LUÍS HENRIQUE ARAÚJO RAPOSO

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"AVALIAÇÃO CRÍTICA DOS PARÂMETROS DE ENSAIOS MECÂNICOS ENVOLVENDO MATERIAIS ODONTOLÓGICOS - ANÁLISE LABORATORIAL E POR ELEMENTOS FINITOS"

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UNIVERSIDADE ESTADUAL DE CAMPINAS
FACULDADE DE ODONTOLOGIA DE PIRACICABA

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Orientador: Prof. Dr. Lourenço Correr Sobrinho

Doctorate thesis presented to the Dental Materials PostgraduationProgramme of Piracicaba Dental School of the State University of Campinas to obtain the Ph.D. grade in Dental Materials.

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“Algo só é impossível até que alguém

duvide e prove o contrário.”

Albert Einstein

RESUMO

Ensaios mecânicos laboratoriais são essenciais no estudo e desenvolvimento dos materiais odontológicos restauradores, como forma de predizer o comportamento clínico dos mesmos frente às variadas condições existentes no meio oral.Apesar das condições encontradas *in vivo* dificilmente serem inteiramente representadas *in vitro*, o completo entendimento dos fatores que impactam o protocolo dos testes é determinante na análise dos resultados. Entretanto, muitos dos testes utilizados para caracterização dos materiais restauradores não são realizados nos padrões necessários, levando a resultados ambíguos para materiais similares, além de dificultar a comparação eimplementação dos mesmos. O objetivo deste estudo foi avaliar o efeito de diferentes parâmetros empregados em ensaios mecânicos utilizados no teste de materiais odontológicos restauradores para obtenção de propriedades mecânicas ou verificação da resistência de união. Assim, variadas configurações do ensaio de flexão de três pontos e do teste de microcisalhamento foram analisadas de maneira crítica. Pode-se concluir que as modalidades de ensaios mecânicos testadas necessitam de melhor padronização para que os resultados obtidos apresentem menor discrepância e consequentemente maior representatividade clínica. A maior padronização dos ensaios mecânicos utilizados na caracterizaçãodos materiais odontológicospermite melhor compreensão do comportamento mecânico dos mesmos, levando a um desenvolvimento mais

controlado destes produtos, o que por sua vez, resultará em maior qualidade dos procedimentos reabilitadores.

Palavras-chave: Cerâmicas, Ensaios mecânicos, Materiais dentários, Análise por elementos finitos, Propriedades mecânicas, Resinas compostas

ABSTRACT

Mechanical tests are essential for the study and development of restorative materials and to predict their clinical behavior facing the numerous conditions existing at the oral environment. Despite the situations found in vivo are hardly represented completely in vitro, the full comprehension of the factors that can affect the testing protocols is important for the analysis of laboratory assays. However, most of the tests used for characterizing restorative materials are not performed in the adequate manner, leading to ambiguous results for similar materials and challenging comparisons between materials, besides impairing their improvement. On this way, the present study aimed to evaluate the influence of different testing parameters employed in mechanical tests for obtaining the mechanical properties of dental materials or to check their bond strength. Thus, different three-point bending and microshear designs were evaluated. It was concluded that the mechanical testing modalities studied need better standardization in order to produce results with minimized discrepancies and consequently increased clinical importance. Well standardized mechanical tests for dental materials testing provide better understanding of their mechanical behavior, allowing more controlled development of these products and resulting in an increased quality for rehabilitative procedures.

Keywords: Composite resins, Ceramics, Dental materials, Finite element analysis, Mechanical properties, Mechanical tests.

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INTRODUÇÃO

A odontologia adesiva tem se aprimorado cada vez mais com o desenvolvimento de novos materiais e técnicas restauradoras, resultando em procedimentos restauradores que possuem excelente estética aliada à menor complexidade e maior previsibilidade clínica. Diversos ensaios mecânicos têm sido aplicados rotineiramente na avaliação das propriedades e caracterização do comportamento físico-mecânico dos materiais odontológicos. Esses testes são essenciais para o estudo e desenvolvimento dos materiais restauradores, possibilitando predizer aproximadamente o comportamento clínico dos mesmos frente às variadas condições existentes no meio oral(Garoushi *et al.*, 2012).

Os testes mais comumente empregados na literatura para avaliação de materiais odontológicos restauradores são voltados para a verificação da qualidade e resistência de união de sistemas adesivos/cimentos resinosos à materiais restauradores/tecidos dentários (Armstrong *et al.*, 2010). Diversos ensaios destinados à caracterização das propriedades físicas e mecânicas dos materiais restauradores odontológicos são também aplicados com frequência. Entretanto, muitos destes testes não são executados de forma padronizada nas diferentes avaliações laboratoriais disponíveis (Ghassemieh, 2008; Armstrong *et al.*, 2010; Raposo *et al.*, 2012).

Dos vários métodos para mensuração da energia de fratura de um material, o teste de flexão de três pontos apresenta o mérito de ser simples e confiável, oferecendo resultados válidos quando a falha se propaga de modo quase-

estático(Cooper, 1977). O teste de flexão de três pontos é também comumente utilizado na caracterização de materiais restauradores odontológicos, como as resinas compostas e outros, por sua aparente simplicidade e praticidade, oferecendo informações valiosas como a resistência à flexão ou a tenacidade à fratura dos mesmos (Christiansen *et al.*, 1974; Cooper, 1977). Existem textos técnicos que normatizam a execução deste teste (ISO4049, 2009; ISO178, 2010), sendo que o mesmo consiste de barras retangulares ou cilíndricas posicionadas sobre dois apoios com uma carga axial aplicada perpendicularmente ao seu centro, levando primariamente, ao desenvolvimento de tensões de tração e compressão no espécime. No entanto, não existe consenso a respeito da parametrização e utilização do ensaio de flexão de três pontos na literatura odontológica envolvendo o teste de materiais restauradores (Yap & Teoh, 2003; ISO4049, 2009; Boaro *et al.*, 2010; ISO178, 2010; Pick *et al.*, 2010; Oliveira *et al.*, 2012).

Modificações nos parâmetros deste teste são comumente realizadas como forma de se ajustar a configuração do ensaio de flexão de três pontos de acordo com a conveniência do material ou necessidades da pesquisa, muita vezes sem que normas técnicas sejam seguidas previamente (ISO178, 2010). Alterações nas geometrias, dimensões, pontas aplicadoras, suportes, distância entre apoios, dentre outras, podem provocar alterações nos campos de tensões que ocorrem nos espécimes em teste, levando à grande variação dos resultados, o que torna comparações entre diferentes estudos e materiais um grande desafio (Alander *et al.*, 2005; Garoushi *et al.*, 2012). Além disso, algumas aplicações que utilizam a

configuração do ensaio de flexão de três pontos como base podem exceder as indicações do mesmo em determinadas situações (Cooper, 1977).

Em relação à avaliação da qualidade e resistência de união de materiais restauradores à diferentes substratos, os testes convencionais de tração e cisalhamento têm sido utilizados há alguns anos, embora tenha sido demonstrado que os resultados obtidos por essas metodologias não representem com fidelidade a resistência de união dos materiais testados (Van Noort *et al.*, 1989; Van Noort *et al.*, 1991). Isso ocorre pelo fato dos espécimes utilizados nesses testes serem mais propensos a ter falhas incorporadas na interface adesiva ou nos substratos devido às maiores dimensões dos mesmos (Griffith, 1921). Além disso, pelo modo como os espécimes são carregados e pela própria geometria destes, tensões não uniformes podem ser induzidas nas regiões de interesse, levando a grandes variações nos resultados obtidos (DeHoff *et al.*, 1995; Versluis *et al.*, 1997; Tantbirojn *et al.*, 2000; Armstrong *et al.*, 2010).

Na tentativa de superar as limitações destes testes, foi proposta a avaliação da resistência de união interfacial em áreas adesivas reduzidas (1,0 mm^2) utilizando o teste de microtração (Sano *et al.*, 1994). Com essa metodologia foi possível a obtenção de vários espécimes a partir de um único dente, além da possibilidade de mensuração da resistência adesiva em diferentes regiões do substrato dental. As vantagens desta metodologia na avaliação da resistência de união de diferentes materiais foram ressaltadas por inúmeros autores (Pashley *et al.*, 1995; Pashley *et al.*, 1999; Armstrong *et al.*, 2010; Raposo *et al.*, 2012) e atualmente, este é o teste laboratorial mais comumente utilizado para este fim.

Entretanto, a popularização desta metodologia permitiu que inúmeras modificações fossem introduzidas (Pashley *et al.*, 1999).

Assim como ocorreu com o teste de microtração(Armstrong *et al.*, 2010; Raposo *et al.*, 2012), a constante utilização do ensaio de microcisalhamento (Phrukkanon *et al.*, 1998)levou à proposição de modificações na abordagem inicial sugerida para esta metodologia (Shimada *et al.*, 2002; Marchesi *et al.*, 2012). Desta forma, materiais similares avaliados em diferentes configurações deste teste podem apresentar resultados conflitantes entre si. Alguns parâmetros importantes do teste de microcisalhamento são a forma de posicionamento dos espécimes e a direção do carregamento aplicado sobre os mesmos durante o ensaio. Da mesma forma, as pontas de carregamento também podem influenciar os resultados de resistência de união obtidos utilizando o microcisalhamento.

Independente do teste empregado para verificação da resistência de união existe grande variação nos dados obtidos laboratorialmente(Scherrer *et al.*, 2010; Roeder *et al.*, 2011). Esse fato pode ser mais bem compreendido por meio de análises utilizando o método de elementos finitos, nas quais se observaacúmulo de tensões nas regiões de interesse em diferentes testes devido a variáveis envolvendo geometria, modo de carregamento, propriedade dos materiais e forma de preparo dos espécimes (Versluis *et al.*, 1997; Tantbirojn *et al.*, 2000; Meira *et al.*, 2004; Silva *et al.*, 2006; Placido *et al.*, 2007; Coelho *et al.*, 2008; Ghassemieh, 2008; Neves *et al.*, 2008; Soares *et al.*, 2008a; Soares *et al.*, 2008b; Neves *et al.*, 2009; Armstrong *et al.*, 2010; Roeder *et al.*, 2011; Raposo *et al.*, 2012).

Assim, é aceitável presumir que diferentes parâmetros de teste, resultem

em distribuições de tensões diversificadas nos espécimes e que essas diferenças produzamresultados conflitantes também laboratorialmente. Portanto,é importante a análise de diferentes modalidades de ensaios mecânicos empregados na verificação das propriedades e caracterização de materiais odontológicos por meio de avaliações numéricas e laboratoriais como meio de se indicar a forma mais adequada para a execução de tais testes, buscando maior padronização e comparabilidade entre as diferentes avaliações realizadas envolvendo os materiais restauradores. Deste modo, se faz relevante a avaliação da real influência de diferentes parâmetros envolvendo a execução dos ensaios mecânicos de flexão de três pontos e de microcisalhamento.

Os objetivos do presente estudo *in vitro*, composto por três artigos científicos, foram:

1. Avaliar os parâmetros do teste de flexão de três pontos na mensuração das propriedades mecânicas de resinas compostas (Capítulo 1);
2. Verificar a aplicabilidade do teste de flexão de três pontos associado à extensometria na obtenção das propriedades mecânicas dos materiais odontológicos (Capítulo 2);
3. Avaliar diferentes configurações do teste de microcisalhamento na distribuição de tensões na interface adesiva dos espécimes (Capítulo 3).

O presente trabalho é apresentado no formato alternativo de tese de acordo com as normas estabelecidas pela deliberação 002/06 da Comissão Central de Pós-Graduação da Universidade Estadual de Campinas. Os artigos referentes aos Capítulos 1 e 2 serão submetidos para publicação no periódico

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CAPÍTULO 1 - *Three-point bending test parameters to obtain elastic modulus of composites.*

ABSTRACT

Three-point bending (TPB) testing is routinely employed in studies characterizing dental materials; however, testing parameters are not well standardized, making inter-study comparisons challenging. This study evaluated different TPB parameters used in ISO standards and dental papers on the elastic modulus (E) calculation and stress distribution of composite beams. Nanofill composite cylinders were produced as control for E measurements using Knoopmicrohardness (KH) (n=10). After, six molds with different beam dimensions were built and filled using nanofill resin-based composite photoactivated for 20 s (n=10). Specimens were submitted to TPB test according to each paper/standard and E was obtained. Data were statistically analyzed ($p<0.05$). Six three-dimensional models were generated according to the laboratory designs and finite element analysis (FEA) was carried-out considering the E calculated by KH. Analyses using von Mises, maximum principal stress and shear stress were performed. Higher E was verified for the composite tested using KH and different E were obtained with the TPB tests. FEA also showed distinct stress distribution at the beams for the different groups. The present study showed that the TPB test

should be revisited when used to E measurements and a minimum span-to-height ratio of 16 should be used for TPB testing of dental materials.

Key words: elastic modulus, finite element analysis, Knoopmicrohardness, resin-based composite, three-point bending.

Laboratorial tests are important to obtain mechanical properties and to estimate the clinical behavior of dental materials(Garoushi *et al.*, 2012).The elastic modulus (*E*) is an important mechanical property that indicates the rigidity matrix of materials. This property is a linear relation between stress and strain that defines the stiffness of a body and is primarily calculated by taking into account the elastic behavior of specimens within a load range(Plotino *et al.*, 2007).

The resin-based composites exhibit varied *E* values among different types and brands(Boaro *et al.*, 2010), which is predominantly correlated to the organic matrix composition and amount of inorganic content(Goncalves *et al.*, 2010a; Goncalves *et al.*, 2010b). Different mechanical tests can be performed for measuring the *E* of resin-based composites, such as nanoindentation(El-Safty *et al.*, 2012b), microhardness(Salerno *et al.*, 2012), ultrasonic (Borba *et al.*, 2011), and three- or four-point bending (Boaro *et al.*, 2010; Melander *et al.*, 2011). However, the three-point bending test (TPB) is the most used, consisting of rectangular/cylindrical specimens positioned above two supports with a loading applied perpendicularly to its long-axis, creating longitudinal tensile and compressive stresses at the beam(ISO178, 2010; ISO4049, 2009).

There is no consensus regarding the parameterization of TPB in the current dental literature (Boaro *et al.*, 2010; Oliveira *et al.*, 2012; Pick *et al.*, 2010; Yap and

Teoh, 2003). Modifications in the dimensions, geometry, span-length, or in span-to-height ratio (*L/h*) may provoke abrupt changes in the stress-strain fields of the testing beam, making comparisons between studies challenging(Alander *et al.*, 2005; Garoushi *et al.*, 2012). Also, these differences can cause problems when misinterpreted *E* data are used as input to feed numerical models. Computational methods, such as finite element analysis (FEA), are being increasingly used in studies assessing dental materials and their interaction with the dental structures(Meira *et al.*, 2010; Raposo *et al.*, 2012). This method allows useful stress-strain analysis of numerous mechanical problems, which is not always possible with other methodologies(Versluis, 2009).Despite some studies had shown the influence of *L*/*h* on the TPB outcomes (Christiansen *et al.*, 1974; Cooper, 1977), there is still lack of computational analysis evaluating the stress distribution at dental materials testing with different parameters.

Therefore, the objective of this study was to evaluate the effect of TPB test variables used in ISO standards and dental papers for obtaining *E* data of resin-based composites and to evaluate the stress distribution on the different test designs by FEA. Then, two null-hypotheses were proposed: 1) the *E* of the resin-based composite would not be influenced by the testing methods or by the different TPB designs; 2) the stress distribution at the beams would not differ among the different TPB designs.

