Parameters that influence microtensile bond testing of adhesive systems

Parâmetros de influência no ensaio de microtração de sistemas adesivos

Estevam Carlos de Oliveira LULA[1]
Taissa Helena Martins LEITE[2]
Cláudia Maria Coelho ALVES[1]
Ivone Lima SANTANA[1]
Ana Maria Lima ALMEIDA[1]
José Ferreira COSTA[1]

ABSTRACT

The aim of this study was to provide a critical review of the literature regarding factors that can interfere with microtensile test results for enamel and dentin adhesive systems. Primarily, reports in English, Spanish, or Portuguese that were published between July 1994 and September 2009 and are catalogued in MEDLINE and BBO were used. Additionally, we compiled relevant articles found in the references of these articles and dissertations and theses available in electronic databases of Brazilian universities that examined factors that can influence implementation of the microtensile test at each stage. The search strategy included searching for the following key term groups: microtensile and test; microtensile and assay; microtensile and test and parameters; microtensile and test and factors; microtensile and specimen; microtraccón and especimen. We reviewed 25 selected articles and found that they showed that even after adjustment of test parameters, changes found in the dentin could be responsible for variations observed amongst results. This influence could potentially be reduced by using the cohesive strength value of dentin adjacent to the adhesive interface as a standard for comparison, but more studies are needed to confirm whether such an approach would be reliable. Data analysis methodology should be taken into account when comparing studies.

Indexing terms: Dental materials. Dentin-bonding agents. Materials testing. Tensile strength.

RESUMO

O objetivo deste estudo foi realizar uma revisão crítica da literatura sobre os fatores que podem interferir nos resultados do teste de microtração de sistemas adesivos ao esmalte ou dentina. Foram utilizados trabalhos publicados nas bases de dados eletrônicas MEDLINE e BBO, em inglês, espanhol ou português, entre julho de 1994 e setembro de 2009, referências destes artigos, além de dissertações e teses disponíveis em bancos de dados eletrônicos de universidades brasileiras enfocando o estudo das variáveis que podem influenciar cada fase da execução do ensaio de microtração. A estratégia de busca incluiu os termos: microtração e teste; microtração e ensaio; *microtensile* e *test* e *parameters*; *microtensile* e *test* e *factors*; *microtensile* e *specimen*; *microtracción* e *espécimen*. Os 25 artigos selecionados demonstraram que mesmo após a padronização de parâmetros do ensaio, as alterações encontradas na dentina podem ser responsáveis pela variação do resultado. Uma maneira de reduzir esta influência seria utilizar os valores de resistência coesiva da dentina adjacente à interface adesiva como padrão de comparação, porém mais estudos são necessários. Aspectos relativos à análise dos dados devem ser levados em consideração quando se compara os estudos.

Termos de indexação: Materiais dentários. Adesivos dentinários. Teste de materiais. Resistência à tração.

INTRODUCTION

With the growing importance of bonding agents in clinical restoration work and the rise of new products in the dental market, it has become necessary to establish comparative parameters for the various bonding systems available. Clinical data obtained from randomised and controlled trials is the most reliable source for evaluating bonding systems[1]. However, it is difficult to apply this method, since longitudinal studies are needed to obtain meaningful information[2]. Therefore, laboratory tests have been refined such that, although they cannot predict the clinical outcome of bonding systems, they can provide useful information about how bonding systems interact with dental substrates and allow us to compare the same characteristics across different materials[3-4].

Although there is no test that is able to determine all of the mechanical properties of dental materials, the microtensile strength test has grown in prominence at

[1] Universidade Federal do Maranhão, Curso de Odontologia. Av. dos Portugueses, 1966, Bacanga, 65085-580, São Luís, MA, Brasil. Correspondência para / *Correspondence to*: ECO LULA. *E-mail*: <estevamcarloslula@hotmail.com>.
[2] Pontifícia Universidade Católica de Minas Gerais, Faculdade de Odontologia. Belo Horizonte, MG, Brasil.

dental bonding research centers in the last 15 years because it is purported to overcome some of the limitations of traditional tests of shear strength and tensile strenght[5-8].

However, despite the microtensile strength test[7] representing the best stress distribution assessment available to date, the microtensile strength test results in the literature still vary widely, even when the same bonding system is compared across similar experimental conditions[9-10]. This variation may stem from the absence of standardization of microtensile strength testing methodology. Thus, elucidating the parameters that influence microtensile strength test results is important for correctly interpreting associated findings in the literature and obtaining a clear understanding of the research. Therefore, we conducted a critical review of the literature related to factors that might affect microtensile strength test results for enamel and dentin bonding systems.

