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Análise Mecânica de Retentores Intra-Radiculares

Tese apresentada à Faculdade de Odontologia de Piracicaba, da Universidade Estadual de Campinas, para obtenção do Título de Doutor em Materiais Dentários.

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RESUMO

Este estudo avaliou os aspectos mecânicos de retentores intra-radiculares pré-fabricados reforçados com fibra, de forma progressiva por meio de etapas distintas e seqüenciais, divididas em três experimentos. No Capítulo 1, foram avaliados parâmetros como: efeito da conicidade do pino, distância entre os suportes, e propriedades dos pinos na flexão e tensão máxima pelo método de elementos finitos na realização de ensaios de flexão de 3 pontos. Foi gerado modelo tri-dimensional de pino de fibra de vidro (RelyX post, 3M-Espe), variando posições dos suportes inferiores e inclinação do pino. Empregou-se propriedades elásticas, características ortotrópicas e isotrópicas. A inclinação dos pinos cônicos para nivelá-los nos suportes teve pouco efeito nas tensões. A flexão aumentou quando 50% da porção carregada do pino envolveu conicidade. Quando o posicionamento do pino envolveu 20% da porção cônica, os valores de flexão foram similares ao modelo de referência, que é o pino cilíndrico, sem inclinação. Propriedades ortotrópicas resultaram em aumento da flexão comparado ao pino isotrópico. No Capítulo 2, propriedades mecânicas de diferentes sistemas de pinos pré-fabricados foram avaliadas por ensaio experimental de flexão de três pontos, assim como análise da correlação entre propriedades mecânicas e características estruturais visualizadas por microscopia eletrônica de varredura associado à software de processamento de imagens. Os resultados demonstraram que as características estruturais afetaram significativamente as propriedades mecânicas dos pinos. Resistência à flexão está diretamente correlacionada com a razão fibra/matriz de resina, enquanto módulo de flexão está inversamente relacionado ao número de fibras/mm^2. No Capítulo 3, foi avaliada resistência de união de pino de fibra ao núcleo de preenchimento de resina composta por meio do teste de push-out, variando o tipo de agente silano: três silanos pré-hidrolizados e um sistema de silano de dois componentes; e a temperatura de secagem com ar após aplicação do silano: 23°C e 60°C. Empregou-se ainda grupo controle negativo, no qual nenhum tratamento do pino foi realizado. A aplicação de silano préhidrolizado e secagem com ar quente (60°C) dos pinos não influenciou a resistência de união. O agente silano de dois componentes com temperatura de secagem de 23°C apresentou os maiores valores de resistência de união. Desta forma, dentro das limitações dos três experimentos, conclui-se que teste de flexão de três pontos empregado para avaliação de pinos pré-fabricados é válido quando limita a área de ensaio à porção cilíndrica dos pinos; as características estruturais dos pinos têm correlação com as propriedades mecânicas dos mesmos; e a aplicação de ar quente sobre as superfícies de pinos silanizados não tem efeito significativo na resistência de união entre pinos de fibra e preenchimento com resina composta.

Palavras-chave: pino reforçado com fibra, propriedades mecânicas, elementos finitos, microscopia eletrônica de varredura, silano, push-out.

ABSTRACT

The fiber-reinforcement composite (FRC) posts introduced the new concept of restorative system, in which the various components of the reconstruction: post, cement, filling material and dentin have now become a complex mechanically homogeneous. The knowledge of the structure, composition and physical properties of these fiber posts systems is important in order to minimize the failures and the unsuccessfully clinical. Thus, the present study was conducted to analyze sequentially the biomechanical parameters of FRC posts through different stages divided into three experiments. In Chapter 1, the effect of taper, specimen supports and the properties on flexure and stress response during three-point bending were analyzed using finite element analysis. Three-dimensional non-linear finite element model of a fiber-reinforced composite post was created. Different support positions were evaluated during a simulated three-point bending test. The applied properties were elastic and orthotropic. Tilting the tapered posts to level those in the test setup had little effect on the outcome. Flexure increased when 50% of the bent portion involved taper. If 20% of the bent post involved taper, the flexure values were close to control group. The orthotropic properties also resulted in increased flexure compared to an isotropic post. Maximum stresses were only a little higher when 50% of the bend structure involved taper, while the orthotropic properties had little effect. In Chapter 2, the mechanical properties of different FRC posts were assessed with three-point bending test and evaluated the correlation between mechanical properties and structural characteristics by scanning electron microscopy (SEM) associated with an software of image processing. Then, the structural characteristics significantly affect the mechanical properties of fiber posts. The flexural strength is directly correlated with fiber/matrix ratio, whereas the flexural modulus is inversely correlated with number of fibers per mm2 of post. In the Chapter 3, was evaluate the effect of three prehydrolyzed silanes and one two-bottle coupling agent and two air-drying temperature (23°C and 60°C) on the bond strength between glass fiber posts and composite resin core using micropush-out testing. Additionally, it was tested a control group, which no treatment of fiber post were realized. The results showed that drying with warm air and post silanization with pre-hydrolized silanes had no significant effect on push-out bond strength between glass fiber post and composite resin core. For 23°C air-drying groups, the bond strength with two-bottle coupling agent was higher than the other groups. Then, based on the findings of this study, and within the limitation of this investigation, could be concluded that the regardless of leveling, the flexural stress determination with tapered fiber-reinforced posts in the three-point bending test was valid as long as the tapered portion was limited. Additionally, the mechanical properties have correlation with structural characteristics of FRC posts. Finally, the warm air-drying in silanizated post surface had not significant effect on bond strength between fiber post and composite resin core.

Key Words: fiber-reinforced post, mechanical properties, finite element analysis, scanning electron microscopy, silane, push out.

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INTRODUÇÃO

Dentes com severa destruição da coroa, resultado de cáries ou fraturas dentárias, desvitalizados e endodonticamente tratados, normalmente apresentam-se com perda elevada de estrutura dental, que dificulta a estabilidade de materiais restauradores coronários. Dependendo da quantidade de estrutura coronária perdida, torna-se necessária a utilização de retentor intra-radicular com finalidade principal de promover retenção e estabilidade das restaurações diretas ou indiretas ao tecido dental remanescente (Trope *et al.*, 1985; Sorensen & Engelman, 1990; Martínez-Insua *et al.*, 1998; Goto *et al.*, 2005; Santos-Filho *et al.*, 2008).

Há décadas os procedimentos reabilitadores de dentes tratados endodonticamente têm sido objetivo de vários estudos, com a finalidade de predizer o método que torna o complexo restaurador (raiz, pino intra-radicular, cimento, núcleo de preenchimento e coroa) mais resistente às forças e tensões mastigatórias. No momento do planejamento e da seleção do material que será empregado no procedimento restaurador, a opção por materiais cujas propriedades se assemelhem às propriedades da dentina radicular é importante, pois isso pode favorecer a distribuição das tensões transmitidas ao canal radicular e ao longo da interface pino/cimento (Soares *et al.*, 2008), reduzindo assim, o risco de fraturas catastróficas no remanescente radicular (Boschian *et al.*, 2006).

Os pinos metálicos têm sido comumente utilizados para ancoragem das restaurações protéticas. Entretanto, pinos metálicos fundidos ou pinos pré-fabricados metálicos, quando carregados, apresentam modo de fratura desfavorável devido a ocorrência de fraturas radiculares (Santos-Filho *et al.*, 2008). Ao contrário, os pinos pré-fabricados reforçados com fibra quando falham, causam principalmente fraturas do núcleo de preenchimento ou do próprio pino, possibilitando a substituição da restauração (Santos-Filho *et al.*, 2008). Dessa forma, materiais que mimetizem a estrutura dentária são desejáveis, de maneira que apresentem propriedades que se assemelhem ao máximo possível ao esmalte e dentina perdidos (Akkayan & Gülmaz, 2002).

Fatores como à alta taxa de fratura radicular em dentes restaurados com pinos metálicos, o aumento da demanda estética contemporânea, associada ao fato dos pinos pré-fabricados reforçados com fibra exibirem propriedades biomecânicas mais similares às da dentina do que os pinos metálicos (Plotino *et al.*, 2007; Santos-Filho *et al.*, 2008), tem motivado pesquisadores ao estudo cada vez mais crescente dos pinos reforçados com fibras.

Ensaios mecânicos de flexão são mais freqüentemente empregados na mensuração das propriedades desses pinos pré-fabricados para avaliação e melhor aplicabilidade clínica, devido à dificuldade da realização de outros ensaios que teste este tipo de material (Rodrigues Jr. *et al.*, 2008). A partir deste teste, é possível a obtenção de dados quantitativos da deformação sofrida pelo material sujeito à força de flexão. Os principais resultados encontrados são a resistência à flexão e o módulo de flexão. Entretanto, estes resultados podem variar com a distância entre os dois suportes inferiores nos quais o pino é posicionado, pela força aplicada no centro e sobre o pino, e pela geometria e diâmetro do pino (Drummond, 2000; Mannocci *et al.*, 2001; Lassila *et al.*, 2004; Galhano *et al.*, 2005; Grandini *et al.*, 2005; Plotino *et al.*, 2007; Seefeld *et al.*, 2007).