MATERIALS & METHODS

Microhardness Testing

Nanofill composite cylinders (Filtek Supreme XT, A2 shade, 3M-ESPE, St. Paul, MN, USA) were produced as controls for *E* measurements under Knoopmicrohardness testing (KH) (n=10). Specimens were prepared in individual PVC matrixes, 2.0 mm in diameter and 2.0 mm height. The resin was placed in bulk increment and photoactivated using quartz-tungsten halogen (QTH) light for 20 s (Optilux 500, Demetron, Kerr, Orange, CA, USA, 550 mW/cm^2). Ten indentations were made per specimen (200 g/20 s). The major diagonal (D), minor diagonal (d) and microhardness (KHN) were obtained for calculating the *E* of the composite (Marshall *et al.*, 1982; Meredith *et al.*, 1996), using the following formula:

$$E = \frac{0.45 * KHN}{\left[\left(0.140647 - \frac{d}{D}\right)\right] * 100}$$

Three-point bending test

Beams were prepared according to the specifications of two ISO standardsand four dental papers, forming six groups: A (ISO4049, 2009); B (ISO178, 2010); C (Yap and Teoh, 2003); D (Boaro *et al.*, 2010); E (Pick *et al.*, 2010); and F (Oliveira *et al.*, 2012) (Table 1).Six stainless-steel molds were filled with nanofill composite (Filtek Supreme XT, A2, 3M-ESPE) in bulk increment, covered with glass slide and photoactivated by QTH for 20 s (n=10). Afterwards, the beams were hand-polished with #1200 SiC papers. The dimensions were checked with a digital caliper and the beams were stored in dark moisture-free vials at 37° C for 24 h. The specimens were tested according to each experimental

design (Fig. 1). The loading and supporting rods were 2.0 mm in diameter for all groups. The TPB was performed in a mechanical testing machine (DL 2000, EMIC, São José dos Pinhais, PR, Brazil) at 0.5 mm/min crosshead-speed. The relationship for calculating the *E* was the following:

$$E = \frac{F(L^3)}{4w(h^3)D} * 10^{-3}$$

where, *E is the* elastic modulus in GPa, *F* is the maximum loading in N, *L* is the span-length, *w* is the width,*h* is the height, and *D* is the deflection of the beam, all in mm.The data for the KH and TPB tests were checked for homoscedasticity and analyzed using one-way analysis of variance (ANOVA), Tukey's and Dunnet tests ($p<0.05$).

Table 1 –Three-point bending test parameters, *E* (GPa) and standard deviation (±) for the resin-based composite according to the groups.

Test Design	**Beam length (*l*)**	**Beam width (*w*)**	**Beam height (*h*)**	**Span-length (*L*)**	***Span-to-height* ratio (*L/h*)**	***E* (GPa)**
Control	-	-	-	-	-	19.4±0.5
A	25 mm	2 mm	2 mm	20 mm	10	$9.3±0.9^{BC*}$
B	20 mm	2.5 mm	1 mm	16 mm	16	$16.3±1.8^{A*}$
C	12 mm	2 mm	2 mm	10 mm	5	$4.1±0.4^{D*}$
D	12 mm	2 mm	1 mm	10 mm	10	$10.2±0.9^{B*}$

E	10 mm	2 mm	1 mm	8 mm	8	8.2±1.0^{C*}
F	6.5 mm	2 mm	1 mm	5 mm	5	3.7±0.4^{D*}

Different letters indicate significant difference between rows; *Indicates significant difference between control and experimental groups (p<.05).

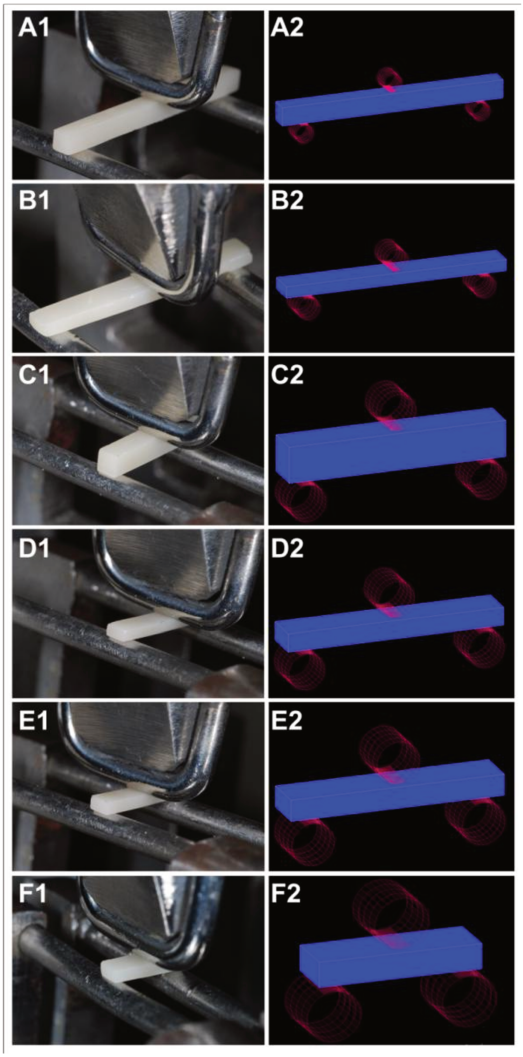

Figure 1 – Laboratory set ups (1) and numerical models (2) according to the TPB designs (A-F).

Finite Element Analysis

Three-dimensional (3D) models were generated and meshed using eight-node hexahedral elements (MSC Mentat 2010, MSC Software Corporation, Santa Ana, CA, USA) (Fig. 1b). All structures and materials were considered homogeneous, linear-elastic and isotropic. The mechanical properties for the composite were calculated and defined considering the *E* found in the KH(19.4 GPa) and Poisson's ratio (0.3). The loading/supporting rods were generated according to the laboratory tests. The contacts were simulated assuming a 0.3 coefficient of friction(Novais *et al.*, 2011), and the same theoretical loading was applied perpendicularly to the beams. Static structural analysis was performed considering non-linear contacts and constraints at X and Z axes.The results were analyzed using von Mises, maximum principal and shear stresses (Figs. 2 and 3).

RESULTS

The *E* values obtained with the microhardness testing were significantly higher(19.4±0.5) compared tothose verified for the TPB groups. Among the TPB results, the B groupshowed the highest *E* values (16.3±1.8), followed sequentially by D (10.2±0.9), A (9.3±0.9) and E (8.2±1.0) groups. The lowest *E* values were found for the C (4.1±0.4) andF groups (3.7±0.4) (Table 1).

The TPB designs with *L/h* equal or superior to 10, presented higher *E* values. Lower *E* values resulted from low *L/h*, mainly for values below 8. As described, the B group (*L*/*h*=16) showed the closest *E* value to those obtained with the microhardness testing, followed by D (*L*/*h*=10), A (*L*/*h*=10) and E (*L*/*h*=8), C (*L*/*h*=5) and F (*L*/*h*=5) groups, respectively (Table 1). A proportional relationship

was observed for the specimens presenting the same *L/h*, since they were categorized in the same statistical groups for the *E*. This finding was also valid for B and E groups, which despite presenting the half *L/h* (16 and 8, respectively), showed the half relationship for the *E*.

The FEA results showed distinct stress distribution patterns for the different beams. According to the von Mises analysis, the groups with low *L/h*showed greater loading stresses (Figs. 2 a-f). Also, the beams with low *L/h* exhibited increased tensile stress concentration when using maximum principal stress analysis (Figs. 2 g-l). For the shear analysis, stresses were well distributed along the beams with high *L/h* (Figs. 2 m-o), while great stress concentration was observed at the loading regions for low *L/h* beams (Figs. 2 p-r). The different stress distribution among the TPB groups is detailed on Figure 3. The C and F groups exhibited higher peaks of shear stress located at the supports, with A and B groups showing a better distribution of the shear stresses along the beam, with lower stresses at the supports (Fig. 3c).

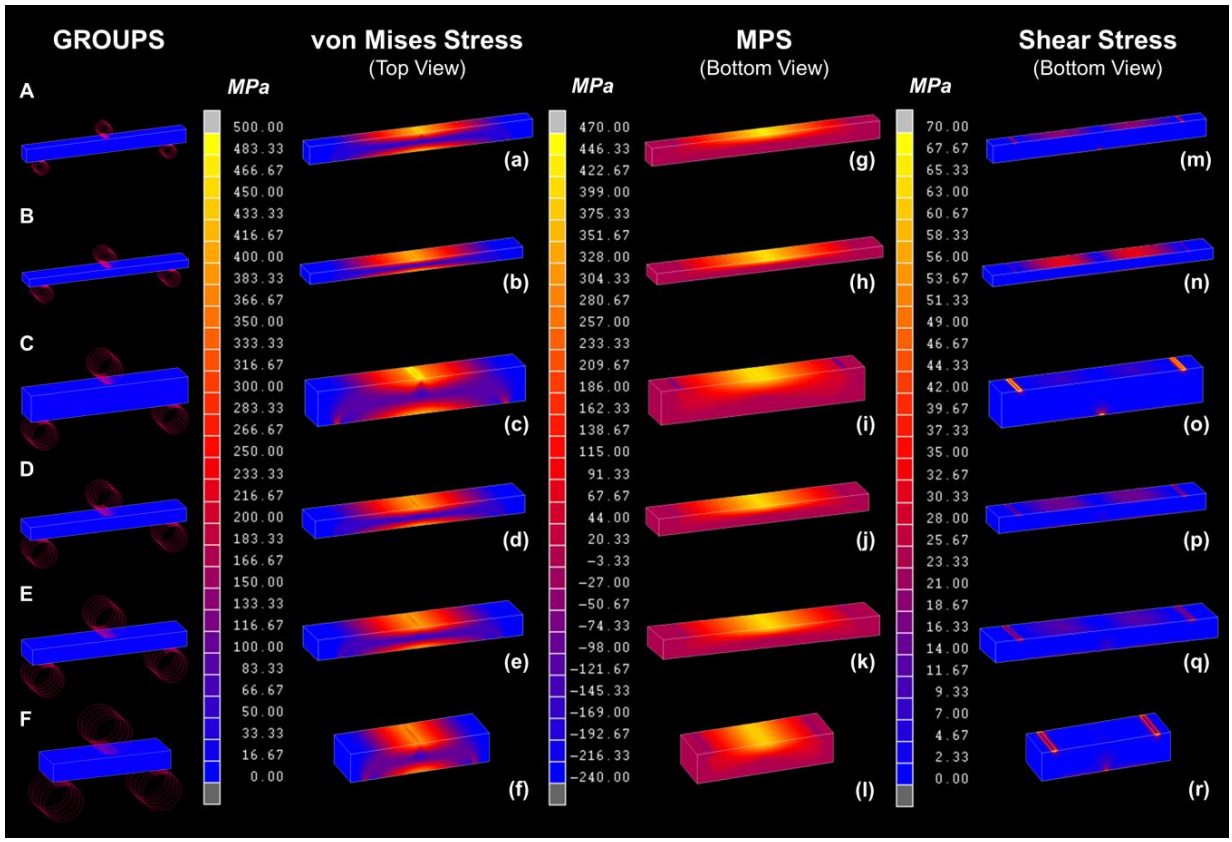

Figure 2 – Finite element results according to the TPB designs (A-F): von Mises stress in top view (a-f); MPS - Maximum principal stress in bottom view (g-l); Shear stress in bottom view (m-r).

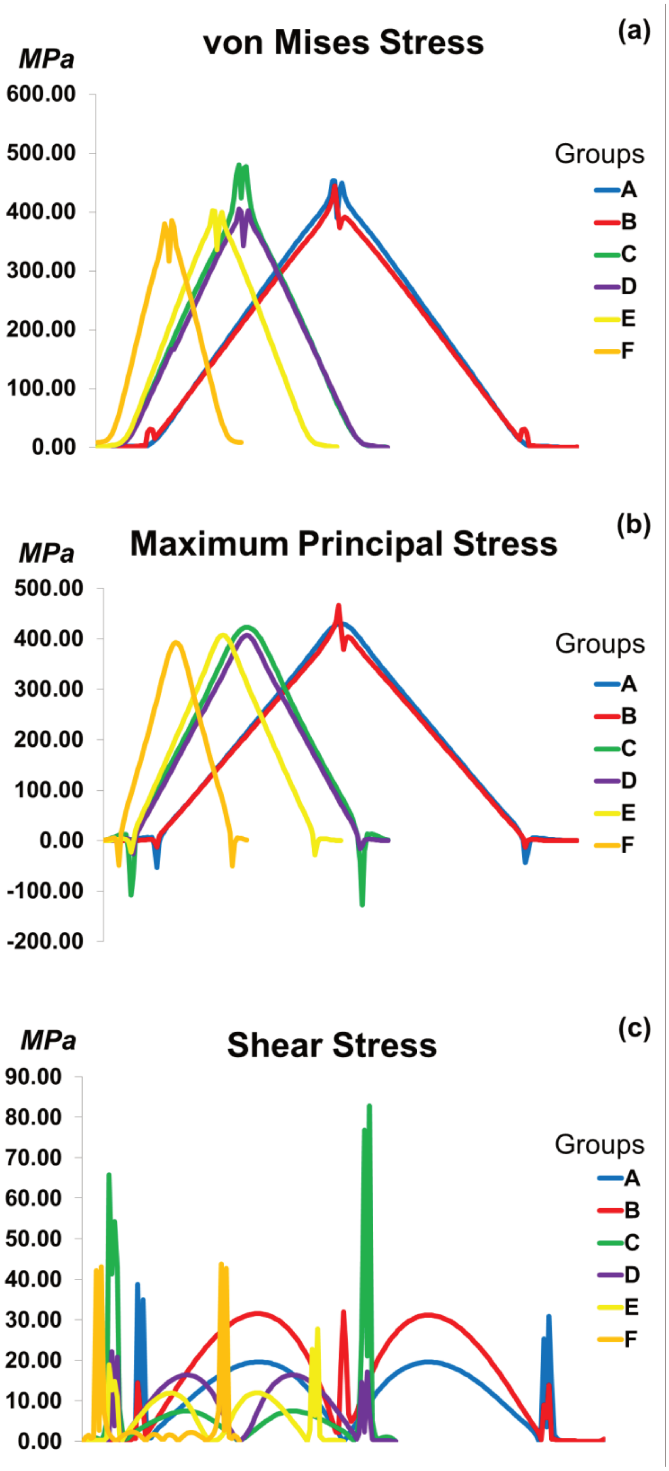

Figure 3 – Graphs showing finite element results according to the TPB designs (A-F): (a) von Mises stress; (b) Maximum principal stress; (c) Shear stress.

DISCUSSION

The *E* of the composite was influenced by the testing methods, since values obtained by the microhardness differed significantly from those found for the TPB groups. Discrepant *E* values were also verified among the different TPB designs. Therefore, the first null-hypothesis was rejected. The stress distribution at the testing beams was influenced by the different TPB parameters and the second null-hypothesis was also rejected.

The results showed that the static method used to measure the *E*(KH) presented similar values to those found in literature(El-Safty *et al.*, 2012a). Static tests, such as the Knoopmicrohardness or nanoindentation approaches, seem adequate methodologies for obtaining reliable *E* values (Table 1), since testing parameters are well standardized with reduced variables affecting results(El-Safty *et al.*, 2012a; Soares *et al.*, 2012). Among the quasi-static methods used for *E* measuring, the TPB test figures as the most employed methodology used in dental papers due to its apparently simple execution. TPB has been commonly used for measuring the *E* of polymeric and non-polymeric dental materials. However, the variables existing among test parameters may be responsible for the large discrepancies observed in this study(Boaro *et al.*, 2010; Oliveira *et al.*, 2012; Pick *et al.*, 2010; Yap and Teoh, 2003).However, as seen, the TPB testing parameters used for evaluating dental materials in different papers poorly follow the standards defined at technical norms. Modifications are commonly carried out in order to adjust TPB design according to the convenience of the material or research requirements, what may lead to great variability in the results, as showed by the

different *E* values verified in this investigation. The mechanical concepts for the TPB test designate that the correct proportions for the specimens must be assured when reductions in the original size are required(ISO178, 2010). Otherwise, several interferences can negatively affect the results (Figs. 2 and 3).