METHODS

We searched for publications in the MEDLINE and BBO electronic databases in English, Spanish, and Portuguese that were published between July 1994 and September 2009. Specifically, we focused our search on studies that examined variables that might influence each phase of execution of the microtensile strength test. The following terms searches were conducted: microtensile and test; microtensile and assay; microtensile and test and parameters; microtensile and test and factors; microtensile and specimen; microtensile strength and specimen. We also used references from the articles retrieved from these databases as a research source, as well as dissertations and theses from the electronic databases available at Brazilian universities.

Two independent and blind evaluators selected studies based on their titles and abstracts, using the following inclusion criteria: studies about the effect of variation in parameters of microtensile strength assessments of adhesive resistance and stress distribution. The exclusion criterion was: comparative studies of adhesive materials or techniques. The evaluators solved disagreements through consensus.

RESULTS

Employing the search strategy described above, we identified 204 studies, of which 179 were excluded based on the exclusion criterion. The remaining 25 studies, which we included, evaluated the following parameters: 1) in relation to specimens: geometric aspects[5-6,9,11-21] cutting speed[22-23], cutting method[24], wear-and-tear and integrity method after cutting[23], storage time[22], inclusion of specimens with premature failure[24], imperfection during manufacture[16], bonding surface area[5,19-20,25], wear-and-tear method for the dentin[26], form of filling and fixing[6,15,19;27]; 2) storage time of teeth used in the assay[28]; 3) angle of the bond interface[29]; 4) incline of the dentin walls[30]; 5) variation in adhesive resistance between the teeth or within the same tooth[31]; 6) adhesive thickness[30]; 7) use of the cohesive resistance of dentin near the bonding interface as a standard of reference[18]; 8) loading speed during the test[19,32]; 9) thickness of remaining dentin[19]; 10) imperfections in the adhesive layer[16]. The characteristics of the included studies are summarized in Chart 1.

DISCUSSION

Laboratory tests play a fundamental role in evaluating bonding systems as new products are being added to the market constantly[8]. All of the current tests used to evaluate the strength of the bond between an adhesive and dentin involve the application of force to the interface area. Therefore, it is important to understand the test mechanics and stress distribution in the interface[3-4,33]. The microtensile strength test was developed to overcome certain deficiencies of shear strength and tensile strengh tests[7,34], especially the inability to use these methods to measure bond resistance with the most recent adhesive systems[5]. This test differs from the others in terms of the dimensions of the adhesive area used. In shear strength and tensile strengh tests, an area of approximately 4-mm^2 is used, whereas in the microtensile strength test, the area used is in the range of 0.5-1.5 mm^2. This reduction in test area yields a number of advantages: it reduces the number of teeth needed for research; it reduces the chance of there being defects generated by fractures in the substratum; it allows for adhesive faults to be obtained in the majority of samples; and it allows adhesion to be measured in small areas[33-34].

Since its introduction in 1994, use of the test has been growing among research groups working on adhesion. Several researchers have made suggestions for how to obtain a more precise test that would optimize the stress distribution on the adhesive interface and allow comparison across studies. For example, adhesive resistance and stress distribution on the resin-dentin interface could be improved by: storing teeth to be used as sources of specimens for no more than 30 days in thymol or formalin[28]; standardizing the cutting speed, giving preference to higher speeds, so as to produce less disk oscillation and reduce specimen surface damage[22-23]; cutting with diamond-impregnated thread to maintain specimen integrity[24]; using stick-shaped specimens at least 1.5 mm thick in enamel bonding tests[6]; testing the bonding to dentin using stick-shaped[9,14,17,23], dumbell[21] or hourglass-shaped specimens[7] with a 1-mm^2 adhesive area and a 1-mm radius of curvature for the notch[35]; fixing specimens at both ends[6,15,27]; storing specimens for no more than a week in distilled water[22]; using a notched jig[19]; using sandpaper to wear dentin instead of burs[26]; and not using specimens with steeply inclined bond interface[29].

