond strength as the material's property, the mechanical study of the fracture should be performed because it is easy to execute and more successful.

CONCLUSION

It can be concluded that:

- The microtensile test has smaller rate of cohesive fractures, mainly at adhesive interfaces with forces higher than 20MPa;

- Further studies are necessary to correct the failure of the suggested bond strength tests;

- A better documentation and communication of how the bond strength tests are conducted would help in understanding the limitations and would decrease the knowledge gap between the laboratorial and the clinical practice.

- The bond strength tests are subjected to many variables during laboratorial tests, and it is difficult to compare the results of different researches. The most adequate manner of interpreting the results found in the literature is to evaluate a tendency towards the bond strength of the materials and not the numerical comparison of the results.

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