The ISO 178 standard recommends that when is not possible to use the preferred specimen dimension (l=80±2, w=10±0.2, h=4±0.2 mm), the test specimen shall be in the same ratio as for the preferred specimen, or with a length 20 times higher than its thickness, what is, *l/h*=20±1 mm(ISO178, 2010). The span-length for the supporting rods also shall comply with the following reason, *L*=(16±1)*h. Except for the B group, which followed the required proportioning, these dimensions/proportions were not verified for the other experimental groups. Even the testing design of the A group, which was carried out in accordance to the designations of a technical standard (ISO4049, 2009), does not match the preferred specimen ratio (*l*=25±2, *w*=2±0.1, *h*=2±0.1 mm) or desirable span-length reason (*L*=20 mm)(ISO178, 2010). In fact, none of the TPB specifications recommended for the groups A, C, D, E and F suggest the proportioning of the specimens according to the preferred specimen dimensions(ISO178, 2010).As seen, the span-length, likewise plays an important role in the TPB testing(Garoushi *et al.*, 2012), because during the test a complex stress field is generated at the beam, with compression, tensile and shearing stresses acting. When the supporting rods are positioned closer, with a reduced span-length, more shearing stresses are observed within the testing beam at the region between the

loading/supporting rods (Figs. 2 and 3). On this way, variations in the span-length may result in different *E* for the same material.

The effect of different span-to-length ratios on the stress developed at the beams was evaluated. High shear stresses were concentrated at the contact regions of the loading/supporting rods, mainly when the *L/h* was under 8 (Figs. 2 p-r). This fact can impose a high load force with great concentration of shear stress principally at the neutral zone, and this can propitiate unstable failures decreasing the *E* (Table 1). The stress distribution verified for the different TPB designs indicated that the testing parametersaffected the three-point bending test (Fig. 2). This fact is probably related to the *L/h* of each test design, which could explain why so different *E* were found for the same composite (Table 1), what is in accordance to previous investigations(Alander *et al.*, 2005; Eckrote *et al.*, 2003; Garoushi *et al.*, 2012). These studies showed that by increasing the *L/h*, the maximum load values decrease and flexural modulus increase, which is in agreement with the present results. The groups B, A and D, which respectively have higher*L/h*, resulted in high *E* (Table 1), showing proportionality between these variables. The von Mises FEA results, which consider the sum of all stresses, showed increased loading stress in specimens with low *L/h* (Figs. 2 d-f). This was probably caused by the difficulty of the beam in deflect during the test, what increases the load required for testing, causing premature failure of the specimen.

Moreover, an increased concentration of shear stress occurs inside the specimen instead of tensile stress(Cooper, 1977; Eckrote *et al.*, 2003). The high shear stress concentration verified at the loading/supporting regions for the low

*L/h*beams (Figs. 2 p-r), may be responsible for premature failures of specimens under these stresses (Alander *et al.*, 2005). The bending of beams with low *L/h* accumulates up to five times more energy than when specimens with high s*L/h* are loaded in TPB (Cooper, 1977). In small beams, energy is primarily dissipated to the loading/supporting devices and the specimen cannot be ideally bent. Also, the shear stresses occurring at the neutral zone of the beam lead to more unstable fractures.

Unlikely, the beams with high *L/h* (Figs. 2 m-o) are more prone to undergo bending, and shear stresses are better distributed along the entire beam. In these cases, the association of the total stress derived from shear, tensile and compressive stresses may cause failures primarily by bending. The fracture load of beams with high *L/h* is smaller, since the more flexible beam can store sufficient energy remaining in the elastic regimen. To assure failures by tensile/compressive stresses, beams must be adequately bent, what increases the stability of the test, according to the FEA results (Figs. 2 g-i) and laboratorial data (Table 1). This was clearly observed in the beams with *L/h*, which showed tensile stress more distributed along the entire body and consequent increased *E* values, since the TPB test was better performed. Additionally, specimens with high *L/h* present a beam aspect, differing from those with low *L/h*, which show a bar aspect.

As seen, the *E* data must be extremely precise, obtained in well standardized tests for being used as input data for FEA or other. Thus, obtaining adequate mechanical properties that accurately characterize the behavior of materials is essential. This fact evidences the importance in providing suitable

properties, which will be decisive to the accuracy of the results achieved with the finite element method. On this way, the interaction between laboratory and computational methodologies seem very important for understanding the biomechanical behavior of dental structures and restorative materials.

The TPB test plays a very important role in flexural strength and fracture toughness tests(Christiansen *et al*., 1974; Cooper, 1977). However, the present study showed that this test should be revisited and improved when used to *E* measurements. According to our results, a minimum *L/h* of 16 should be used in specimens for TPB testing of dental materials, which is in agreement to the preferred specimen dimension proposed by the ISO 178 standard. Other methodologies, such as strain-gauge method or laser interferometry, can also be associated to the TPB test in order to make *E* measurements at the elastic regimen, without reaching plastic deformation or even specimen failure.

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CAPÍTULO 2 - *Assessing mechanical properties of dental materials using three-point bending test associated to strain-gauge method.*

ABSTRACT

Three-point bending testing (TPB) is widely used to obtain the mechanical properties of dental materials, i.e., elastic modulus (*E*); however its association with strain-gauge method can make measurements more accurate. Aluminum (AL) and nanofill composite (NC) specimens were used to compare the *E* values obtained with Knoopmicrohardness test (KH), TPB, and three-point bending test associated to strain-gauge method (TPBS). Cylindrical and beam specimens of each material were made for the KH and TPB tests, respectively, and the conventional tests were carried-out. For the TPBS, beam specimens were built, and a strain-gauge was attached at the center of the bottom surface. A compressive load was applied to the specimen and the strain data was recorded for *E* calculation. Afterwards, the *E* of other composite-based materials: NFC- nanofillflowable composite resin;CRC-conventional dual-cure resin cement; and ADH- adhesive system (ADH); were obtained using TPBS. The mean *E* (GPa) for KH were: AL (69.01±8.67) and NC (19.42±0.18); for TPB: AL (22.85±4.62) and NC (9.26±0.59); and for TPBS: AL (68.35±3.11) and NC (20.03±1.42); and NFC (17.03±1.14), CRC (18.37±0.93) and ADH (5.45±0.76). The TPB test presented the lower*E*values for AL and NC.The TPBS test showed similar *E*values to those found with KH test for the both materials. The proposed TPBS method offers a reliable approach for obtaining the

E of dental materials and TPB seems not the most adequate method for *E* calculations.

Key Words: elastic modulus, strain gauge method, Knoopmicrohardness, resin-based composite, three-point bending.

INTRODUCTION

The knowledge on the properties of dental materials is very important to characterize their behavior under different test conditions and at the oral environment. Laboratorial methods used to assess the mechanical properties of these materials have been established in several dental studies (El-Safty*et al.*, 2012; Garoushi*et al.*, 2012). Elastic modulus (*E*) has great importance amongst mechanical properties once it represents the inherent stiffness of a material within the elastic range representing a linear relationship between stress and strain. It can be determined from the slope of the stress/strain curve, by considering the elastic behavior of specimens within a load range (Plotino*et al.*, 2007). The *E* can be acquired by using uniaxial tensile tests (Chabrier*et al.*, 1999); however, the classic uniaxial tensile test is often problematic for dental materials and tissues due to the specimen dimensions dictated by material/tissue size, costs, and/or manufacturing limitations. Resin-based materials such as composite resins, adhesive systems and resin cements exhibit varied *E* values among different types and brands (Boaro*et al.*, 2010), which is primarily correlated to the organic matrix composition and amount/type of inorganic content (Goncalves*et al.*, 2010a; Goncalves*et al.*,

2010b). On this way, efficient methods to assess *E*of these materials becomes very important.

Different mechanical tests can be performed for measuring the *E* of resin-based materials, such as nanoindentation (El-Safty*et al.*, 2012), microhardness (Salerno *et al.*, 2012), ultrasonic (Borba*et al.*, 2011), and three- or four-point bending tests (Boaro*et al.*, 2010; Melander*et al.*, 2011). Measuring the dimensions of the short and long diagonals obtained with the Knoop indentation test (KH), the elastic modulus (GPa) can be determined by an empirical relationship, yielding a simple and low cost method in this sense (Marshall *et al.*, 1982; Meredith *et al.*, 1996).

The three-point bending test (TPB) is one of the most used approaches on dental materials testing and also for *E* calculations due to its apparently simple execution. It consists of rectangular/cylindrical specimens positioned above two supports with axial loading applied perpendicularly to their center, creating longitudinal tensile and compressive stresses at the specimen (ISO4049, 2009). There are many differences in TPB testing parameters which may provoke abrupt changes in the stress-strain fields of the testing beam, making comparisons between studies challenging (Alander*et al.*, 2005; Garoushi*et al.*, 2012). The TPB is mainly indicated to flexural strength and fracture toughness assessments and its application to *E* measurements would be inadequate (Cooper, 1977).

Destructive mechanical tests, such as TPB, are important for *in vitro* biomechanical analysis of restorative materials, showing specimens behavior in punctual high load situations. The evaluation of specimen's internal behavior is,

however, limited with these tests (Soares*et al.*, 2006). Apparently the application of non-destructive tests, as strain-gauge method (Sakaguchi*et al.*, 1991) seems more suitable for checking the interference of internal factors on the restorative process. The strain gauge method is able to assess strains at the surface of the material by converting electrical signs in metrical units as microstrain (μs). This test is capable to detect the lower interatomic variations and to show when the specimen moves from elastic to plastic regimen, allowing to ignore the last phase and the failure limit. The association of strain gauge method and TPB would be important to evaluate the elastic limit in a more accurate form, since the measurement of specimen's deflection by mechanical testing machines is not always reliable.

Therefore, the aim of this study was to evaluate if the three-point bending test associated to strain gauge method is a viable approach for obtaining *E* values by comparing the results with classical TPB and KH tests. The hypothesis of this study was that the type of test would not influence the *E* values.Then, the new methodology will be used to assess the *E* of other resin-based materials, such as a nanofill composite flow resin, a conventional resin cement, and an adhesive system.

MATERIALS & METHODS

Microhardness Testing

Aluminum cylinders with gloss-polished surfaces(6351-T6, DPA Alumínio, São Paulo, SP, Brazil) were used as control for Knoopmicrohardnessmeasurements (KH) (n=10)since the mechanical properties of

this material are well established in literature (ASM, 1990). Nanofill composite cylindrical specimens (Filtek Supreme XT, A2 shade, 3M-ESPE, St. Paul, MN, USA) were prepared using individual PVC matrixes, 2.0 mm in diameter and 2.0 mm height (Fig. 1A). Resin was placed in bulk increment and photoactivated by using quartz-tungsten halogen (QTH) light-source for 20 s (Optilux 500, Demetron, Kerr, Orange, CA, USA, 550 mW/cm^2). Ten Knoop indentations were made on each specimen for booth groups (200 g/20 s). The major diagonal (*D*), minor diagonal (*d*) and KH were obtained and applied for calculating the *E* of the materials(Meredith *et al.*, 1996), employing the following formula:

$$E = \frac{0.45 * KHN}{\left[\left(0.140647 - \frac{d}{D}\right)\right] * 100}$$

Three-point bending test

Beams were prepared from AL and NC (n=10) according to ISO 4049 standard (ISO4049, 2009), 25.0 mm in length, and 2.0 mm in width and height. The AL beams were made by cutting an aluminum slab 2.0 mm in thickness (Fig. 1B) into the specimens. A stainless-steel mold was filled with nanofill composite (Filtek Supreme XT, A2 shade, 3M-ESPE) in bulk increment, covered by glass slide and photoactivated by QTH for 20 s (n=10). Afterwards, the beams were hand-polished with n. 1200 SiC papers. The dimensions were checkedby using digital caliper and the beams were stored in dark moisture-free vials at 37° C for 24 h previous to testing. The specimens were tested in TPB set up, with 20.0 mm span length and2.0 mm diameter loading/supporting rods. The TPB was performed in a

mechanical testing machine (DL 2000, EMIC, São José dos Pinhais, PR, Brazil) at 0.5 mm/min crosshead-speed. The relationship for calculating the *E* was the following:

$$E = \frac{F(L^3)}{4w(h^3)D} * 10^{-3}$$

where, *E is the* elastic modulus in GPa, *F* is the maximum loading in N, *L* is the span-length, *w* is the width, *h* is the height, and *D* is the deflection of the beam, all in mm.

Three-point bending test associated to strain-gauge method

The AL and NC TPBS specimens were built following the same steps described for the TPB (n=10). Additional specimens of nanofillflowable-resin composite (NFC) (Filtek Supreme XT Flow, A2, 3M-ESPE), conventional dual-cure resin cement (CRC) (RelyX ARC, A2, 3M-ESPE), and adhesive-system (ADH) (Scotch Bond Multi-Purpouse, Bond, 3M-ESPE) were built. All resin-based materials were used following manufacturer´s specifications and were also stored in dark moisture-free vials at 37° C for 24 h previous to testing. Then, a strain gauge (PA-06-038AA-120-LEN, Excel Sensores, São Paulo, Brazil) was attached at the center of the bottom surface parallel to the beam long axis using cyanoacrylate-based adhesive (Super Bonder; Loctite, Itapevi, SP, Brazil). The strain gauge grid had an area of 1 mm^2, an electrical resistance of 120 Ω and a gauge factor of 2.12 (Soares*et al*, 2008).The sensors were connected to a data

acquisition system (ADS0500IP; Lynx, Sao Paulo, SP, Brazil) using quarter Wheatstone bridge.

The 50KgF load cell was hardwired to the mechanical testing machine and to the strain-gauge system. Then, a compressive perpendicular load was applied at 0.5 mm/min crosshead speed and the strain pattern (μS) of each specimen were recorded (Fig. 1C). The data were recorded at 0.4 hertz and transferred to a computer employing specific acquisition signal transformation and data analysis software (AqDados 7.02 and AqAnalisys; Lynx, São Paulo, SP, Brazil). When the stress/strain curve suffered variation showing the transition from elastic to plastic regimen (Fig. 2), the values obtained at peak were marked and were applied in the formula:

$$E = \frac{1}{48} * \frac{FL^3}{fJ}$$

where, *E is the* elastic modulus in GPa, *F* is the maximum loading in N, *L* is the span-length in mm, *f* is the maximum flexa and *J* is the moment of inertia.

The data for the microhardness, TPB and TPBS tests were checked for homoscedasticity and analyzed using one-way analysis of variance (ANOVA) and Tukey's test ($p<0.05$).