A falta de padronização das condições deste teste em estudos experimentais demonstra ampla variabilidade, principalmente em relação à distância entre os suportes inferiores e geometria e diâmetro dos pinos (Drummond, 2000; Mannocci *et al.*, 2001; Lassila *et al.*, 2004; Galhano *et al.*, 2005; Grandini *et al.*, 2005; Plotino *et al.*, 2007; Seefeld *et al.*, 2007; Novais *et al.*, 2009). Para limitar tais variações nos resultados, a padronização dos ensaios de flexão de pinos parece necessária, apesar de a determinação de um padrão universal ser de difícil aplicação, pois a conicidade e tamanho dos pinos dificultam ou limitam a adaptação a rigorosos padrões geométricos. Assim, é fundamental estudar os fatores relacionados com a metodologia do ensaio de flexão empregada em diversos estudos para análise de pinos pré-fabricados, a fim de se verificar qual a influência das variáveis do teste mecânico nos resultados, além de determinar meios que possam contribuir para a sua padronização.

Outra questão relacionada aos pinos pré-fabricados que notoriamente reflete em seu desempenho clínico é a sua constituição (Soares *et al.*, 2009). Os pinos reforçados com fibra são constituídos por elevada percentagem de fibras contínuas embebidas em matriz polimérica, normalmente resina epóxi (Seefeld *et al.*, 2007). Uma vez que os pinos de fibra são na essência materiais compósitos, parece lógico esperar que suas propriedades mecânicas aumentem com o aumento do conteúdo de fibras (Grandini *et al.*, 2005). A correlação entre propriedades mecânicas e características estruturais dos pinos reforçados com fibras pode ser influenciada por aspectos relativos à composição desses pinos, tais como concentração, arranjo ou orientação das fibras, volume de fibras (Grandini *et al.*, 2005), impregnação das fibras na matriz, propriedades individuais das fibras e matriz (Drummond, 2000) e união entre matriz e fibras (Galhano *et al.*, 2005).

Possíveis explicações para menor resistência à flexão de um pino poderia ser a fraca ligação interfacial entre fibra e matriz, gerando irregularidades, que é, provavelmente, consequência do processo de fabricação (Seefeld *et al.*, 2007), ou ainda pela presença de espaços vazios presentes na resina ou a descontinuidade ao longo da interface entre fibras e matriz (Grandini *et al.*, 2005). No entanto, ainda não está claro como as propriedades estruturais dos pinos reforçados com fibra influenciam as suas propriedades mecânicas (Seefeld *et al.*, 2007). Portanto, a avaliação das propriedades mecânicas associadas à análise estrutural de retentores intra-radiculares é importante para correta indicação e determinação do desempenho clínico.

Além das características intrínsecas destes materiais, o conhecimento dos mecanismos envolvidos no processo de união deste complexo restaurador também é essencial para o sucesso clínico (Soares *et al.*, 2008). A falha mais comum de dentes tratados endodonticamente e restaurados com pinos reforçados com fibra durante um teste de fratura é a falha envolvendo a porção coronária (Santos-Filho *et al.*, 2008). A retenção e estabilidade entre sistemas de pinos e núcleo de preenchimento é um importante fator para o sucesso restaurador (Gateau *et al.*, 1999). Sendo assim, uma das preocupações se concentra na interface pino-preenchimento, sendo o uso do agente silano um dos fatores de influência. O objetivo da aplicação do silano é auxiliar não só no aumento da imbricação micromecânica do cimento resinoso inserido na porção radicular, mas também, sua interação com o material de preenchimento que será aplicado na porção coronária (Soares *et al.*, 2008).

Uma união química pode ser obtida entre a matriz de resina composta e as fibras de vidro dos pinos que estão expostas. E para acelerar o mecanismo de interação química entre o silano e a superfície inorgânica, a reação pode ser catalisada por calor (Shen *et al.*, 2004). O tratamento com calor do vidro silanizado é rotineiramente realizado na indústria para maximizar a resistência de união (Barghi *et al.*, 2000). Segundo Monticelli *et al.* (2006), o uso de diferentes temperaturas na superfície de pinos silanizados altera os valores de resistência de união de compósitos aos pinos intra-radiculares. Isto ocorre devido à maior temperatura contribuir para evaporação do solvente presente na composição do silano, e que se persistir sobre a superfície do pino, pode prejudicar a união (Abate *et al.*, 2000). No entanto, existem poucas informações quanto à influência da secagem com ar quente após a aplicação do agente silano na resistência de união entre pinos de fibra e preenchimento de compósito.

Deste modo, o presente estudo avaliou aspectos mecânicos de pinos intra-radiculares pré-fabricados, de forma seqüencial por meio de metodologias experimentais e computacionais, divididas em três capítulos, que objetivam:

CAPÍTULO 1: Avaliar, por meio do método de elementos finitos, o efeito da conicidade e propriedades físicas do pino de fibra de vidro e da distância entre os suportes na resposta à flexão e tensão máxima durante o teste de flexão de três pontos;

CAPÍTULO 2: Analisar as propriedades mecânicas de diferentes sistemas de pinos pré-fabricados reforçados com fibras, e a possível correlação entre essas propriedades e as características estruturais;

CAPÍTULO 3: Determinar a influência do calor e de diferentes agentes silanos na resistência de união entre pino de fibra de vidro e material de preenchimento em resina composta.

CAPÍTULO 1*

Three-point bending testing of fibre posts: critical analysis by finite element analysis

Abstract

Aim Shape and non-isotropic properties of fibre posts create complications in three-point bending tests. The aim of this study was to evaluate the effect of taper, specimen supports and the properties on flexure and stress response during three-point bending using finite element analysis.

Methodology A three-dimensional finite element model of a fibre post was created. The occlusal portion was cylindrical while the apical portion was tapered. Five different support positions were evaluated during a simulated three-point bending test: M1- support distance of 10 mm centralized and no tilt; M2-10 mm centralized with tilt; M3-10 mm not centralized and no tilt; M4- 10 mm no centralized with tilt; M5- 6 mm not centralized with no tilt. A sixth post model (M6) was a centralized post without tapered section. The applied properties were elastic and orthotropic.

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Results Tilting the tapered posts to level them in the test setup had little effect on the outcome. Flexure increased when 50% of the bent portion involved taper (M1, M2). If only 20% of the bent post involved taper (M3, M4), the flexure values were close to M6 (no taper). The orthotropic properties also caused increased flexure compared to an isotropic post. Maximum stresses were only a little higher when 50% of the bend structure involved taper, while the orthotropic properties had little effect.

Conclusions It was concluded that regardless of leveling, the flexural stress determination with tapered fibre posts in the three-point bending test was valid as long as the tapered portion was limited.

Keywords: fibre post, finite element analysis, flexural properties, strength

Introduction

Non-metallic post system have been used to restore root filled teeth. Stiffness behavior of fibre posts that is closer to dentin than metallic post has been an important motivation for their acceptance (Rosentritt *et al.* 2000, Santos-Filho *et al.* 2008, Santos *et al.* 2010).

In vitro measurement of stiffness and strength properties for assessment and quality control of fibre posts is most often performed in flexural tests. In these tests, the posts are bent between three points, creating longitudinal tensile and compressive stress conditions. Tensile stresses are considered most critical, because they are most likely to initiate failure (Rodrigues *et al.* 2008). Flexural response is determined by the distance between the supports, post design and diameter (Drummond 2000, Mannocci *et al.* 2001, Lassila *et al.* 2004, Galhano *et al.* 2005, Grandini *et al.* 2005, Plotino *et al.* 2007, Seefeld *et al.* 2007). Since fibre posts come in different shapes (conical, cylindrical, cylindrical-conical), the stress distribution in posts and thus outcome of flexural tests is likely to be affected. Differences in diameter have been claimed to affect flexural strength, where thick posts return lower strength values than thin posts (Lassila *et al.* 2004). Variations in post geometry and span distance may be responsible for differences between data reported in flexural test studies (Drummond 2000, Mannocci *et al.* 2001, Lassila *et al.* 2004, Galhano *et al.* 2005, Grandini *et al.* 2005, Valandro *et al.* 2006, Plotino *et al.* 2007, Seefeld *et al.* 2007, Novais *et al.* 2009). Thus, standardization of flexural tests for non-metallic posts may be needed. Designing a universal standard, however, is complicated because posts, which often feature partial taper, cannot be easily adapted to strict geometrical standards.

The aim of this study was to evaluate the stress and deformation in various fibre post designs to create insight into the biomechanical behavior during bending tests. Finite element analysis (FEA) was used, which is a versatile tool to test interactions between physical factors (Soares *et al.* 2008). Test design parameters such as distance between supports and post orientation were varied to test the null hypothesis that they do not affect the maximum stress in the posts.

Materials and methods

Flexure and stress distributions in fibreglass posts were evaluated during three-point bending for various testing conditions using FEA (MSC.Marc, MSC.Software Corporation, Santa Ana, CA, USA). Configuring the FEA comprised of creating the geometrical models, assignment of material properties, and prescribing loading and support conditions.

The three-dimensional (3D) finite element model of the post was generated based on the dimensions of a Relyx Fiber Post (3M-ESPE, Seefeld, Germany). The total length of the post was 20 mm, where the 10 mm coronal portion was cylindrical (1.9 mm diameter) and the 10 mm apical portion tapered (apical end 0.893 mm diameter). The fibreglass model mesh consisted of 8 node hexahedral elements.