However, even with standardization of some microtensile strength test parameters, the mechanics of laboratory tests on biological substrates with enamel and dentin remain highly variable, especially with respect to dentin's dynamic characteristic that is, its differences in mineral content, tubular density, and collagen orientation[36]. The range of properties related to the interaction of adhesive materials with dentin reported in the literature stems not only from different evaluation methods, but also from differences in the intrinsic structure of dentin, which is sensitive to humidity and demineralization. Adhesive resistance is higher in surface dentin than in deep dentin, and higher in normal dentin than in dentin underlying excavated caries or sclerotic dentin[37].

Bovine teeth or human third molars have been used to reduce substrate variation[4,10]. However, there is no way to predict the level of mineralization or permeability of dentin, the depth of the preparation, and the amount of change inflicted upon dentin due to the effects of storage and other factors[4,10,38]. The precise intrinsic resistance of dentin varies from the enamel-dentin junction to the area near the pulp because of pre-existing defects in its structure, which function as "amplifiers" for the stress promoted by the test[39].

De la Macorra and San-Nicolás[18] and Perálvarez-Aguilera et al.[40] suggest that substrate variation in testing can be minimized by using the cohesive resistance values for the dentin adjacent to the adhesive interface as a standard for comparison in microtensile strength assays. They made notches in the dentin that were 1.5 mm deep and wide, with an inter-notch distance of 1.5 mm. Next, they made a resin block and obtained sticks from the notch area (to measure adhesive microtensile strength) and from the region between the notches (to measure cohesive microtensile strength). This method has enabled dentin's cohesive resistance and adhesive resistance to be evaluated at equivalent depths in the same tooth, under the same conditions of preparation. Thus, since we do not know the ideal mechanical resistance for an adhesive interface, this approach should give a sample that is at least close to the proposed substitution tissue[18,40]. Nevertheless, we found only two studies[18,40] that evaluated how this method affected test results, limiting our ability to assess the benefits of the method relative to the conventional microtensile strength test.

There are other areas related to data analysis that may contribute to variations in microtensile strength test results and that should be taken into consideration when comparing studies[8,23], such as including specimens with premature failure, including outliers (discrepant values), in the statistical treatment and the definition of the experimental unit[8,31]. Data means and distributions are affected when specimens with premature failure are included in the calculations. Since such specimens have negligible adhesive resistance values, their inclusion alters data normality and may preclude the use of parametric tests[8]. On the other hand, if these specimens are excluded, adhesive resistance may be overestimated[23-24]. Similarly, including outliers can shift data patterns and affect research conclusions.

The definition of the experimental unit (i.e., teeth or sticks) can also affect the results. When multiple sticks from the same tooth are considered to be an experimental unit, a statistical flaw ensues. Sticks obtained from the same tooth cannot be considered independent samples, without increasing variation in adhesive resistance[31]. For this reason, each tooth should be considered a sample unit, and mean values calculated for all specimens coming from the same tooth should be analyzed.

Adhesive resistance test results do not provide a direct reflection of a property of the material itself, but rather serve as an index of the behavior of the bonding system in the particular configuration of the test. Changing any test component can lead to completely different results[38]. Hence, interpretation of microtensile strength test results requires careful evaluation of the methods used and the type of analysis performed.

Chart 1. Summary of studies included in the review.