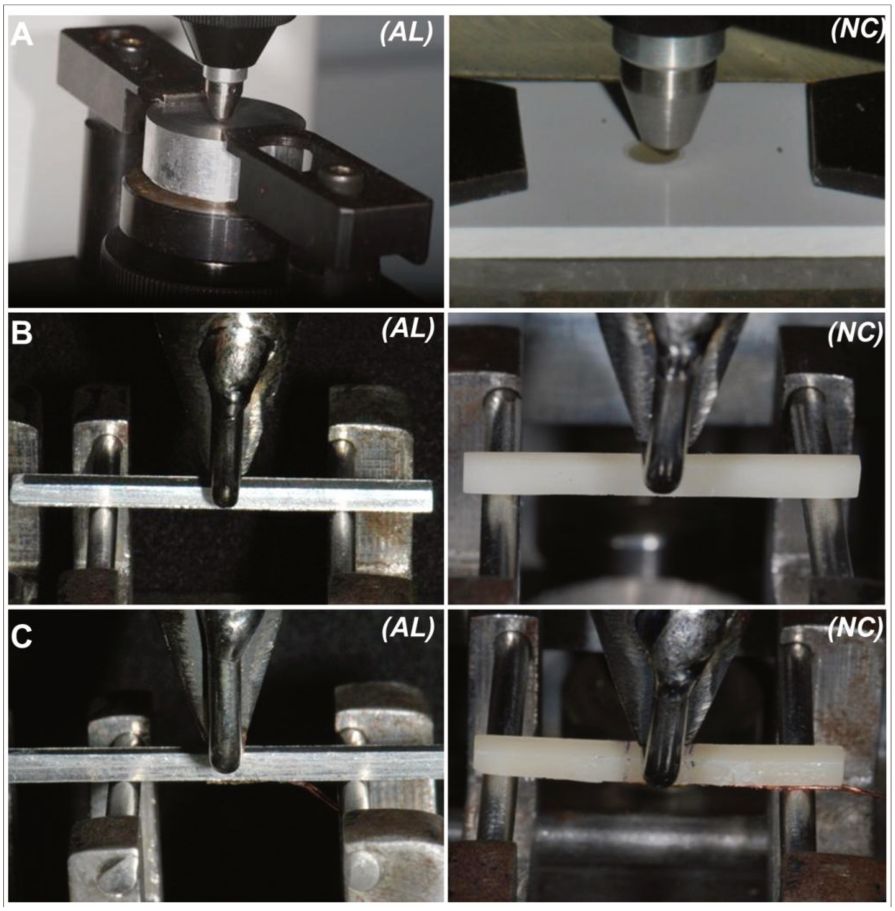

Figure 1 – Different tests used for calculating the *E* of aluminum (AL) and composite resin (NC): A- Knoopmicrohardness (KH); B- Conventional three-point bending test (TPB); C- Three-point bending test associated to strain gauge method (TPBS).

RESULTS

The *E* values obtained with the microhardness testing (KH) were: AL (69.01±8.67) and NC (19.42±0.18) (Table 1). For the conventional three point-bending test (TPB) the values were: AL (22.85±4.62) and NC (9.26±0.59) (Table 1). For three point-bending strain-gauge method (TPBS) the values were:

AL(68.35±3.11) and NC (20.03±1.42). Comparisons among the different tests showed no statistical differences between the *E* values for KH and TPBS groups.The TPB test presented lower*E* values compared to KH and TPBS tests, for both the AL and NC groups. The *E* values obtained for the other resin-based materials were: NFC (17.03±1.14), CRC (18.37±0.93) and ADH (5.45±0.76) (Table 1).

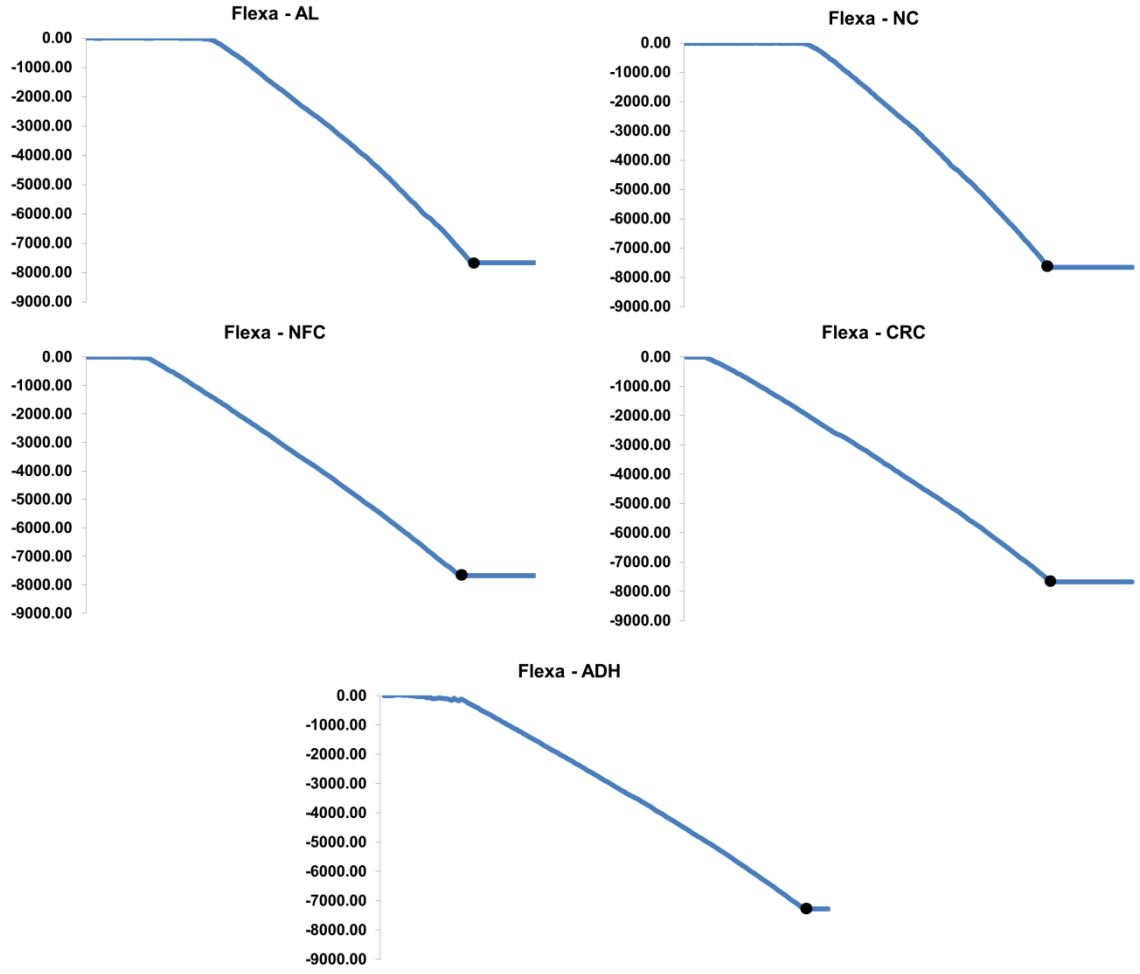

Figure 2 – Graph plotting showing the elastic deformation of the specimens at the TPBS*E* measurements.

Table 1 -Elastic modulus (GPa) and standard deviation for the experimental groups.

Mechanical Test	**Testing materials**				
	AL	*NC*	*NFC*	*CRC*	*ADH*
KH	69.01±8.67^{A}	19.42±0.18^{a}	-	-	-
TPB	22.85±4.62^{B}	9.26±0.59^{b}	-	-	-
TPBS	68.35±3.11^{A}	20.03±1.42^{a}	17.03±1.14	18.37±0.93	5.45±0.76

Different symbols indicate significant difference between rows (p<.05).

DISCUSSION

The *E* of the materials evaluated was influenced by the testing methods, since the values obtained by the KH test were statistically similar to those of TPBS, which differed significantly from the results obtained with the conventional three-point bending method. Therefore, the hypothesis of this study must be rejected, since the *E* values verified for the AL and NC groups were affected by the testing methods.

The results showed that static methods, such as KH are efficient to assess the elastic modulus of materials (Marshall *et al.*, 1982).This is in accordance to the

results of this study, which presented similar values to those found in literature (Table 1), both for aluminum (AL) (ASM, 1990), and for the nanofill composite resin (NC) (El-Safty*et al.*, 2012). Static tests, such as the Knoopmicrohardness or the nanoindentation approaches, seem adequate methodologies for obtaining reliable *E* values compared to TPB (Table 1). This probably because the testing parameters of some static methods are better standardized with reduced variables affecting the results (El-Safty*et al.*, 2012; Soares*et al.*, 2012). The *E* calculation was performed based on a method already applied in dentistry (Meredith *et al.*, 1996), which relies on the ability of material's elastic recovery in the walls of the indentation after removing the applied load. While applying the load, the ratio between the major diagonal (D) and minor diagonal (d) is constant (D/d = 7.11). However, when the load is removed, the elastic recovery occurs without affecting the minor diagonal. The length of this elastic recovery depends on the relation between the microhardness and the elastic modulus of the material.

The TPB test plays a very important role in flexural strength and fracture toughness tests (Christiansen *et al.*, 1974; Cooper, 1977). However, when TPB test is used for *E* measurements, the beam specimens are commonly loaded until their failure. The resulting load and displacement values recorded are then submitted to a mathematical equation in order to obtain the flexural modulus, which is theoretically equivalent to *E*. But, at this point, the beam already reached its plastic regimen, what consequently distorts the *E* results, since it should be calculated at the elastic regimen of the material. On this way, this test should be revisited and improved when used to *E* measurements. An association with non-

destructive tests can be useful in order to make *E* measurements at the elastic regimen, without reaching plastic deformation or even specimen failure.

The new approach proposed for associating the three point-bending test to strain-gauge method (TPBS), exhibited similar *E* values to those obtained with the microhardness test measurements (KH) (Table 1). This new modality of test adds the accuracy of strain gauge measurements to obtain the elastic limit at the stress/strain curve for specimens under testing at the flexural strength set up. Most of the software used in mechanical test machines has poorly capacity to determine the exact point of transition from elastic to plastic regimens. Additionally, some equipment may not make accurate measurements of the displacement experienced by specimens. The TPBS test allows ease determination of the elastic limit when the displacement of the specimen recorded by the strain gauge method is analyzed (Fig. 2). Other advantage of TPBS, know related to KH, is that the former is less subjective than the last, not depending upon the visual accuracy of the researcher or on the more suitable measuring region as in fully automatic microhardness testers.

Using aluminum as control for validating the TPBS test is justified by the fact that this material has its mechanical properties well characterized in the current literature (ASM, 1990). After checking its applicability for aluminum, the TPBS test was applied for measuring the *E* of a nanofill composite and also showed its efficacy. Thereby, the test was also applied for calculating the *E* of other resin-based materials showing its feasibility (Table 1). Despite some engineering studies had already used strain gauge method for measuring *E* of several materials, it was

associated to tensile tests and not to three-point bending test as in the present investigation. Tensile tests present less variations in parameters compared to three-point designs; however, it present some limitations for dental specimens due to their dimensions as mentioned before.

The present study showed that the three-point bending test is not the most adequate method for measuring *E* when used isolated. But the association of the three-point bending test with the strain gauge method can overcome the previous limitations providing more reliable results on dental materials testing. Thus, this new testing approach seems very promising to be applied for *E* measurements on several materials.

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CAPÍTULO 3 - *Effect of specimen positioning and loading on microshear bonding outcomes: a non-linear finite element analysis.*

ABSTRACT

Objectives: This study compared the effect of different testing parameters on the stress distribution of microshear specimens. *Methods*: Three-dimensional models consisting of disilicate-based ceramic plates with two resin cement cylinders were generated. The distances between the two cylinders were varied (1.0, 1.5, 2.0, 2.5, 3.0 mm). A 10 N load was applied on one cylinder using a 0.2mm diameter orthodontic-looped wire in three different directions: Y (perpendicular to adjacent cylinder), X+ (towards adjacent cylinder) and X- (away from adjacent cylinder). Additionalthree-dimensional microshearmodelswith one resin cement cylinder were loaded (10 N) by: large stainless-steel tape (LT),small stainless-steel tape (ST), chisel (CH), orthodontic-looped wire (OW),or customized chisel (CC). *Results*: Stress concentration arising from the loaded cylinder reached the adhesive region of the adjacent non-loaded cylinder for 1.0, 1.5 and 2.0 mm models for all loading directions. When 3.0 mm apart, no stress elevation was found at the non-loaded cylinder.For the CH and OW loading, tensile stresses were more dominant at the interface. The model loaded with the CC scheme presented lower tensile and shear stresses. A predominance of shear stresses was verified for the ST and LT loading systems.*Conclusion*: A 3.0 mm separating space between cylinders seems a safe distance to avoid unwanted stress to reach the adhesive interface of the

non-loaded cylinders. Loading the cylinders perpendicularly (Y direction)with straight-aligned cylinders appears the most suitable condition. Loading specimens with small (ST) and large (LT) stainless-steel tapes seem the better alternative.

Key-words: dental ceramics, finite element method, microshear, resin cement, testing parameters.

INTRODUCTION

The adhesive dentistry has been progressively improved with the development of new restorative materials and techniques, resulting in less complex clinical procedures with increased longevity. Several mechanical tests are routinely applied to characterize the behavior of dental materials or to evaluate their mechanical properties. Assays verifying bonding quality and strength of adhesive systems/resin cements to dental substrates/restorative materials are the tests most commonly employed for dental materials [1].

Conventional tensile and shearing tests have been applied for several years in dental materials evaluation, although it has been shown that the results obtained with these methodologies do not fully represent the bonding strength of the testing materials with great accuracy [2,3]. This occurs because the specimens used in these tests are more prone to have failures incorporated at the adhesive interface or at the bonding substrates due to their increased dimensions [4]. Moreover,

owing to the loading mode and the geometry of the specimens used on these methodologies, uneven stress can be induced at the bonding interfaces, leading to large variations in the results [1,5-7].

In an attempt to overcome the limitations of the previous bonding tests, Sano *et al*., proposed to evaluate interfacial bond strength from smaller bond test areas (1.0 mm^2) using the microtensile approach[8]. With this methodology, it became possible to obtain several specimens from a single tooth, measuring bond strength in different regions while reducing scatter and achieving adhesive failures in the majority of specimens. The advantages of this approach for testing bonding interfaces have been highlighted by numerous studies and today it is the most used mechanical test for this purpose [1,9-11]. However, the dissemination of this methodology allowed adaptations to be suggested through the original approach [9], resulting in conflicting results.

As occurred with the tensile/microtensile methods, the tendency toward evaluating bonding interfaces in reduced adhesive regions in order to incorporate less failures and variables during the test was also conceived for the shearing approach. The shearing test using specimens with adhesive interfaces inferior to 1.0 mm^2 was initially proposed, being nominated “microshearing” [12]. Another microshear modality was also suggested for checking the bond strengths of dental materials, using very small cylinders of resin-based cements (ø 0.75 x 0.5 mm) bonded to different substrates (dentin, enamel, ceramics, etc.) [13-15]. With this methodology, it became possible to prepare multiple bonding specimens in a single

ceramic surface or even at a single tooth region, without requiring additional procedures that may cause test variables, such as the trimming of the adhesive region necessary in many microtensile specimens [16,17].

Since microshear specimens have reduced dimensions, this test design allows reduced failure incorporation, providing more accurate results for bonding strength evaluation. Some comparative investigation have shown that the microshear test can provide bond strength results as reliable [18], or even more accurate [19,20], than the obtained using the microtensileapproach. The viability of this methodology is also demonstrated by its crescent utilization in the recent literature [21-25]. However, independently of the mechanical test used for evaluating bond strengths, large variation exists in the results [26,27]. This fact can be better comprehended by finite element analysis, in which uneven stress distribution are commonly observed at the regions of interest for different tests due to variables involving the mechanical properties, geometry, loading mode, and the preparation of the specimens [1,11,26,28,29].

The popularization of the microshear methodology also allowed modifications to be introduced [15,30], what can lead to conflicting results between studies. However, the description of materials and methods in several studies generally fails to report important test parameters. A wide range of configurations were used to load specimens in the shearing tests, such as orthodontic-looped wires, sharp wedges, stainless-steel tapes and chisels [31-34]. However, loading was shown to be more effective when performed with orthodontic-looped wires [5]

or stainless-steel tapes [35] that with chisels, because the last induces uneven stresses at the periphery of the adhesive layer. Likewise, the loading applicators may affect the microshear test results. Other parameters, like the distance between the multiple microshear cylinders on a specimen and the loading direction can also influence the bonding results as well.

Therefore, the aim of this study was to evaluate the effect of different loading applicators, loading mode and specimen positioning on the stress distribution at the adhesive interface of microshear specimens with resin cement cylinders bonded to a disilicate-based ceramic using finite element analysis. The hypothesis that these parameters could influence the results of finite element simulations was investigated.