The fibreglass post was considered homogeneous, linear-elastic and orthotropic. Mechanical properties were obtained from the literature (Asmussen *et al.* 1999, De Santis *et al.* 2000, Ferrari *et al.* 2000). The longitudinal z-direction was the principal direction; x- and y-directions indicate the perpendicular directions. The elastic modulus in longitudinal direction Ez was 37.0 GPa; Ex and Ey in the perpendicular directions were 9.5 GPa. The Poisson's ratios ηzx, ηxy, and ηyz in the orthogonal planes (zx, xy and yz) were 0.27, 0.34, and 0.27, respectively. The shear moduli Gzx, Gxy, and Gyz were 3.1, 3.5, and 3.1 GPa, respectively.

The posts were bent between three supports, modeled as rigid contact bodies (2.5 mm diameter). The two lower supports were fixed. The upper support, on which the flexural load was applied, was only allowed to move along the vertical load axis. Friction between post and supports was considered negligible. Five different testing conditions were simulated with regard to the support span and position, and tilt of the post (Fig. 1):

• Model 1 (M1): 10 mm span distance between lower supports with post centered (both cylindrical and tapered portions loaded) and horizontal;

• Model 2 (M2): 10 mm span distance between lower supports with post centered (both cylindrical and tapered portions loaded) and tilted;

• Model 3 (M3): 10 mm span distance between lower supports with post positioned off-center and horizontal;

• Model 4 (M4): 10 mm span distance between lower supports with post positioned off-center and tilted;

• Model 5 (M5): 6 mm span distance between lower supports with post positioned off-center (only cylindrical portion loaded) and horizontal.

Additionally, a post without tapered portion (Model 6) was evaluated as reference, both for orthotropic (M6) and isotropic properties (M6-iso). Furthermore, analytical flexure and stress solutions were calculated for a 1.9 mm diameter post using the regular engineering bending theory expression for 6 mm or 10 mm support distances, A6 and A10, respectively.

Displacement at the load application point and the maximum tensile stress in the post were calculated and recorded during simulated flexural loading.

Results

Force-displacement curves plot the relation between the bending load during flexure and the displacement of the center of the posts (Fig. 2). Tilting specimens did not have much effect on flexure values (M1 and M2; M3 and M4). Changing the placement of posts on the supports changed the curves. Placing more of the tapered portion between the supports (M1, M2) increased flexure compared to cases where less taper was involved (M3, M4). Flexure of the untapered model (M6) was close to the M3 and M4 values. Reducing support span decreased flexure (M5). Flexure of the orthotropic post (M6) was higher than of the isotropic post (M6-iso).

Force-stress curves plot the relation between the bending load during flexure and the maximum tensile stresses found at the bottom of the posts (Fig. 3). Tilting specimens did not have much effect on flexure stress values (M1 and M2; M3 and M4). Placing more of the tapered portion between the supports (M1, M2) caused only a small increase in flexure stress compared to the cases where no or less taper was involved (M3, M4, M6). Reducing support span decreased flexure stress under the same bending load (M5). Maximum flexural stress values in the orthotropic and isotropic posts were nearly identical (M6 and M6-iso).

Discussion

Flexural tests have been used to determine stiffness and strength of non-metallic post systems (Torbjörner *et al.* 1996, Lassila *et al.* 2004, Grandini *et al.* 2005, Valandro *et al.* 2006, Novais *et al.* 2009). The International Organization for Standardization (IOS) has developed specifications for three-point bending tests of polymer-based materials (IOS). These specify rectangular or circular specimen cross-sections to be bent under a compressive loading applied at equal distance between two supports. The tensile stresses it creates in the specimen initiates the failure (Rodrigues *et al.* 2008). Since no specific standard exists for materials reinforced with fibre, non-metallic posts are often tested using the same bending setup as specified for polymer-based materials. Maximum tensile stress δ_{max} is subsequently calculated using the engineering bending theory expression:

$$\delta_{max} = 8F_{max}l/\pi d^3 \qquad [1]$$

where F_{max} is maximum applied load, l is distance between the supports (span) and d is diameter (Torbjörner *et al.* 1996, Lassila *et al.* 2004, Grandini *et al.* 2005, Valandro *et al.* 2006). Fibre posts, however, differ from the IOS-standard specimens because of their tapered sections and reinforcing fibres. The objective of this study was to investigate and discuss the complications and validity of using three-point bending experiments for testing of fibre posts.

The first practical concern of using fibre post in three-point bending tests is that many have a tapered section. How should tapered specimens be placed on the supports? Should they be leveled? The results of the current analysis show that leveling the posts had little effect on flexure and maximum stress values.

A tapered section also raises concerns about the validity of using expression [1] for calculation of maximum stresses. The expression assumes a prismatic beam (constant cross-section), and will become inaccurate for large deflections (supports too far apart), interference of the support areas (supports too close together and/or support diameter too large), or if the material is not homogeneous. In this study the maximum stress and flexure values were calculated using FEA because, unlike expression [1], FEA can take the tapered shape and orthotropic properties into account and allows a systematic investigation of experimental variables (Versluis *et al.* 2006).

By varying the position of the supports, our analysis showed that the effect of taper on flexure can be substantial. If half the span involved taper (M1 and M2), flexure increased substantially compared to the untapered post (M6). However, when only 20% of the span involved taper (M3 and M4), the effect on flexure was not substantial. The corresponding maximum stress values showed a similar, although less pronounced trend. The larger effect of taper on flexure could be expected considering its expression according to the engineering bending theory:

$$v = 4Fl^3/3E\pi d^4 \quad [2]$$

where v is flexure and E is elastic modulus. The higher order diameter term in [2] compared to [1] indicates a higher sensitivity to diameter changes for flexure than for the maximum stress.

Unlike the expressions [1] and [2], which assume a constant cross-section and isotropic properties, FEA calculated flexure and stresses in the posts with their actual geometry and property. Comparison between engineering bending theory and FEA shows a large difference for the flexure values while the maximum stress values are relatively close. Differences between flexure values can be largely attributed to the orthotropic properties. This was verified by applying isotropic properties, showing that the difference between the flexure outcomes of the two calculations narrowed (M6-iso). Post flexure is thus affected by properties in all dimensions. On the other hand, stress, which expresses how a force is distributed through a body, is determined by force-moments and cross-sectional areas. Stress values were less affected by the material properties (isotropic/orthotropic), hence the similar values obtained by the engineering bending theory and FEA.

Besides taper and properties, span distance also affects flexure and stresses because of associated bending displacement and interference of supports. When a beam flexes, its contacts change, effectively reducing span distance and even potentially affecting loading locations. In this analysis two span distances (10 mm and 6 mm) were modeled. The 6 mm span was chosen to explore if the test could be achieved on the cylindrical section alone, avoiding the complications posed by the tapered section. The analysis showed that flexure increase reduced for bending loads above 30 N for the model with 10 mm span (M6), visible as a reduction in the inclination of the force-flexure curve. For the 6 mm span (M5) the flexure increase reduced above 35 N. The observed reduction could be explained by the decrease in support distance at higher flexure values, and thus stiffer beam response. A slight increase in maximum stress value was observed above 15 N for the 10 mm span (M6) and above 20 N for the 6 mm span (M5). This increase in longitudinal tensile stress may have been the effect of the combination of flexure and friction with the immovable support positions, neither of which is taken into account in the engineering beam theory or IOS standards. However, disregarding these flexural effects, overall results for the 6 mm span (M5) were consistent with the general assessment of a common 10 mm span test design (M6), and the maximum stress in the 6 mm span obtained by the engineering bending expression can be expected to be close to the outcome of the FEA.

It thus appears that maximum stress values for fibreglass posts are acceptable using methodology similar to the IOS standards for polymer materials, provided that the taper between the supports is minimized. IOS methodology for elastic modulus determination should not be used because the flexure calculation in expression [2] is not accurate for tapered sections and non-isotropic materials.

The above assessments assumed a homogeneous distribution of material properties in the posts. Although fibres and resin matrix components may be uniformly distributed, they have different properties. In this study, fibres and matrix were not modeled separately. Instead, resultant stiffness properties of the FRC material were applied. Therefore, the calculated bending behavior of the FRC post can be expected to be close to reality. Stresses, however, may be locally elevated due to interfacial concentrations. Calculated maximum stresses should be viewed as homogenized values of the whole post responding as one material, and should not be associated directly with the ultimate fibre or matrix strength.

The results of this study support flexural strength testing of fibreglass posts as an appropriate procedure for quality control. However, clinically, post strength is probably not a major concern because they hardly fracture. The mechanical significance of clinical application of fibreglass posts is their reduced stiffness in comparison with metallic post. The resultant elastic modulus of fibreglass posts is closer to dentine, which has been shown to provide more beneficial stress distributions in the tooth structure (Santos-Filho *et al.* 2008, Soares *et al.* 2008). It can be argued that measuring flexure to determine elastic modulus of the post is clinically more important than flexural strength. Unfortunately, this study suggests that taper is a larger obstacle in flexure measurements than it is for maximum stress determination.

Conclusion

It can be concluded that three-point bending tests can be used to determine flexural strength of fibre posts, even when they involve some taper.

Tilting the specimens to level the posts is not necessary. Errors due to taper and amount of nonlinear flexure were relatively small, and can be reduced by limiting the taper in the tested section. Flexural strength values determined using expression [1] can be considered a property of a fibreglass post, assuming fibres and matrix respond as one homogeneous material. If taper is less than 50% of the span, the largest diameter value can be applied in the maximum stress calculation. Differences in post diameter between manufacturers should not be a limitation, because the strength property should be independent of post diameter if errors due to taper and flexure are minimized. Standardizing exact post or support dimensions is therefore less useful than standardizing the relationship between post diameter, support span, diameter of supports, and the maximum allowable flexure. To further improve accuracy, error corrections for taper can be developed by FEA.