Reference	Parameter(s) evaluated	Main results
Sadek et al.[5]	S shape (stick, hourglass) with bonding areas of 0.5 mm^2 or 1 mm^2	Notching → Enl defects, reduced AR. ↓ bonding area → ↓ AR
Meira et al.[6]	FEA in single-material, hourglass S, ranging from fixation point in jig to fixation area height, S width, and notch C_{rad}	Notch C_{rad} affected AR. Fixing S on both sides ↓ concentration of stress
Bianchi[11]	Hourglass-shaped S: thickness (0.5, 1, 2, 3, 4 mm), neck width (1, 2 mm), filling mode (one, two sides), notch shape (acute, round)	↑ thickness, filling on one side or an acute notch reduced AR
Betamar et al.[12]	Hourglass-shaped S (circular, parabolic, and gentle curve). FEA	AR was greatest for circular, followed by parabolic and gentle curve. StDi followed the reverse pattern
Betamar et al.[13]	S was stick, hourglass, or notched; FEA	No difference between shapes with same bonding system
Chen et al.[14]	Hourglass vs. stick S shape	Cohesive failure in majority of conventional S; stick-shape had higher AR and SD and majority of failures
El Zohairy et al.[15]	Variation in thickness and width in the area of the S fixation. FEA	↑ thickness → ↓ AR; no effect of width. Fixing S at both ends improved StDi
Ghassemieh[16]	S shape (hourglass, stick, or notched) and imperfections during the manufacture. FEA	Hourglass shape yielded ↓ AR and variance; others did not differ. Adhesive bed defects affected AR
Goracci et al.[17]	Substratum (enamel/dentin), shape (stick/hourglass), and thickness	↑ AR in dentin S (stick shaped or not thick). Avoid carving in Enl. Hourglass S transverse area should be ≤ 1.0 mm^2
de La Macorra & San-Nicolás[18]	Use of cohesive resistance of Dn adjacent to adhesive interface as a reference	No difference in adhesive vs. cohesive microtraction
Poitevin et al.[19]	S fixation, geometry, area of adhesion, thickness of remaining Dn, jig placement method, loading speed	S orientation relative to interface influenced results and failure pattern. Notched jig → ↑AR. S w/o grooves and w/ square transverse section ≥1.0 mm^2 had ↑ AR. Dn thickness affected AR contrary to placement shape and loading speed
Phrukkanon et al.[20]	Transverse area (cylindrical/rectangular) and adhesion area FEA	Adhesion areas ≤1.1 mm^2 had ↑AR. Transverse area shape had a negligible influence
Soares et al.[21]	S shape (stick, hourglass, notched) and filling (posterior, superior, lateral; all) FEA	Increase in n. filled sides improved StDi. Notch-shape produced more uniform StDi
Reis et al.[22]	S storage time (distilled water), cutting speed	Optimal AR obtained with 1 wk storage, 2.6 m/s cutting
Sadek[23]	Cut speed (100, 200, 400 rpm); S integrity	Dentin greater integrity than enamel, esp. at low speed
Sadek et al.[24]	Cutting equipment (diamond disk/thread), S shape (stick/hourglass), inclusion of S w/ premature failures	Only inclusion of S w/ premature failures reduced AR of Dn. In Enl, cutting w/ disk, hourglass shape, and inclusion of S with failures → ↓ AR
Phrukkanon et al.[25]	Area of adhesion	Area < 1.2 mm^2 → ↑ AR vs. ≥2 mm^2, dentin-adhesive failure
Sattabanasuk et al.[26]	Dn stressing method: 120-, 400-, 1200-grain sandpaper, diamond burs; carbide bur	Diamond bur produced more compact smear level and adhesion compromise than sandpaper. Carbide bur → low AR
Poitevin et al.[27]	Flat or notched jig fixation of S	Notched jig had ↑ AR, better StDi when S fixed on both ends
Santana et al.[28]	Storage of teeth (0.2% thymol; 10% formalin; 0.2% sodium azide) for 7, 30 and 180 days	Thymol and formalin had a negative effect on AR when teeth were stored for 180 days
Silva[29]	Bond interface angle; FEA	Increase in interface incline reduced AR and stress
Pazzinato[30]	Incline of Dn walls and Dn variation	// vs. ⊥ to gravity and cervical vs. occlusal Dn no effects on AR
Loguercio et al.[31]	Intra- and inter-S variation in AR	Greater variation within tooth than between different teeth
Reis et al.[32]	Loading speed (0.1, 0.5, 1, 2, 4 mm/min)	No effect on AR
Meira et al. [35]	S shape (stick, hourglass) and loading conditions. FEA	Stick concentrates stress outside adhesive interface and cohesive fractures. Hourglass, 1-mm C_{rad} → favorable stress

Note: StDi - stress distribution; FEA - StDi by finite element analysis; S - specimen; Dn - dentin; Enl - enamel; AR - adhesive resistance; C_{rad} - radius of curvature, SD - standard deviation; // - parallel; ⊥ - perpendicular; w - with; w/o - without; ↑ - increased; ↓ - decreased; → - yielded.

CONCLUSION

Examination of the results of the studies reviewed herein led us to conclude with confidence that microtensile strength test results can be influenced by several parameters related to the specimen, the bond interface, the adhesive layer, and the substratum. Even after standardizing test parameters, variability in dentin may be responsible for variations in results. Using the cohesive resistance values of dentin adjacent to the bond interface as a standard for comparison may minimize substratum variation, although further studies are needed to confirm the validity of this approach. Other aspects such as inclusion of specimens with premature failure, inclusion of outliers, and the definition of experimental units need to be considered when comparing results across studies.

Collaborators

ECO LULA, THM LEITE, CMC ALVES, JF COSTA, IL SANTANA, AML ALMEIDA and JF COSTA selected the articles and were involved in writing the study.

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