Materials and Methods

Finite Element Analysis

Strain and stresses under a given loading can be calculated by finite element (FE) analysis on the basis of the specimen geometry, boundary conditions, and material properties. Numeric methods such as FE showed good correlations with experimental methods [36]. Therefore, three-dimensional (3D) finite element models of the various microshear testing conditions considering the specimens (ceramic plates and resin cement cylinders) were generated and

meshed using eight-node hexahedral elements (MSC Mentat 2010, MSC Software Corporation, Santa Ana, CA, USA). A 1.0 mm thickness ceramic plate was defined with two 0.7 mm diameter and 0.5 mm height resin cement cylinders. The distances between the two cylinders were varied in the models (1.0, 1.5, 2.0, 2.5, 3.0 mm). A 10 N perpendicular load was applied on one cylinder using a 0.2-mm diameter orthodontic-looped wire, placed close to the adhesive interface, in three different directions: Y (perpendicular to adjacent cylinder), X+ (towards adjacent cylinder) and X- (away from adjacent cylinder).

Additional 3D models of the ceramic plate with one cylinder and one of the follow loading systems were also generated: large stainless-steel tape (LT), small stainless-steel tape (ST), chisel (CH), orthodontic-looped wire (OW), or a customized chisel conforming the cylinder (CC). All loading systems were considered contacting surfaces and a perpendicular 10 N load was applied. The materials were assumed as homogeneous, linear-elastic and isotropic and the mechanical properties of the materials used are described on Table 1 [37-40]. Friction between the loading systems and the cylinder was considered negligible. Constraints were applied at the back of the ceramic plate, simulating specimen attachment in the laboratory set-up. Static structural analysisconsidering non-linear contacts was performed (MSC Marc 2010, MSC Software Corporation)and the results were analyzed using von Mises (VM), Maximum Principal (MPS), and shear (G) stresses.

Table 1 –Mechanical properties used to develop finite element models.

Material	Elastic modulus (GPa)	Reference	Poisson's Ratio	Reference
Disilicate-based ceramic	96.0	[37]	0.23	[40]
Dual-cure resin cement	8.0	[38]	0.3	[38, 39]

RESULTS

Stress concentration arising from the loaded cylinder reached the adhesive interface of the adjacent non-loaded cylinder for models that had it placed 1.0, 1.5 and 2.0 mm apart, for all loading directions (Y, X-, X+) (Figs. 1-3). Higher stress concentration was found mainly for the 1.0 and 1.5 mm models. For the 2.5 mm apart cylinders, only a slight stress increase was found at the non-loaded cylinder during the X+ and X- loadings (Figs. 1-3). When placed 3.0 mm apart from the loaded cylinder, no stress elevation was found at the non-loaded cylinder for any loading direction (Y, X-, X+) (Figs. 1-3). Pulling loaded specimen away from the adjacent cylinder (X-) resulted in high tensile stresses at the adhesive interface of the non-loaded cylinder for all distances. Loading in Y or X+ direction caused less critical stresses (Figs. 1-3).

For the CH and OW loading systems, tensile stresses were more dominant at the interface than shear stresses (Figs. 4 and 5). The highest loading stress was

verified for the OW system (Fig. 5a), with high shear and tensile stresses associated at the interface (Figs. 5b and c). The model loaded with the CC system had lower tensile and shear stresses at the adhesive interface compared to the CH and OW models, with a more uniform stress distribution (Figs. 4 and 5). For the models loaded with the ST and LT loading systems, a similar behavior was found, with a predominance of shear stresses and almost none tensile stresses at the adhesive interface (Figs. 4 and 5).

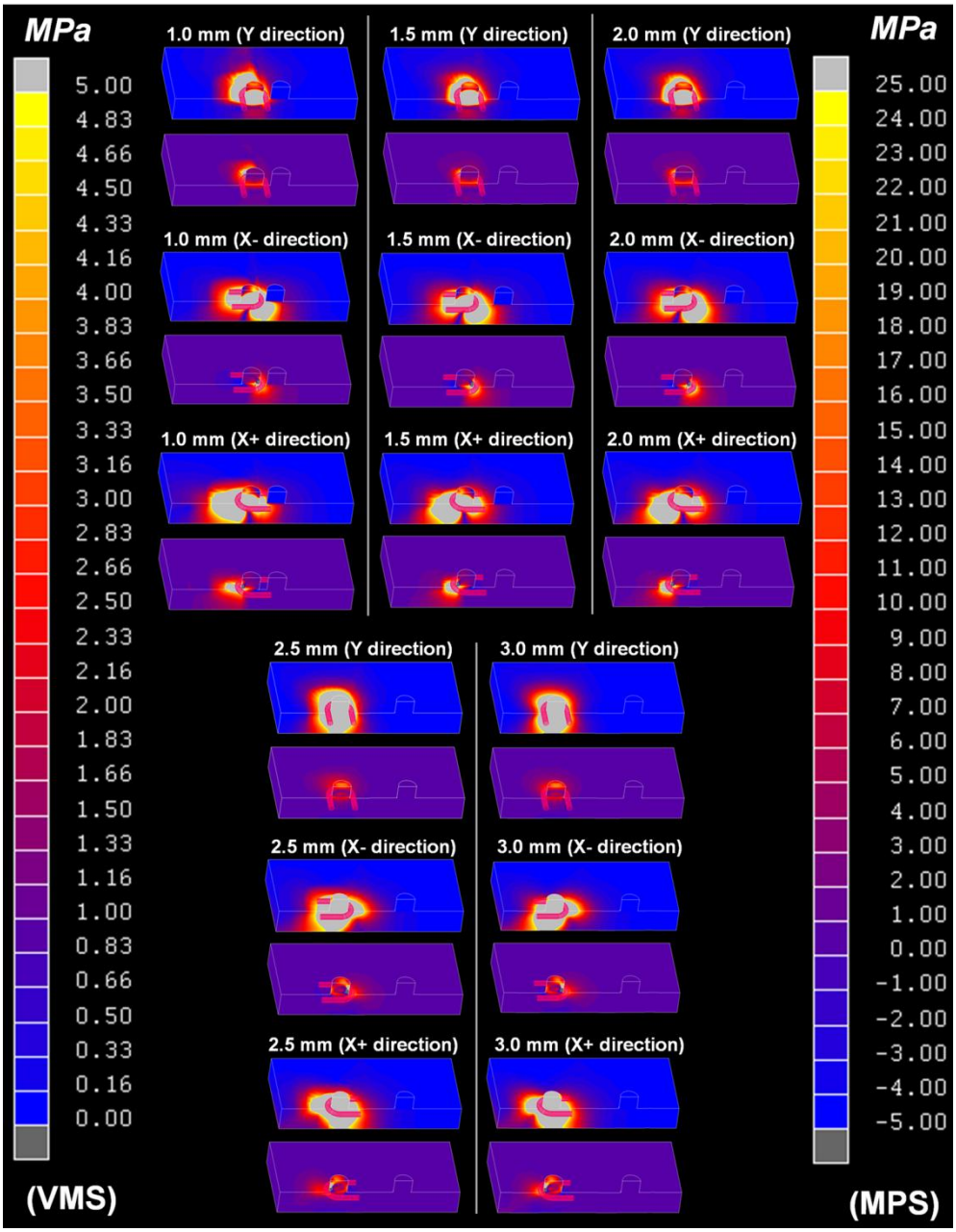

Figure 1 – Finite element results for the cylinder spacing and loading directions: VMS- von Mises stress analysis for the upper images in each row; MPS – Maximum principal stress analysis for the lower images in each row.

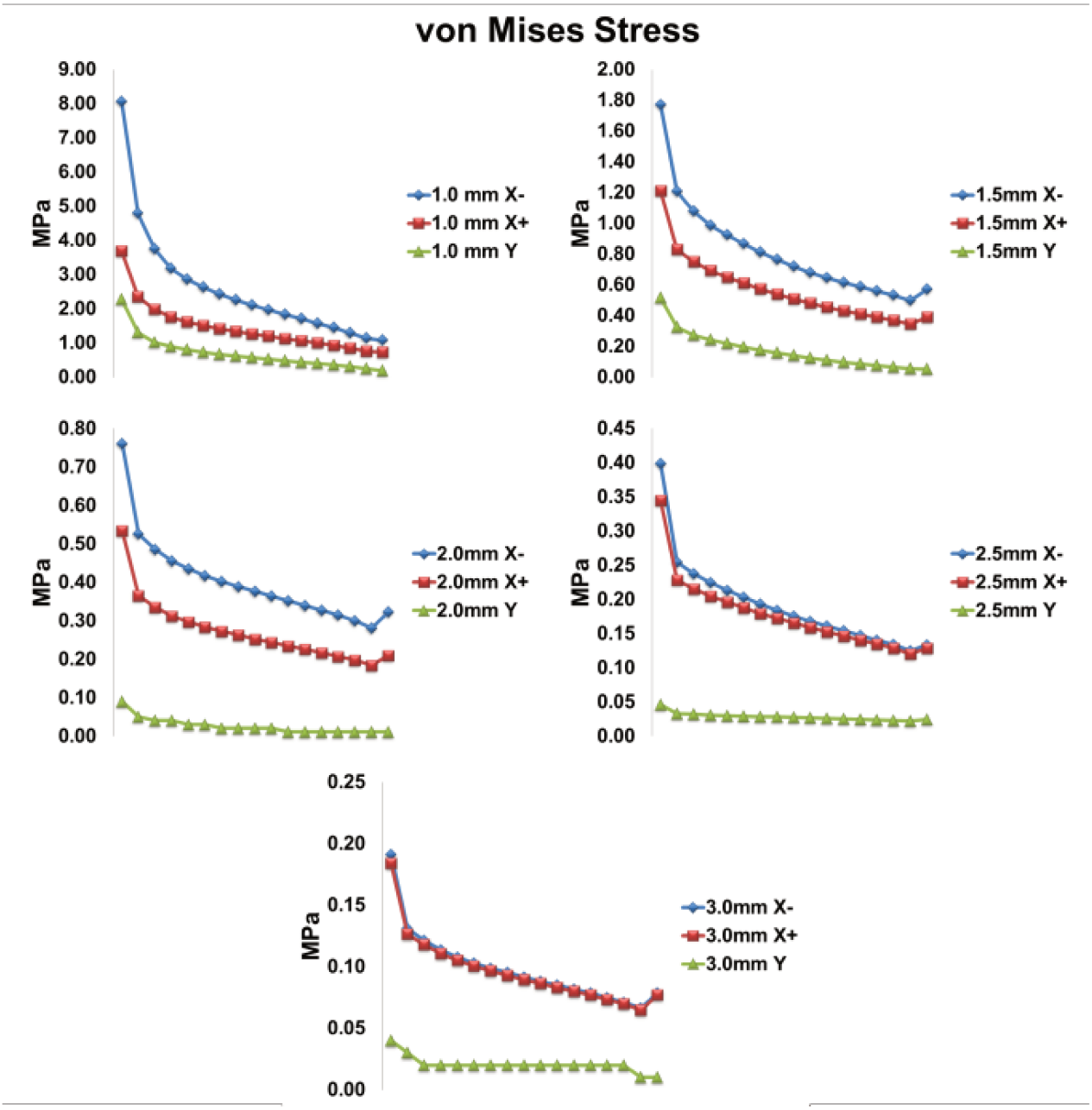

Figure 2 – Graphs plotting finite element results of the adhesive layer for the cylinder spacing and loading directions using von Mises stress.

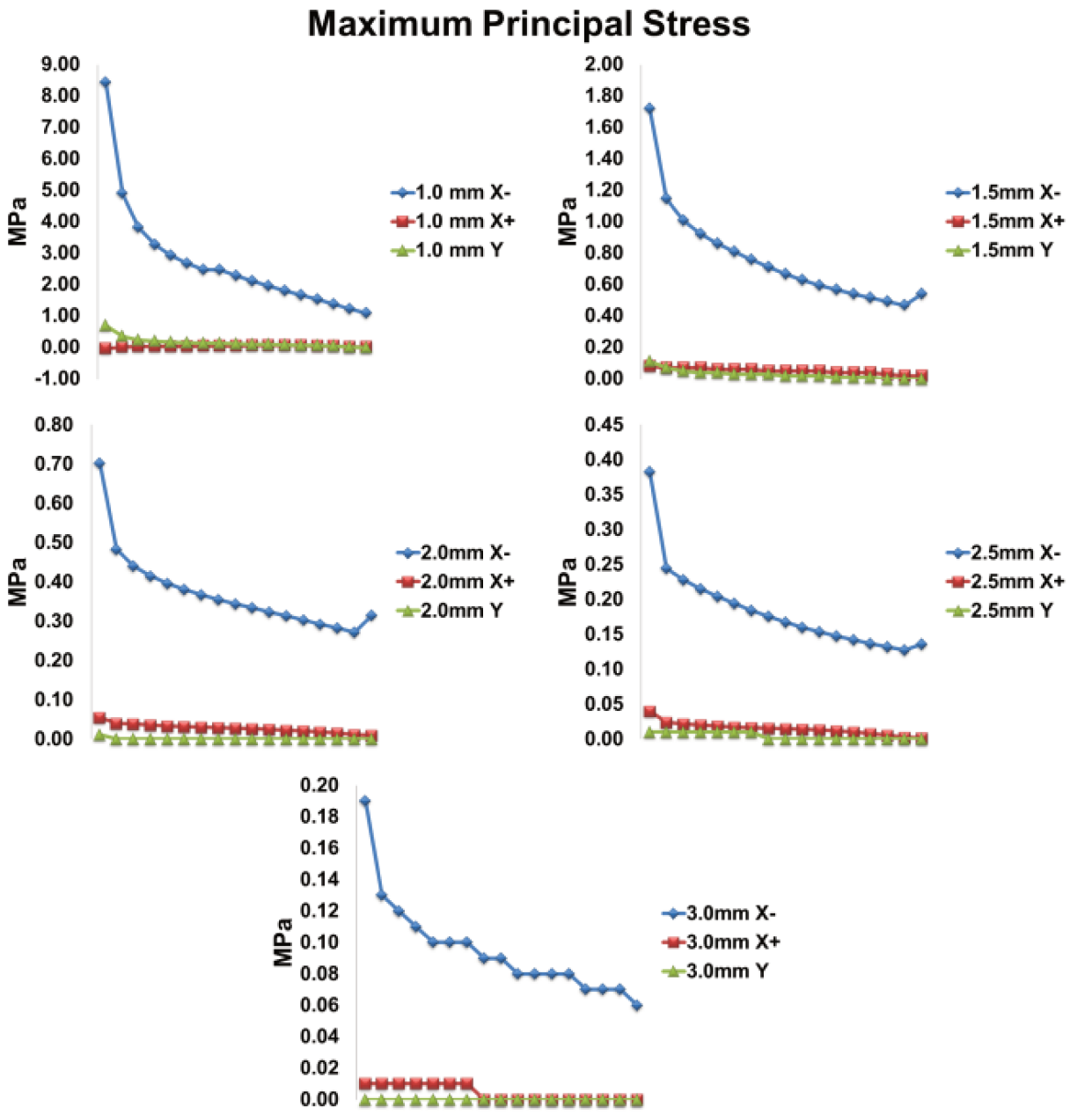

Figure 3 – Graphs plotting finite element results of the adhesive layer for the cylinder spacing and loading directions using maximum principal stress.