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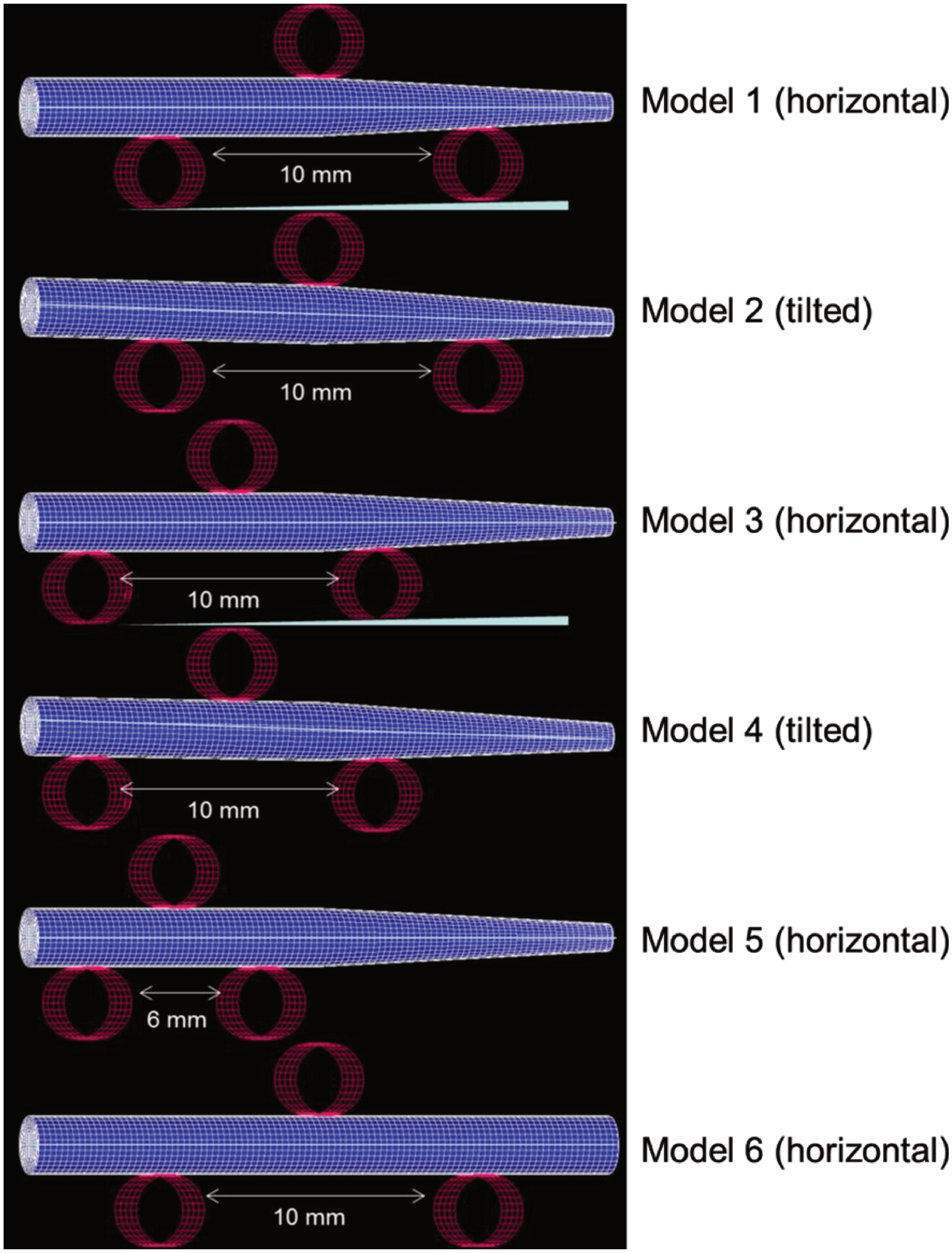

Figure 1 Schematic illustration of 3D-models of the simulated three-point bending tests, showing the different placements, support distances and tilts.

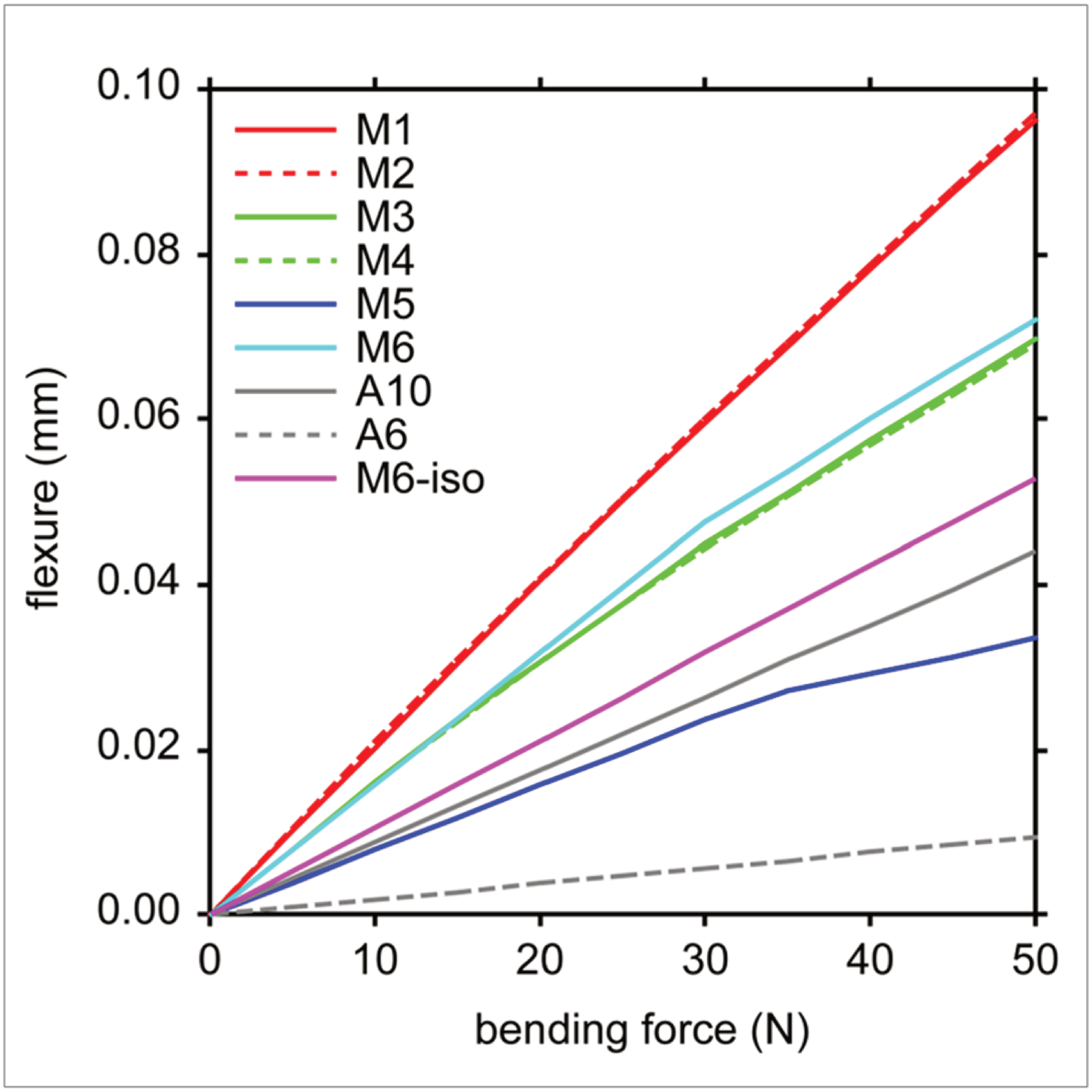

Figure 2 Force-displacement curves for the 6 bending models. The flexure was determined at the load application point. Also shown are the analytical curves for a cylindrical beam calculated for 6 and 10 mm support span distances (diameter 1.9 mm).

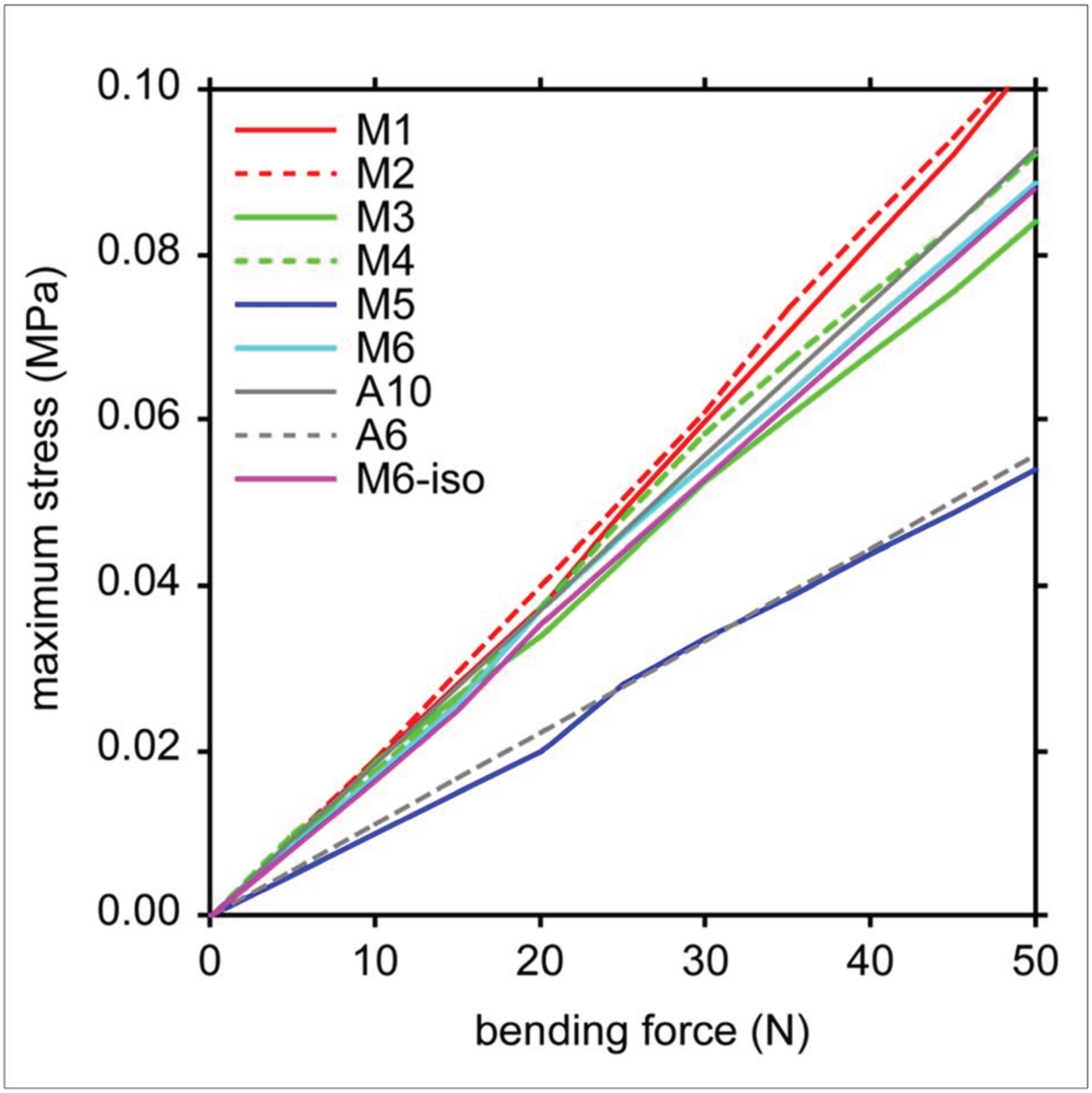

Figure 3 Force-stress curves for the 6 bending models. The stress was the maximum flexural stress found at the bottom of the beams. Also shown are the analytical curves for a cylindrical beam calculated for 6 and 10 mm support span distances (diameter 1.9 mm).

CAPÍTULO 2*

Correlation between flexural properties and structural characteristics of different fiber posts systems

Abstract

Objectives. Evaluate the flexural strength and flexural modulus of different fiber-reinforcement composite (FRC) posts and the correlation between the properties and structural characteristics.

Methods. Eleven brands of endodontic posts were analyzed (n=10): Exacto Cônico (Angelus), DT Light SL (VDW), Relyx Fiber Post (3M-Espe), Glassix Radiopaque (Nordim), Para Post Fiber White (Coltène), FRC Postec Plus (Ivoclar), Aestheti-Plus Post (Bisco), Superpost Cônico Estriado (Superdont), Superpost Ultrafine (Superdont), Reforpost (Angelus), and White Post DC (FGM). Posts were loaded in three-point bending test using a mechanical testing machine (EMIC 2000 DL) at a crosshead speed of 0.5 mm/min. Mean values were submitted to one-way ANOVA and Scott-Knot test ($p<0.05$). The cross-sections of the posts were examined by scanning electron microscopy (SEM) and an image processing and analysis program. The correlation between mechanical properties values and each one of structural variables was measured by calculating Pearson's correlation coefficients ($p<0.05$).

*** Trabalho a ser submetido para o periódico *Dental Materials***

Results. Structural characteristics significantly affect the properties of FRC posts. Flexural strength values ranged from 493 to 835MPa and is directly correlated with fiber/matrix ratio ($r = 0.241$; $p = 0.011$). Flexural modulus ranged from 4500 to 8824MPa and is inversely correlated with number of fibers per mm^2 of post ($r = 0.333$; $p = 0.000$).

Significance. The mechanical properties of FRC posts presented a large variation. A correlation between flexural strength and fiber/matrix ratio, as well flexural modulus and quantity of fiber was found.

Keywords: fiber post; flexural properties; fibers volume; structural characteristics; scanning electron microscopy; dental materials.

Introduction

Fiber-reinforced composite (FRC) posts exhibit biomechanical properties more similar to those of dentin than the metallic posts creating a homogenous restorative system consisting of the post, resin cement, core material along with the tooth substance [1,2,3,4]. FRC posts contain a high percentage of continuous reinforcing fibers embedded in a polymer matrix. Since fiber posts are in essence composite materials, it seems logical to expect that their mechanical properties would increase as a result of an increase in fiber content [5,6].

The correlation between mechanical properties and structural characteristics of posts could be detected for aspects concerning the composition of these posts, such as integrity, size, density, and distribution of the fibers and the nature of the bond between the matrix and the fibers, which may be the determining factors for different flexure strength values [6,7]. A possible explanation about a lower flexural strength for a post could be a weak interfacial bonding among fiber and matrix caused by irregularities produced during the manufacturing process [6]. Another thing is that areas of weakness in a FRC post can be seem in the voids present within the resin or in the discontinuities along the interface between fibers and matrix [5].

It is still not clear how the structural properties of FRC posts influence their mechanical properties [6]. Therefore, the present study was conduct to assess the mechanical properties of different FRC posts and evaluated the possible correlation between theirs properties and structural characteristics of them using scanning electric microscopy. The tested hypothesis was that structural characteristics of the fiber-reinforced composite posts would significantly affect the mechanical properties of them.

Materials and methods

In this study, eleven different endodontic posts were evaluated (n=10). The materials used are described in Table 1.

Three-point bending test

A three-point bending test was used to measure the mechanical properties of all specimens in a mechanical testing machine (EMIC DL 2000; EMIC, Sao Jose dos Pinhais, Parana, Brazil) using a 500N load cell and the 0.5 mm/min crosshead speed. It consists in positioning the sample on two points which define the span length of the test, and applying the load on a third point midway in the span [8]. The two supports and the central loading rod had a 2.0 mm cross-sectional diameter. In order to reduce the influence of the conical end of the posts, the span distance was 6.0 mm to ensure testing of only on the parallel portion of the post. The diameter of each sample was measured at point that load was applied using a digital micrometer (Mitutoyo, Japan). Flexural strength (σ) and flexural modulus (E) were calculated using the followings equations:

$$\sigma = 8F_{Max} L/\pi d^3 \qquad \text{(in MPa)}$$

$$E = 4F_{Max}L^3/(D3\pi d^4) \text{ (in MPa)}$$

where F_{Max} is the applied load (in Newtons) at the highest point of the load–deflection curve, L is the is the span length (6.0 mm), d is the diameter of the posts (in mm), D is the deflection (in mm) corresponding to load F at a point in the straight-line portion of the curve [8].

Scanning electron microscopy - SEM evaluation

All of posts tested were cross-sectioned at the point that load was applied using a diamond saw (Isomet, Buehler, Lake Bluff, NY, USA). The sectioned surface was embedded in polimethylmethacrylate resin and polished. The polishing was first carried out with silicon carbide paper following the sequence 600, 800, 1200 and 1500 grit size under streaming water, and finishing with felt discs impregnated with diamond paste. Following, the specimens were ultrasonically cleaned in deionized water for 10 minutes. The specimens were mounted on metallic stubs, and sputtered with gold in an ion-sputtering device (Bal-Tec SCD 050, Balzers, Germany). Then the specimens were analyzed under a SEM (LEO 435 VP, Carl Zeiss, Germany) at the same magnification (X1000).

Image analysis

SEM posts micrographs were analyzed using an image processing and analysis program (Image Tool 3.0). The number of fibers and the area occupied by fibers per square millimeter of post area were measured.

Statistical analysis

Data analysis was performed (SPSS 11 for Macintosh; SPSS Inc, Chicago, Ill) using one-way ANOVA followed by the multiple comparisons Scott-Knot test at a significance level of α=0.05. To verify the correlation between the mechanical properties (i.e. flexural strength and flexural modulus) and the structural characteristics (i.e. number of fibers per mm^2 and fiber/matrix ratio (%)) for all post, it was used the Pearson's correlation coefficients test at a significance level of α=0.05 using the software for statistical analysis (SPSS 11 for Macintosh; SPSS Inc, Chicago).

Results

Data expressing the correlation between mechanical properties and structural characteristics are summarized in Table 2. Means and standard deviations (SD) of flexural strength and flexural modulus for the experimental groups are displayed in Table 3 and 4, respectively. The fiber/matrix ratio (%) presented a linear and significant correlation with the flexural strength. Multiple comparison tests showed the highest flexural strength obtained with Exacto Cônico and the lowest to Superpost Cônico Estriado (Table 3).

There is a correlation between flexural modulus and number of fibers per total area of post, but inversely proportional. White Post DC showed the highest modulus, whereas Para Post Fiber White, Relyx Fiber Post, Superpost Ultrafine and Reforpost were statistically equivalent with the highest flexibility.