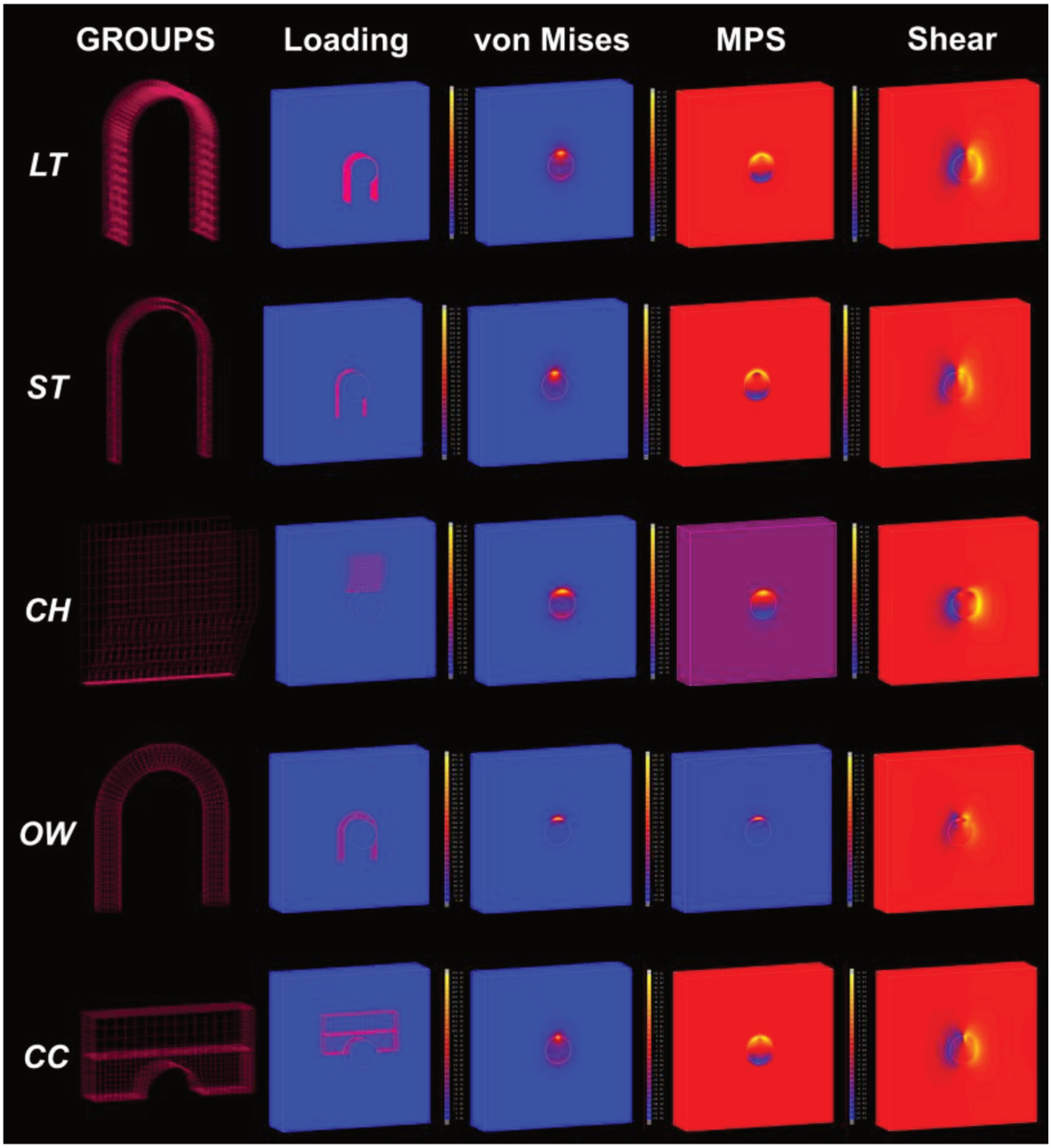

Figure 4 – Finite element results for the different load applicators: LT– large tape; ST- small tape; CH- chisel; OW- orthodontic-looped wire; CC- Customized chisel.

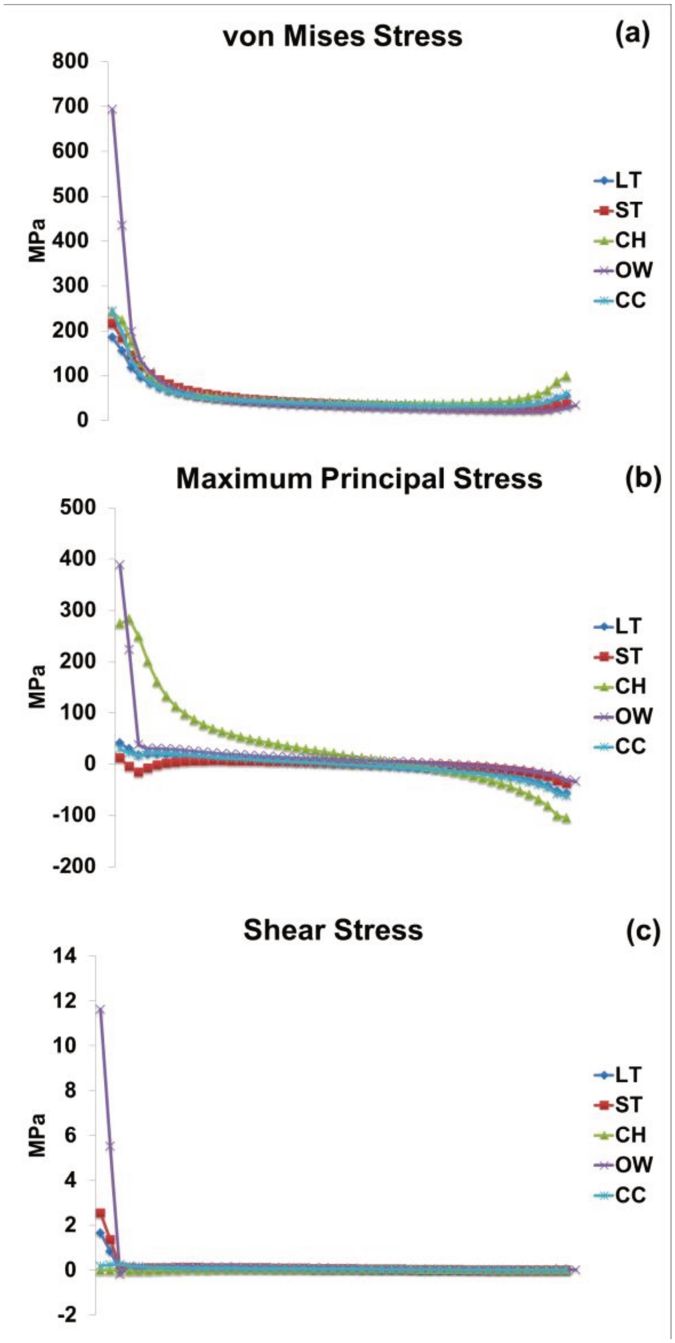

Figure 5 -Graphs plotting finite element results of the adhesive layer forthe different load applicators: LT– large tape; ST- small tape; CH- chisel; OW- orthodontic-looped wire; CC- Customized chisel.

DISCUSSION

The hypothesis proposed by this investigation that the microshear testing parameters could influence the results of finite element simulations must be accepted, since the stress distribution at the adhesive layer of the resin cylinders was influenced by all the testing parameters evaluated. Specimens loaded in any direction (Y, X-, X+) can develop undesired stresses at the adhesive layer of the adjacent non-loaded cylinders when cylinders are placed closer than 2.5 mm from each other (Figs. 1-3). This fact may impact the bonding strength of these cylinders to the substrate, since stresses are reaching their adhesive interface even before they are tested, what can lead to unreliable bond strength data. This scenario can be worsened when more than two cylinders are present in the same specimen, what is very common for this methodology. A 3.0 mm separating space between the cylinders seems a safe distance to avoid unwanted stress to reach the adhesive interface of the non-loaded cylinders during testing (Figs. 1-3). Some space can be previously obtained when using the micro bore tygon tubing (TYG-030, Small Parts Inc., Miami Lakes, FL, USA) for preparing the testing cylinders [15]; however, additional distance will be normally necessary to ensure 3.0 mm spacing.

The loading direction of the testing cylinders was also showed as an important matter, because when the loaded cylinder was pulled away from the adjacent cylinder (X- direction), which is the most commonly used set up, high tensile stresses were developed at the adhesive interface of the non-loaded

cylinder for all distances (Figs. 1 and 3). Pulling the loaded cylinder towards the adjacent cylinder (X+ direction) produced lower influence over the stress distribution at the adhesive interface of the non-loaded cylinder (Figs. 1 and 3). However, this loading direction is quite complex to be used due to the risk of contacting the non-loaded cylinders during testing.

Thus, pulling the loaded cylinder perpendicularly to the adjacent cylinder (Y direction) seems the more suitable loading direction (Figs. 1 and 3), since lower stresses were checked at the adhesive interface of the non-loaded cylinder for all distances with this configuration (Figs. 1-3). As loading cylinders perpendicularly to each other would require successive reattachments of the ceramic plates at the testing support, a flat rolling device may be used to move the specimens without detaching them. Therefore, performing microshear test with the testing cylinders separated by a 3.0 mm space and loaded perpendicularly assured reduced influence on the stress distribution for the adjacent cylinders, which may lead to more reliable laboratory bond results.

The influence of different loading applicators on the conventional shear test was previously showed [5,35], but not for the microshear test yet. The loading systems produced very distinct stress patterns at the adhesive interface of themicroshear specimens (Figs. 4 and 5). The CH and OW loading systems presented high tensile stresses prevailing over shear stresses (Figs. 5), possibly due to some bending moment at the cylinder due to the conformation of the applicators [35]. Additionally, the OW system also showed the highest load stress

peak (Fig. 5a), with the higher tensile and shear stresses at the interface (Figs. 5b and c). This is in accordance with published findings for the conventional shear test, which showed high shear bond strength values when the orthodontic-looped wire was used [35]. The higher strength values for the orthodontic-looped wire may be due to the mechanics of the test and did not necessarily represent increased bond strength [7].

The ST and LT loading systems exhibited similar performance, presenting predominantly shear stresses with very low tensile stresses (Figs. 5b and 5c). Also, the CC system presented low tensile and shear stresses with uniform stress distribution (Figs. 5b and 5c). This probably occurred due to the almost full conformation of these last three applicators to the testing cylinder, resulting in more shear stresses at the adhesive interface compared to the other loading systems (Fig. 5). Despite the stainless-steel tapes have been shown to present low bond strength values in the conventional shear approach, the debonding values depend on complex stress combination occurring in the experimental testing [35]. Thus, the two tape-based applicators showed the most adequate behavior and simple execution for being used in microshear tests since practically only shear stresses were verified at the adhesive interface of the cylinders (Fig. 5), what may result in more reliable bonding outcomes.

Microshear testing is a very useful methodology,which allows reduced failures to be incorporated to the specimens, assuring effective bonding assessments[13-15]. Its feasibility has been proved in several investigations

[18,20]. However, large variation still exists in the available bonding results due to the poor standardization of the testing parameters used on bonding studies [1,11].As seen, small variations in the microshear test parameters can result in very different stresses at the adhesive interfaces of the specimens and consequently in distinct shear bond strength values, which make the comparative clinical performance of resin materials difficult. The research design of this study presents some intrinsic limitations, such as the use of numeric analysis only. However, our findings are in accordance to previous investigations for conventional shear test. Future studies with experimental assessments which overcome these limitations will be of benefit.

CONCLUSIONS

Within the limitations of this study, the following conclusions were drawn:

1. Specimens with cylinders placed closer than 2.5 mm and loaded in any direction can cause undesired stresses at adjacent non-loaded cylinders;

2. A 3.0 mm separating space between the cylinders seems a safe distance to avoid unwanted stress to reach the non-loaded cylinders;

3. Loading the cylinders perpendicularly to each other (Y direction) with straight-aligned cylinders appears the most suitable load condition;

4. Models loaded with small (ST) and large (LT) stainless-steel tapes presented high shear stresses with almost none tensile stresses.

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CONSIDERAÇÕES GERAIS

Ensaios mecânicos têm sido frequentemente aplicados na avaliação do comportamento físico-mecânico dos materiais odontológicos. Esses testes são essenciais para o estudo, desenvolvimento e implementação dos materiais restauradores. Os testes mais comumente utilizadosnaodontologia são empregadosna verificação das propriedades mecânicas ou da qualidade/resistência de união na avaliação dos materiais(Armstrong *et al.*, 2010).Entretanto, muitos dos testes utilizados para caracterização dos materiais restauradores não são realizados nos padrões necessários, levando a resultados ambíguos para materiais similares. A falta de parametrização dos ensaios mecânicos aplicados na avaliação dos materiais odontológicos tem sido fator de frequentes investigações como forma de se obter resultados laboratoriais mais consistentes (Betamar *et al.*, 2007b; Betamar *et al.*, 2007a; Ghassemieh, 2008; Raposo *et al.*, 2012), possibilitando assim, predizer o desempenho dos mesmos nas aplicações clínicas com maior confiabilidade.

Diante dos resultados obtidos no presente estudo, pode se determinar que os ensaios mecânicos utilizados na caracterização de materiais odontológicos necessitam de maior padronização em sua execução e controle na aplicação dos mesmos. Foi verificado que o ensaio de flexão de três pontos convencional resultou em valores discrepantes de módulo de elasticidade para um mesmo material resinoso, sendo que esta propriedade variou de acordo com as configurações utilizadas no teste, tais como dimensões dos espécimes e distância

entre os apoios. Além disso, todos os resultados de módulo de elasticidade obtidos com o teste de flexão de três pontos para a mesma resina composta foram inferiores ao valor obtido utilizando o teste de microdurezaKnoop, que por sua vez demonstrou resultados similares aos da literatura (El-Safty *et al.*, 2012). Isso se deve provavelmente ao fato de que os espécimes testados no teste de flexão de três pontos são flexionados até à fratura dos mesmos e que as fórmulas empregadas no cálculo do módulo de elasticidade utilizam a força máxima obtida durante a flexão, levando em consideração não só o regime elástico do material, mas também o regime plástico e o limite de ruptura.

Desta forma, uma propriedade que idealmente deveria ser única a um dado material, acaba sendo distorcida pelas configurações utilizadas no ensaio e pelo tipo e dimensões dos espécimes. Esse fato demonstrou a limitação do TPB no cálculo desta propriedade tão importante na caracterização dos materiais, evidenciando que este teste não é o mais indicado para ser utilizado na mensuração do módulo de elasticidade de materiais odontológicos. A aplicação do mesmo deveria ser restrita na verificação da resistência flexural e tenacidade à fratura dos materiais (Christiansen *et al.*, 1974; Cooper, 1977). Além disso, as indicações para confecção dos espécimes e configuração do ensaio de flexão de três pontos descritas nas normas técnicas existentes deveriam ser estritamente seguidas (ISO178, 2010), respeitando sempre o proporcionamento adequado para que o teste seja executado de maneira correta.

Diversos ensaios mecânicos, estáticos ou dinâmicos, podem ser utilizados para mensuração do módulo de elasticidade dos materiais odontológicos. Entretanto, como visto, o teste de flexão de três pontos convencional se mostrou inadequado para esta aplicação. Assim, foi proposta associação de metodologias como forma de superar as limitações deste ensaio, sendo empregado o teste de flexão de três pontos em conjunto com extensometria. A utilização de um extensômetro fixado no lado de tração da barra (região inferior) permitiu mensurar a deformação sofrida pelo espécime no teste de flexão de três pontos durante o regime elástico do material, sem que houvesse a necessidade de testar o espécime até que o regime elástico e o limite de ruptura do mesmo fossem atingidos.