Additionally, the flexural modulus increased with decreasing diameter of the FRC posts systems evaluated (Table 4).

The SEM micrographs of cross-sections posts revealed differences between the FRC posts studied (Fig. 1). The fiber/matrix ratio in the posts ranged from 33% to 68%. Glassix Radiopaque, FRC Postec Plus, Superpost Cônico Estriado and Reforpost exhibited bubbles within the post structure. The Relyx Fiber Post, Para Post Fiber White, Superpost Ultrafine and White Post DC presented no structural defects and no gaps between the fibers and the resin matrix in the peripheral area (Fig.1).

Discussion

The results of this study support the research hypothesis, in that the structural characteristics significantly affect the flexural properties of fiber posts. The flexural strength is linearly correlated with fiber/matrix ratio ($r = 0.241$; $p = 0.011$), whereas the flexural modulus is inversely correlated with number of fibers per mm^2 of post ($r = 0.333$; $p = 0.000$).

In the fabrication of endodontic posts, glass, quartz, carbon, and ceramic fibers have been used [9]. This study evaluated nine different glass fiber posts and two quartz fiber posts, and the 3-point bending test demonstrated that glass fiber posts possessed similar values for flexural strength and flexural modulus compared with quartz fiber posts. Commonly, glass fiber posts contain e-glass fibers (electric glass) that consist of SiO_2, CaO, B_2O, Al_2O and a few other oxides of alkali metals in its amorphous phase [6]. Quartz fiber reinforcement used in DT Light SL and Aestheti-Plus Post is made out of pure silica. Its elastic modulus does not differ much from the other types of glasses but its low coefficient of thermal expansion can be benefic to the structural integrity during thermal alteration [5].

The addition of fibers to a polymer matrix leads to a significant increase in physical properties of polymer-based materials. The posts that exhibit a higher fiber/matrix ratio would be expected to yield a greater fracture resistance [4,5,6]. For this, in analysis of this study the fiber/matrix ratio presented linear correlation to flexural strength. The higher fiber content (68%) recorded for Exacto Cônico could explain the higher mean flexural strength value. On the order hand, the lowest value was obtained by Superpost Cônico Estriado (56%). However, Relyx Fiber Post (54%) and Para Post Fiber White (33%) showed fiber/matrix ratio that not accompanied the linearly of means values among other groups tested (Table 3), and this variability could be explain by the individual fiber properties contained in each post type. These results show that the fiber density contributes only partly to mechanical performance.

The present study showed an inversely proportional correlation between flexural modulus and number of fibers per total area of post (Table 4). White Post DC had the higher flexural modulus value in the same time that presented lower quantity of fibers (2924). The corresponding correlation was similar for the Reforpost, with lower flexural modulus and high number of fibers (8024). If the interfacial bond between the fibers and the matrix is inadequate, poor mechanical properties can be expected [10]. An increase in the total interface area (fiber and matrix) will result in better mechanical interlocking and increased stiffness [6]. It could explain the improvement in flexural modulus for White Post DC, which had lower chance of defects due lower contact area between fiber and matrix. By the varying of Aesthetic-Plus Post, that presented the most number of fiber (17452) but intermediary flexural modulus value compared with other groups, once more showed that the individual properties of fiber and matrix can be substantial.

Since three-point bending test is determined by the span distance, post design and diameter of post, differences in diameter have been claimed to affect flexural response [10]. Recommendation for engineering composites, especially for anisotropic FRC is that a high L/d ratio (span distance/ diameter of post) in order of

40:1 or 60:1, should be used in order to eliminate shear effect during bending test [11,12,13]. A lower L/d ratio produces more shear deformation in the FRC specimen [14]. Engineering material standard of ASTM D 2344 for short beam test uses L/d ratio of 4 to determine the interlaminar shear strength of a material. Then, the L/d ratio used in this study was approximately 4.2, considering 6.0mm of span distance and mean diameter values of FRC posts of 1.43mm.

Beyond L/d ratio, the higher order diameter term in expression to calculate flexural modulus ($4F_{Max}L^3/D3\pi d^4$) compared to expression to calculate flexural strength ($8F_{Max}\ L/\pi d^3$) indicates a higher sensitivity to changes in diameter for flexural modulus. White Post DC had the lower diameter; nevertheless it presented the higher flexural modulus (Table 4).

The FRC posts design may also vary in surface characteristics, such as smooth or serrated forms. Serrated posts present higher retention values and lower values of rigidity than smooth posts [15]. Thus, the addition of a serrated process on the surface post for the purpose of retention, which can be seeing in Para Post Fiber White, Superpost Cônico Estriado and Reforpost could suggest to decrease the values of post's flexural strength due of the discontinuous fibers [5].

Variables of fiber and matrix of FRC posts (i.e. volume fraction of fibers, orientation and thickness, bonding to resin matrix, polymerization-induced stress, manufactured process, global integrity of the posts and intrinsic properties of fibers and matrix) are especially important for understand the flexural strength and flexural modulus of FRC posts [5,16].

The first concern is that failures commences from a small structural defect such as a void or microcrack within the material. Therefore, potential areas of weakness in a fiber-reinforced post can be seen in the voids present within the resin or in the discontinuities along the interfaces between fibers and matrix [5]. It was verified in the Glassix Radiopaque, FRC Postec Plus, Superpost Cônico Estriado and Reforpost, showing clearly visible empty spaces and bubbles (Fig. 1, D, F, H and J, respectively). Oppositely, voids and bubbles could not be visualized for Exacto Cônico, Relyx Fiber Post and Aesthetic-Plus Post (Fig.1, A, C and G, respectively), and it is probably the reason for the highest flexural strength values of those posts.

Reforpost presented the fewer flexural strength, and a possible explanation for this could be a weak interfacial bonding caused by irregularities produced during the manufacturing process. Such discontinuities along the interfaces between the matrix and the fibers are proofs that interfacial bond strength is critical [8]. The matrix has a higher thermal expansion coefficient than that of fibers either glass or quartz and this can lead to the generation of stresses, which can cause adhesive failure between the fibers and the matrix [4]. Also, due to the different elastic modulus between glass/silica fibers and resin matrix, stresses normally develop at the interface between the fibers and the matrix, and propagate along the surfaces of the fibers when the posts are loaded [6].

A discontinuity along the interfaces between fibers and matrix was not observed to Relyx Fiber Post, Para Post Fiber White, Superpost Ultrafine and White Post DC (Fig.1, C, E, I and K, respectively). This may have been the effect of the combination between fabrication process used to promote chemical bonding between fiber and resin matrix with the silanization of fiber prior to embedding in the resin matrices. But much of this information is kept under industrial secret [5,17].

The fibers diameter in the Para Post Fiber White and White Post DC were found to be inhomogeneous compared to the other systems (Fig.1, E and K, respectively). The variation in diameter of fibers was remarkable, been possible to note presence of small diameter fibers located next to an area of large diameter fibers. The Aestheti-Plus Post consists of fibers with diameters well below the other systems (Fig.1G). However, flexural strength and flexural modulus have not been affected by differences in diameter of fibers present in FRC posts.

A limitation of this investigation is that no thermal cycling or artificial aging of posts systems was performed, and fiber-reinforce composite posts are directly affected by the influence of water and changes of temperature [4]. In addition, no association with tooth structure was made. This study was not a prediction of the FRC posts' behavior inside the oral cavity, but instead it is indicative of the properties of the materials themselves. Thus, future studies should be conducted to evaluate the fatigue factors to better understand the effectiveness and durability of FRC posts, as well evaluate the behavior of posts cemented on roots instead test just post, to verify if the structural defects presents on posts might cause problems to their long term service and could be limit their use in clinical situations.

Conclusion

Within the limitations of this study, the following conclusion can be drawn:

1. The structural characteristics significantly affect the mechanical properties of fiber posts. The flexural strength is directly correlated with fiber/matrix ratio ($r = 0.241$; $p = 0.011$), whereas the flexural modulus is inversely correlated with number of fibers per mm^2 of post ($r = 0.333$; $p = 0.000$);
2. Structural defects like bubbles and discontinuities along the interfaces between the matrix and the fibers influence flexural strength of FRC posts;
3. Flexural modulus and flexural strength are not affected by differences in diameter of fibers present in FRC posts.

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Table 1. Post systems evaluated in the study.

FRC Post	Manufacturer	Fiber	Lot No.
Exacto Cônico	Angelus; Londrina; Brazil	Glass	8214
DT Light SL	VDW Endodontic Synergy; Munich; Germany	Quartz	0706000475
Relyx Fiber Post	3M-Espe; Seefeld; Germany	Glass	045770701
Glassix Radiopaque	Nordin; Montreux Switzerland	Glass	07221
Para Post Fiber White	Coltène/ Whaledent; Mahwah; NJ; USA	Glass	MT-21793
FRC Postec Plus	Ivoclar Vivadent; Liechtenstein; France	Glass	L12279
Aestheti-Plus Post	Bisco; Schaumburg; IL; USA	Quartz	080011029
Superpost Cônico Estriado	Superdont; Rio de Janeiro; Brazil	Glass	-
Superpost Ultrafine	Superdont; Rio de Janeiro; Brazil	Glass	-
Reforpost	Angelus; Londrina; Brazil	Glass	12640
White Post DC	FGM; Joinville; Brasil	Glass	250209

Table 2. Statistical significance of the correlation between posts' mechanical properties and their structural characteristics ($p<0.05$).

Variable	Variable		
		Fiber/matrix ratio (%)	**Number of fibers per mm^2 of post**
Flexural strength	*Pearson´s Correlation Coefficient*	r = 0.241	r = 0.137
	Statistical significance	p = 0.011	p = 0.155
Flexural modulus	*Pearson´s Correlation Coefficient*	r = 0.157	r = 0.333
	Statistical significance	p = 0.102	p = 0.000

Table 3. Mean flexural strength (standard deviations) and fiber/matrix ratio of post systems tested.

FRC Post	Flexural Strength (MPa)	Fiber/matrix ratio (%)
Exacto Cônico	835.9 (46)[a]	68
White Post DC	822.2 (56)[a]	65
Aestheti-Plus Post	811.3 (42)[a]	65
Relyx Fiber Post	805.8 (33)[a]	54
Superpost Ultrafine	690.1 (24)[b]	62
DT Light SL	655.8 (131)[b]	60
FRC Postec Plus	632.7 (77)[b]	63
Para Post Fiber White	627.3 (53)[b]	33
Glassix Radiopaque	584.9 (70)[b]	61
Reforpost	569.5 (42)[c]	59
Superpost Cônico Estriado	493.5 (49)[c]	56

* Similar letters in columns indicate no significant differences between property values as determined by Scott-Knot test ($p < 0.05$).

Table 4. Mean flexural modulus (standard deviations), structural characteristics and diameter of post systems evaluated.

FRC Post	Flexural Modulus (MPa)	Number of fibers per total area of post	Mean post diameter (mm)
White Post DC	8824.3 (1011)[a]	2924	1.26
DT Light SL	5594.0 (528)[b]	5116	1.48
Glassix Radiopaque	5531.5 (778)[b]	4122	1.38
FRC Postec Plus	5479.2 (677)[b]	7691	1.43
Exacto Cônico	5476.2 (365)[b]	7951	1.45
Aestheti-Plus Post	5288.6 (681)[b]	17452	1.40
Superpost Cônico Estriado	5181.3 (530)[b]	7427	1.48
Para Post Fiber White	4957.5 (759)[c]	7547	1.41
Relyx Fiber Post	4924.2 (251)[c]	5326	1.56
Superpost Ultrafine	4830.8 (402)[c]	6645	1.49
Reforpost	4500.5 (515)[c]	8024	1.43

* Similar letters in columns indicate no significant differences between property values as determined by Scott-Knot test ($p < 0.05$).

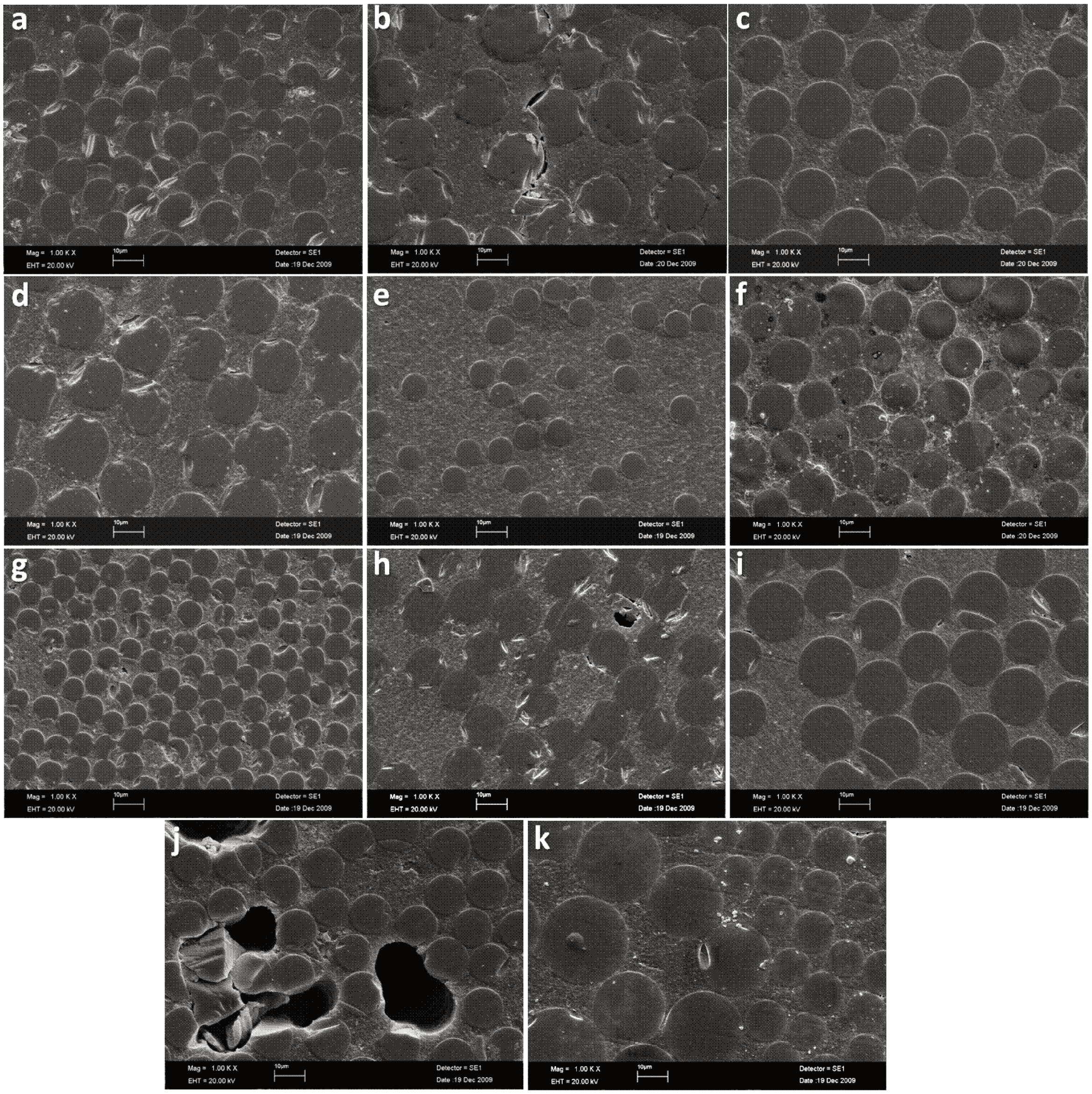

Figure 1. SEM images of posts systems evaluated at X1000 magnification: Exacto Cônico (A), DT Light SL (B), Relyx Fiber Post (C), Glassix Radiopaque (D), Para Post Fiber White (E), FRC Postec Plus (F), Aestheti-Plus Post (G), Superpost Cônico Estriado (H), Superpost Ultrafine (I), Reforpost (J), and White Post DC (K).

CAPÍTULO 3*

Bond strength between fiber posts and composite resin core - influence of temperature on the silane coupling agents

Clinical Relevance: The two-component silane system may impair the bond strength between fiber post and composite resin. The application of warm air after post silanization had no significant effect on push-out bond strength between glass fiber post and composite resin core.

Summary

This study evaluated the effect of air-drying temperature and different silane coupling agents on the bond strength between glass fiber posts and composite resin core. The post surface was cleaned with alcohol and treated with different silane coupling agents, three prehydrolyzed silanes [Silano (Angelus), Prosil (FGM), RelyX Ceramic Primer (3M ESPE)] and one two-component [Silane Coupling Agent (Dentsply)]. Two different post-silanization air-drying temperatures, 23°C and 60°C, were applied. A cylindrical plastic matrix was placed around the silanized post and filled with resin composite. Each bonded post provided seven slices for push-out testing. Each slice was loaded to failure under compression at a cross-head speed of 0.5mm/min. Data were analyzed by two-way ANOVA and Scott-Knott tests (α = 0.05). To compare mean of control group to each of the experimental groups, Dunnett's test was used. Scanning electron microscopy (SEM) was used to evaluate the interface of the fractured slices. For the 23°C air-drying temperature, the RelyX Ceramic Primer resulted in significantly lower bond strength than the other silane coupling agents, while Silane Coupling Agent

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(Dentsply) was higher than the other groups. Only for the Silane Coupling Agent, the bond strength for the 23°C air-drying temperature was significantly higher than for 60°C air-drying.

Introduction

Endodontically treated teeth with excessive wear result in a lack of coronal tooth structure and frequently need post to retain the coronal restorative portion.[1,2] The presence of significant coronal tooth structure loss requires abutment build-up around a fiber-reinforced composite (FRC) post.[1] Posts form a bonded unit between root and coronal dentine, adhesive systems, resin cements, and composite build-up.[3] The most common failure of endodontic-treated teeth restored with FRC post during fracture testing is failure involving core portion.[4] The retention and stability of the posts systems and core build-up is an important factor for successful restoration.[5] Retention of the core portion around the post is promoted by both micromechanical and chemical interaction between post and resin composites.[1]

Silane coupling agents have been widely used in dentistry since the introduction of glass fiber-reinforced resin-based materials. A chemical bond may be achieved between the core resin matrix and the exposed glass fibers of the post.[6] The results of some studies advocate for silane capability of increasing surface wettability resulting in chemical bridges formation with OH-covered substrates (e.g., glass or quartz fibers).[7] Additionally, it is related that silane coupling agent significantly improved the bond strength between FRC posts and composite core build-up materials.[6]

However, silane coupling is considered a technique-sensitive step. The bond strength of composite to FRC posts can be affected by different composition of silane coupling agents and the air-drying temperature.[8] Solvent evaporation plays an important role; a small amount of solvent may be beneficial in promoting silane wetting, but an incomplete removal may compromise coupling.[9] A stream of warm air may be used to assist evaporation of solvents and reaction products on the silane-treated surface, which results in a dried surface.[10] Significant improvement in shear bond strength after heat treatment of the silanized porcelain can be attributed to the elimination of water, alcohol, or acetic acid from the silane-treated surface.[11] Ethanol/water-based silanes have a more stable behavior, probably due to the mixing ratio of the two solvent components. At temperatures of 21°C and 38°C, no significant differences were present in the evaporation rate of water. It is likely that higher values of temperature may facilitate water removal and could increase bond strength.[8] Scarce information exists about the influence of post-silanization air-drying on bond strength between fiber post and core resin regarding different silanes couple agents.