Os resultados de módulo de elasticidade obtidos utilizando esta nova metodologia foram similares aos obtidos utilizando o teste de microdurezaKnoop, que por sua vez, propiciou resultados comparáveis aos disponíveis na literatura para o aço e a resina composta avaliados neste estudo (ASM, 1990; El-Safty *et al.*, 2012). Além disso, a nova abordagem envolvendo a flexão de três pontos associada à extensometria propiciou resultados de módulos de elasticidade mais confiáveis quando comparada com o teste de flexão de três pontos convencional que forneceu valores alterados para esta propriedade. Portanto, a limitação do teste de flexão de três pontos convencional na mensuração do módulo de elasticidade de materiais odontológicos foi superada com a associação da extensometria, permitindo a verificação desta propriedade em uma configuração mais simples que a do teste de tração convencional, que exige corpos de prova

com maiores dimensões, limitando ensaios envolvendo materiais restauradores odontológicos em muitos casos.

A verificação da resistência de união de materiais restauradores a diferentes substratos também figura como ensaio mecânico de grande importância, sendo que o teste de microcisalhamento tem sido bastante utilizado com este propósito em diversos estudos (Adebayo *et al.*, 2012; Hariri *et al.*, 2012; Hori & de Carvalho, 2012; Mobarak & El-Badrawy, 2012; Yousry, 2012). Entretanto, assim como alterações nas configurações do teste de microtração resultam em valores discrepantes de resistência de união (Ghassemieh, 2008; Raposo *et al.*, 2012), modificações realizadas no teste de microcisalhamento também podem gerar dados divergentes. Foi verificado por meio deste estudo que a forma de posicionamento dos cilindros de microcisalhamento e a direção do carregamento aplicado sobre os mesmos durante o ensaio influenciaram a distribuição de tensões na interface adesiva do cilindro adjacente ao espécime no qual a força foi aplicada.

Para evitar que tensões indesejáveis atinjam a interface adesiva dos cilindros adjacentes ao cilindro em teste, foi detectado que um espaçamento mínimo de 3,0 mm entre eles se faz necessário. Um espaçamento inferior entre os cilindros fez com que tensões atingissem a interface adesiva do cilindro adjacente, o que laboratorialmente, poderia levar a uma diminuição artificial da resistência adesiva desse cilindro pelo fato do mesmo ter sido acometido por tensões antes mesmo de ser testado. A direção de carregamento do cilindro em teste também influenciou a distribuição de tensões na interface adesiva do cilindro adjacente,

indicando que quando o cilindro em teste é carregado em direção contrária ao cilindro adjacente, que é o método mais comumente utilizado, tensões podem acometer a interface adesiva do segundo, também podendo causar uma diminuição artificial da resistência adesiva desse cilindro. O carregamento do cilindro em teste perpendicularmente aos demais cilindros promoveu menor acúmulo de tensões na interface adesiva dos cilindros adjacentes, sendo, portanto, mais indicado.

Além disso, o aplicador utilizado para carregamento dos espécimes de microcisalhamento se apresentou como fator determinante do tipo e distribuição de tensões presentes na interface adesiva do cilindro em teste, de acordo com o que foi demonstrado previamente para o teste de cisalhamento convencional (Sinhoreti *et al.*, 2001). Foi demonstrado que apesar do maior carregamento incidido no cilindro pelo fio ortodôntico, altas tensões de tração prevalecem sobre as tensões cisalhantes na interface adesiva quando esta configuração é utilizada. A utilização de um cinzel também resultou em altas tensões de tração com baixo nível de tensões cisalhantes na interface adesiva do cilindro. O cinzel customizado resultou em baixas tensões de tração e cisalhamento na interface adesiva, devido ao menor carregamento incidido no cilindro de teste. O emprego de fitas de aço como aplicadores no teste de microcisalhamento produziu carregamento do cilindro com predominância de tensões cisalhantes na interface adesiva do mesmo, com níveis mínimos de tensões de tração. Esta última configuração se mostrou como a mais adequada, devido a sua aplicabilidade e por propiciar

primariamente que tensões de cisalhamento atuem na interface adesiva dos espécimes, de acordo com o propósito do teste de microcisalhamento.

Em suma, o presente estudo demonstrou que alguns testes empregados na verificação de materiais odontológicos têm suas configurações modificadas de tal forma, que resultados bastante divergentes podem ser obtidos para um mesmo material. Algumas aplicações dos ensaios mecânicos podem mesmo exceder a indicação destes, fazendo com que resultados pouco confiáveis sejam obtidos. Assim, é indicado maior padronização dos ensaios mecânicos utilizados no teste de materiais odontológicos, devendo estes, serem executados de acordo com as normas adequadas de forma que os mesmos ofereçam resultados mais confiáveis e que possuam maior validade clínica. A aplicação de novas metodologias é encorajada no sentido de romper paradigmas que possam existir sobre a caracterização dos materiais odontológicos.

CONCLUSÃO

Dentro das limitações do presente estudo, as seguintes conclusões puderam ser obtidas:

1. Deve existir maior padronização do ensaio de flexão de três pontos, respeitando-se o correto proporcionamento dos espécimes de acordo com normas específicas além de uma relação mínima entre a distância dos apoios e a altura dos espécimes.O teste de flexão de três pontos não se mostrou adequado para mensuração do módulo de elasticidade dos materiais testados.
2. O teste de flexão de três pontos associado ao método daextensometria se mostrou efetivo na obtenção do módulo de elasticidade dos materiais testados sem que houvesse a necessidade de fratura dos espécimes;
3. Espécimes do teste de microcisalhamento devem serposicionados com distância mínima de 3,0 mm entre os cilindros, de forma a minimizar os efeitos de tensões sobre os cilindros adjacentes ao cilindro em teste.O carregamento dos cilindros deveria ser idealmente realizado perpendicularmente aos cilindros adjacentes utilizando aplicadores de fita de aço inoxidável.

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*De acordo com a norma da UNICAMP/FOP, baseadas na norma do InternationalComiteeof Medical JournalEditors – Grupo de Vancouver. Abreviatura dos periódicos em conformidade com o Medline.

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APÊNDICE

Materiais e Métodos - *Capítulo 1*

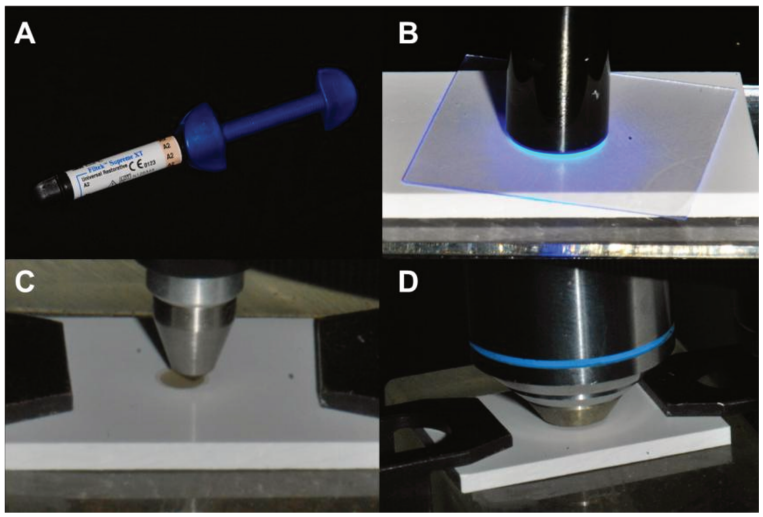

Figura 1 – Avaliação da microdureza Knoop de resina composta nanoparticulada (A) inserida em incremento único e fotoativada por 20 s em matriz de PVC (B) para mensuração do módulo de elasticidade da mesma (C e D).

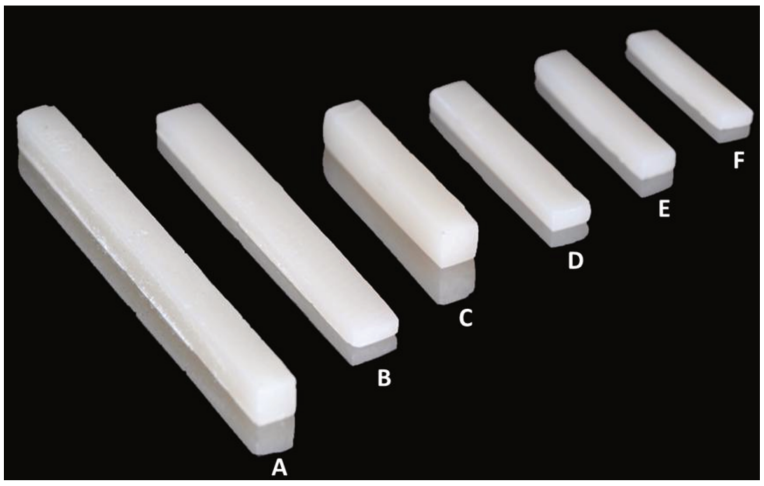

Figura 2 – Diferentes dimensões das barras confeccionadas em resina composta avaliadas no teste de resistência flexural de três pontos (*l* x *w* x *h*): A- 25x2x2 mm; B- 20x2,5x1 mm; C- 12x2x2 mm; D- 12x2x1 mm; E- 10x2x1 mm; F- 6,5x2x1 mm.

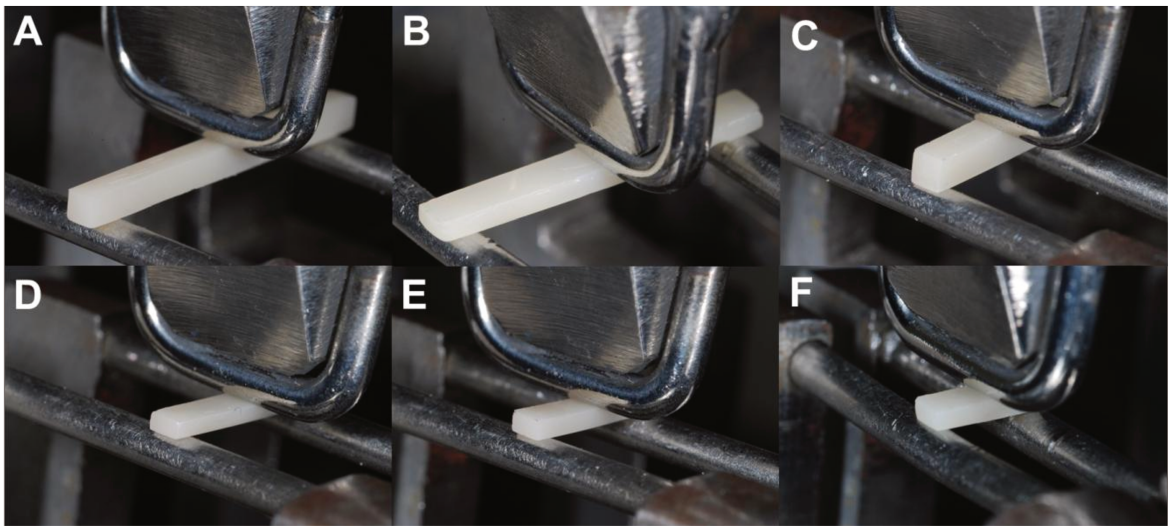

Figura 3 – Diferentes parâmetros empregados no teste de flexão de três pontos: A- ISO 4049, 2009; B- ISO 178, 2010; C- Yap & Teoh, 2003; D- Boaro *et al.*, 2010; E- Pick *et al.*, 2010; F- Oliveira *et al.*, 2012.

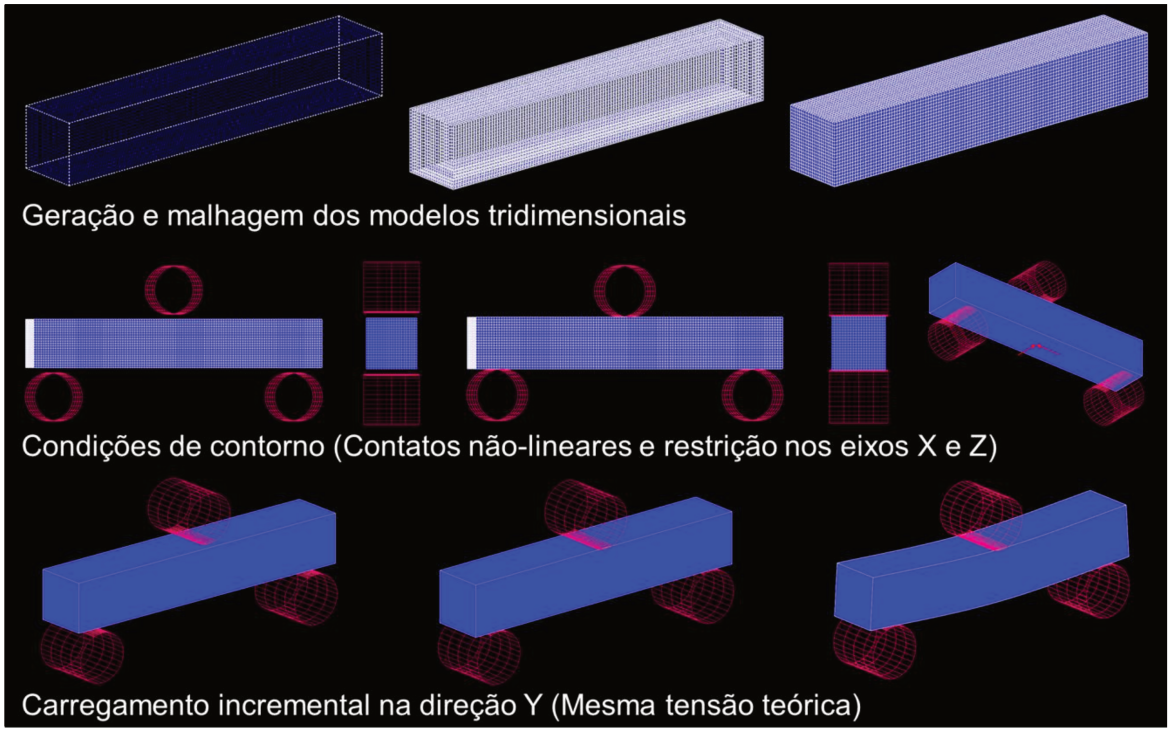

Figura 4 – Desenvolvimento dos modelos tridimensionais simulando o teste de flexão de três pontos para análise pelo método de elementos finitos.

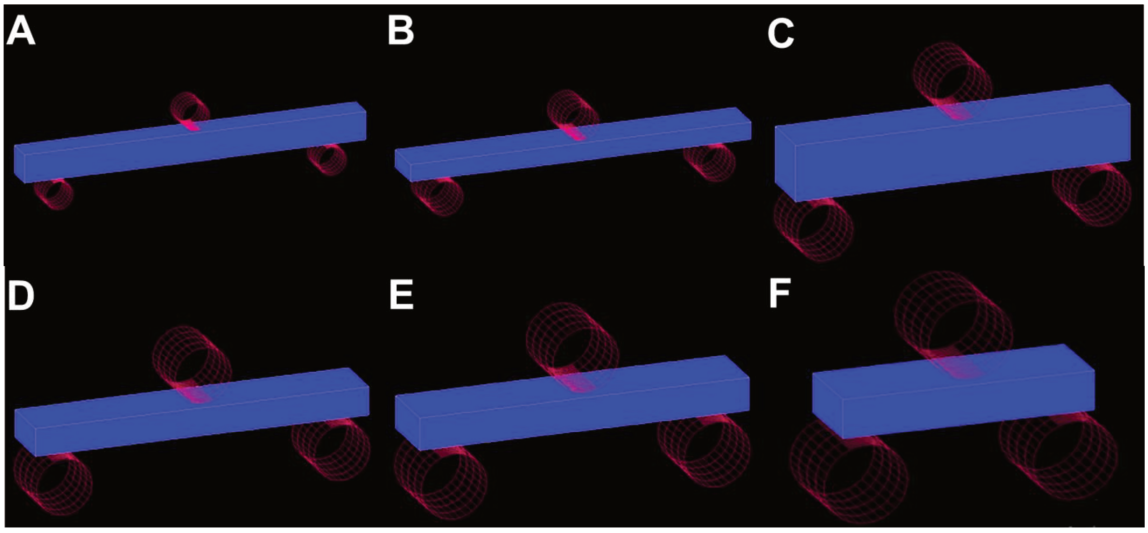

Figura 5 - Modelos tridimensionais gerados de acordo com diferentes parâmetros do teste de flexão de três pontos para análise pelo método de elementos finitos: A- ISO 4049, 2009; B- ISO 178, 2010; C- Yap & Teoh, 2003; D- Boaro *et al.*, 2010; E- Pick *et al.*, 2010; F- Oliveira *et al.*, 2012.

Materiais e Métodos - *Capítulo 2*

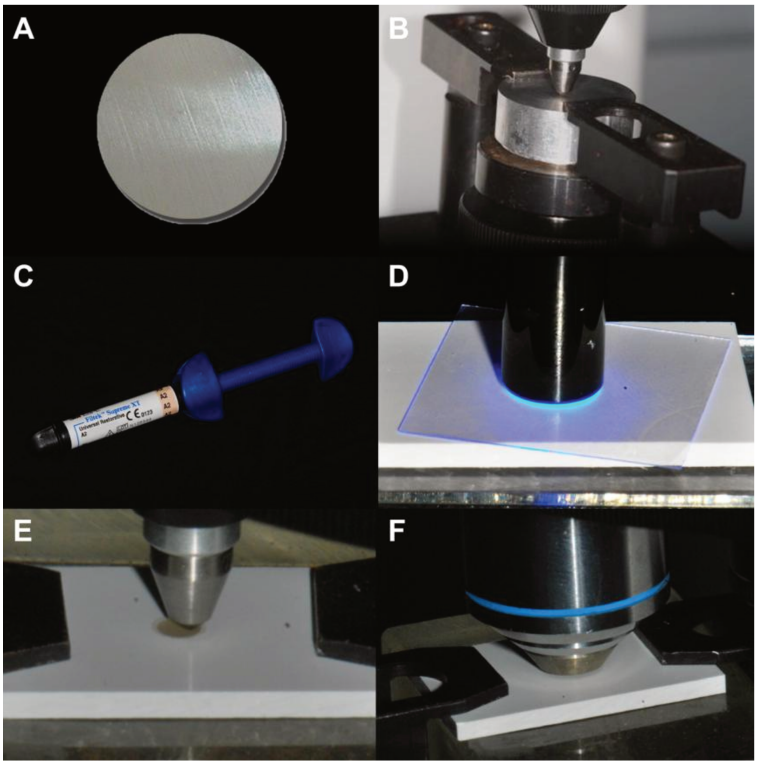

Figura 1 - Avaliação da microdureza Knoop de espécimes polidos de alúmínio (A e B) ede resina composta (C a D) para mensuração do módulo de elasticidade.

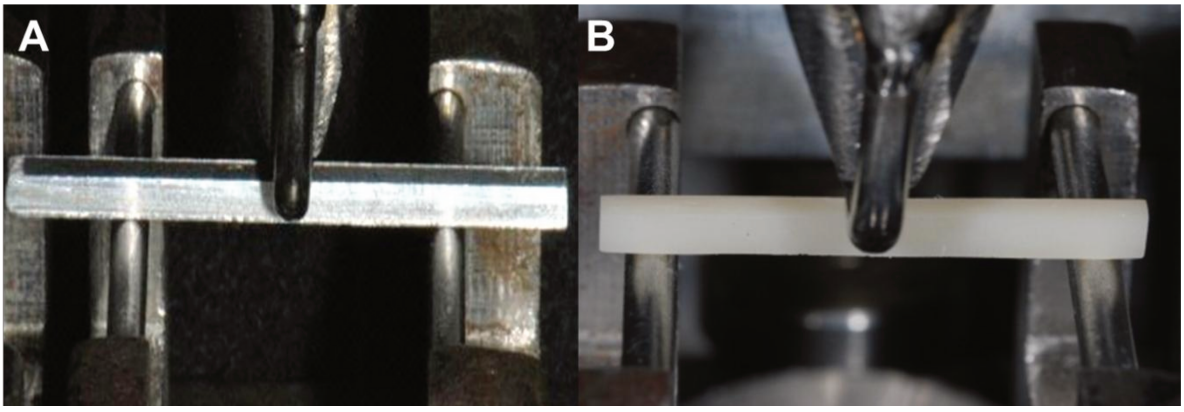

Figura 2 – Teste de flexão de três pontos convencional: espécimes em forma de barra (25x2x2 mm - ISO 4049) de alumínio (A) e resina composta (B).

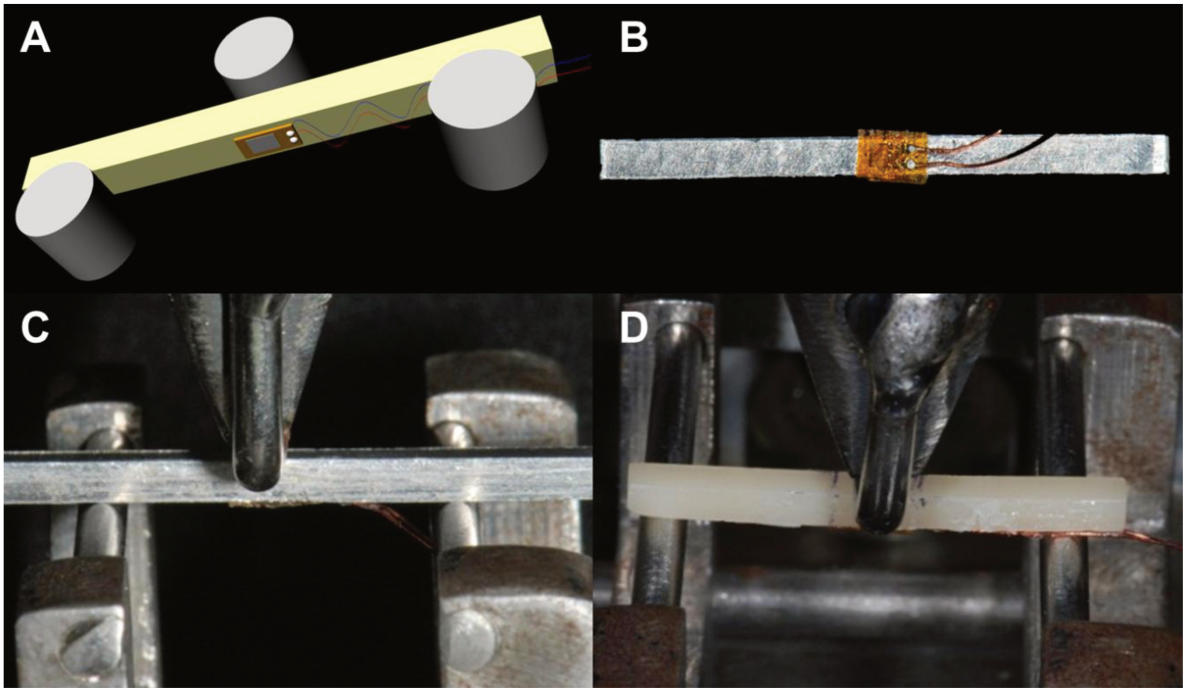

Figura 3 – Teste de flexão de três pontos associado à extensometria: A- espécimes em forma de barra (25x2x2 mm - ISO 4049) com extensômetro aderido no centro da face inferior; B- Extensômetro aderido em barra de alumínio; C e D- Espécimes de alumínio e resina composta com extensometros aderidos carregados em ensaio de flexão de três pontos.

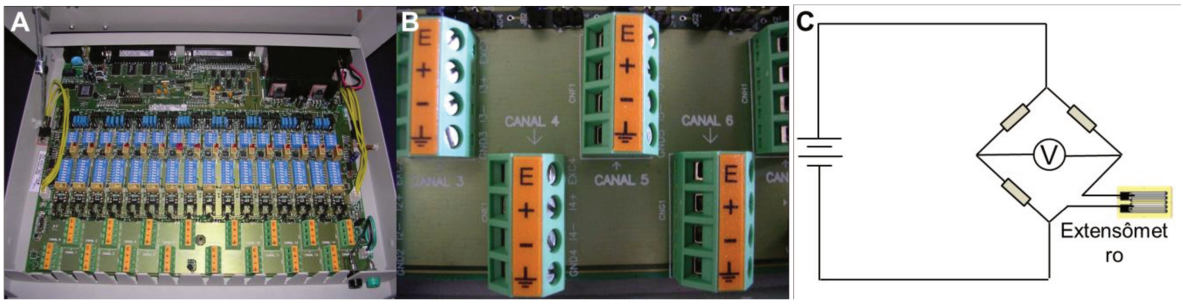

Figura 4 – A- Placa de aquisição de dados; B- Conexões da placa; C- Esquema de conexão dos extensômetros em um quarto de ponte de Wheatstone.

Materiais e Métodos - *Capítulo 3*

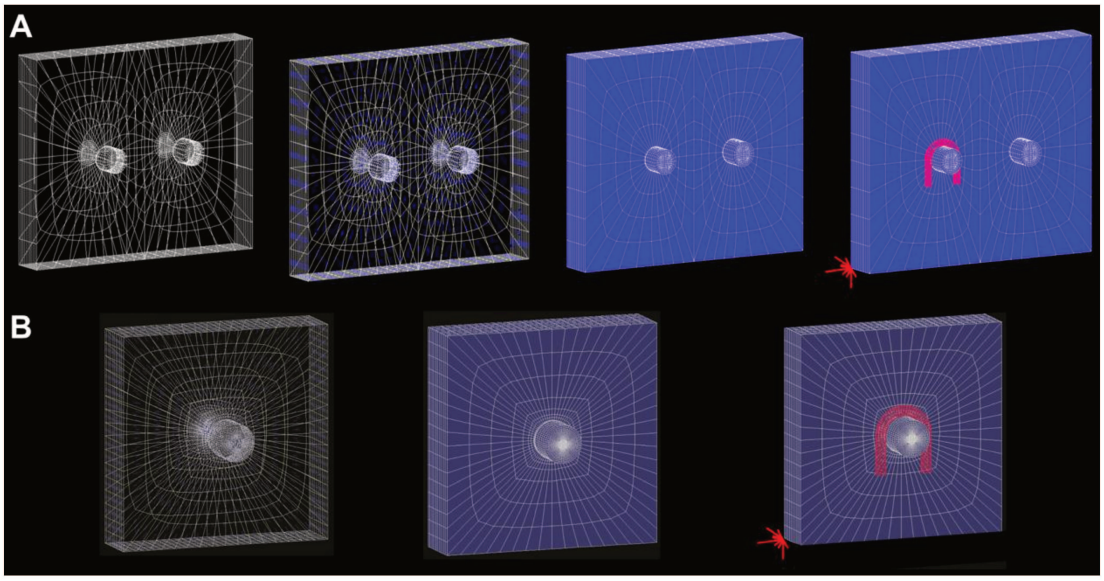

Figura 1 - Desenvolvimento de modelos tridimensionais simulando o teste de microcisalhamento: A- considerando dois cilindros de cimento resinoso em placa cerâmica; B- considerando um cilindro de cimento resinoso em placa cerâmica.

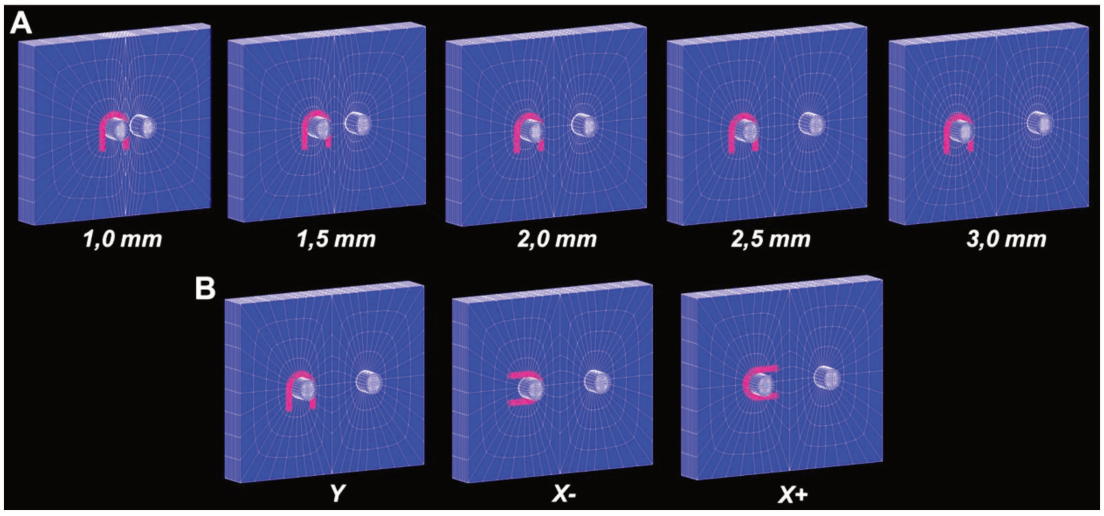

Figura 2 – Modelos tridimensionais: A- posicionamento dos cilindros de teste; B- direção de carregamento dos cilindros.

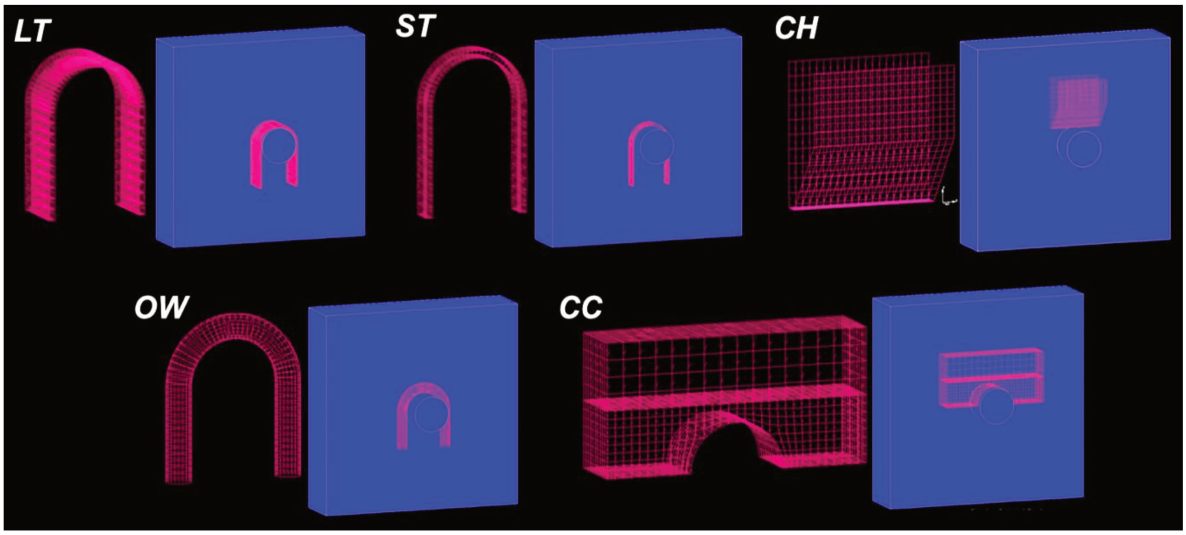

Figura 3 – Simulação das pontas aplicadoras de carga no teste de microcisalhamento: LT- fita larga; ST- fita estreita; CH- cinzel; OW- fio ortodôntico; CC- cinzel customizado.

ANEXO

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Ribeirão Preto, 21 de abril de 2013.

Prezado Professor,

Acusamos o recebimento do *"Effect of specimen positioning and loading on microshear bonding outcomes: a non-linear finite element analysis"*.

Informamos que seu trabalho será enviado para avaliação do corpo editorial. Para esclarecimento posterior o número de registro do seu trabalho é BDJ 740.

Atenciosamente,

Prof. Dr. Manoel Sousa Neto
Editor
Brazilian Dental Journal