Therefore, the aim of this study was to evaluate the push-out bond strength between the glass fiber post and composite resin core influenced by the temperature on different silane coupling agents. The hypothesis generated is that drying with a stream of warm air the post surface after silanization could increase the push-out bond strength of composite resin core to glass fiber post over that produced by a room temperature air-drying procedure.

Methods & Materials

Ninety glass fiber posts Exacto (Angelus, Londrina, Parana, Brazil) size 2 with a length of 17.0 mm and a diameter of 1.50 mm, composed of unidirectional glass fibers (87% volume) embedded in an epoxy resin matrix were used for testing. All the posts were cleaned with alcohol, dried with an air stream, and randomly divided into 9 groups (n = 10). The 9 groups included 1 control group and 8 experimental groups according to the 2 study factors: 4 silane coupling agent types (Table 1) and 2 air-drying temperature of silane application (23^{o}C) that represents room temperature and with warm air-drying at 60^{o}C: Group 1: control group, no surface treatment; Group 2: Silano, air-drying at room temperature 23^{o}C; Group 3: Silano with warm air-drying at 60°C; Group 4: Prosil, air-drying at room temperature 23^{o}C; Group 5: Prosil with warm air-drying at 60°C; Group 6: RelyX Ceramic Primer air-drying at room temperature 23^{o}C; Group 7: Relyx Ceramic Primer with warm air-drying at 60°C; Group 8: Silane Coupling Agent air-drying at room temperature 23^{o}C; Group 9: Silane Coupling Agent with warm air-drying at 60°C. The tested materials were applied following manufacturers' recommendations. The composition and application mode of the tested materials are described in Table 1.

The air-dying at room temperature was performed with gently air of air syringe at 23±1°C. The room temperature was controlled by thermometer fixed closed to the workbench. To perform the air-drying with warm air at 60^{o}C, a miniaturized hairdryer (Sokany, Model 1808A, 50-60 Hz, 1200W) was used. To standardize and control the temperature, a copper-constantan thermocouple (Type T) was confectioned that is widely used for low-temperature measurements in laboratory. This Type T thermocouple wire was connected to the data acquisition device (ADS0500IP; Lynx Tecnologia Eletronica Ltda, São Paulo, Brazil). At the moment that the hairdryer was turned on, the temperature was transferred to a computer that used specific acquisition, signal transformation, and data analysis software (AqDados 7.02 and AqAnalisys; Lynx Tecnologia Eletronica Ltda).

After silanizing the post surface, composite build-up was performed using a special device to centralize the posts.[12] Each post was positioned upright in the center of the device (Figure 1A). Then, a cylindrical plastic matrix was placed around the post and adjusted so that the post would be exactly in the middle. The matrix was 10 mm in length and 5 mm in diameter. The matrix was filled incrementally with a hybrid composite resin (Filtek Z250; 3M ESPE, St. Paul, MN, USA). Photo-activation of each increment of 2 mm was performed at the top of the tube using a LED at 1200 mW/cm^2 (Radii-cal LED, SDI, São Paulo, SP, Brazil).

The matrix ensured standardized shapes of the composite build-ups and equal distribution of the core material around the posts. The specimens were removed from the matrix and stored in distilled water at 37°C for 24 hours.

Push-out testing

Each specimen was cut perpendicular to the long axis into 7 slices of 1.00 ± 0.05 mm (Figure1B) with a slow-speed diamond saw under water cooling (IsoMet 1000; Buehler Ltd, Lake Bluff, Ill, USA). The thickness of each slice was measured using a digital caliper to confirm accuracy, and the value was recorded. Seven slices were obtained from each post-and-core specimen, therefore providing a total of 70 slices per group. To calculate the exact bonding surface, the tapered design of the posts with regard to the respective part of the post was considered. Each specimen was measured with a measuring microscope (TM-505- Toolmaker's Microscopes, Mitutoyo). This step analyzed the integrity of the slices and eliminated specimens with failures like bubbles or cracks. The bonding surface was calculated considering that π is the constant 3.14; R1 is the larger radius; R2, the lower radius; and h, the thickness, and using the formula of a conical frustum:[13]

$$\text{Area} = \pi(R_1 + R_2)\sqrt{}R_1 - R_2)^2 + h^2$$

A compressive push-out load was applied using a custom-built device that consisted of a 3 cm stainless steel base with a 2 mm center hole. The slice is centered over this hole before applying a compressive load at 0.5 mm/min with a 1-mm-wide stainless steel rod in a universal testing machine (EMIC DL2000; EMIC, Sao Jose dos Pinhais, Parana, Brazil) until failure. The force required to cause failure (N) was recorded by a 200 N load cell hardwired to software (TESC; EMIC). The push-out bond strength was expressed in Megapascals (MPa) by dividing the load (Newton) at failure by the bonded area of the post segment (mm^2).[14,15]

After analyzing the bond strength data for the normality of data distribution (Kolmogorov–Smirnov test) and homogeneity in variances (Levene's test), data were analyzed by two-way ANOVA to evaluate the effect of the study factors (silane and air-drying temperature). Multiple comparisons were performed with Scott-Knott test ($\alpha = 0.05$). Dunnett's test compares the mean of each experimental group with that of control group.

Specimens tested were mounted on aluminum stubs and coated with gold using a sputter coater (Bal-Tec SCD 050, Balzers, Germany) and examined under a scanning electron microscope (SEM) (LEO 435 VP, Carl Zeiss, Germany). SEM images were obtained at different magnifications to illustrate the failures.

Results

Push-out bond strength means values and standard deviations for 8 experimental groups are reported in Table 2. The Scott-Knott test indicated that for the air-drying at room temperature (23°C), the RelyX Ceramic Primer resulted in significantly lower bond strength than the other silane coupling agents. The warm air-drying at 60°C for Silane Coupling Agent (Dentsply) decreased significantly the bond strength when compared to that produced by a room temperature air-drying procedure. No significant difference was found between the temperatures for other silane tested in this study. Dunnett's test showed that Silane Coupling Agent (Dentsply) associated with air-drying at room temperature (23°C) resulted in significantly higher bond strength than control group. All the other experimental groups presented statistical similarity to control group (Table 3).

SEM and optical observations showed that mixed failure involving partial adhesive failure between post and composite resin core was the exclusive mode of failure in all specimens (Figures 2 and 3). Figure 2 shows the extruding of post in relation to composite build-up, representing a partial adhesive failure. Figure 3 reveals that the post surface was partially covered with core material after testing.

Discussion

The use of warm air-drying after silanization of fiber post did not improve the bond strength values to composite resin core. The warm air-drying at 60°C applied to Silane Coupling Agent (Dentsply) decreased significantly the bond strength when compared to that produced by a room temperature air-drying procedure. Thus, the hypothesis is rejected.

Fiber posts systems are usually selected for rehabilitation of endodontically treated teeth to retain the coronal portion and did not reinforce the tooth-restoration complex.[16] The combination of the fiber post with an adhesive restoration is capable of creating homogenous stress distribution and decreasing catastrophic root fracture, with the possibility of replacing the restoration. However, resin core or post fractures are the failures that affect this type clinical procedure,[4] justifying the evaluation of factors related to bond between FRC post and composite resin core located at coronal portion of teeth.

The push-out test is a method that has been considered more dependable than the microtensile test for testing interfacial bond strength for bonded posts.[14,16,17] The push-out test involves the use of an indentor to push a small diameter fiber into a composite specimen with a thickness of several millimeters. In this case, the test specimen is sufficiently thin (approximately 1mm) to enable the entire length of fiber to slide out of the matrix.[18] So, an additional advantage of using the “thin slice” push-out test is that multiple slices may be retrieved from a single bonded fiber post/composite core sample.[1]

The push-out test demonstrated a more homogenous stress distribution by FEA and less variability in mechanical testing when compared with microtensile bond test.[15] This experimental method presents features of testing conditions that must be considered, such as tensile stresses produced by the bending moment at load application.[19] Study using finite element analysis demonstrated that the tensile forces during loading could be minimized by optimizing specimen dimensions and load application location.[20] Therefore, this study used devices that are compatible with the diameter of the FRC post, where the load applicator had a diameter of 1.2 mm, the push-out device had a role with 2.0 mm in diameter, and the post had a diameter of 1.5. Then the stress is concentrated at the interface homogeneously, which is a characteristic essential for a bonding test.

Silanes can be presented as single-phase pre-activated solutions (prehydrolyzed) or two-component systems that must be mixed just before application in order to initiate the hydrolysis reaction.[21] Silanes have the ability to bond inorganic materials such as glass, mineral fillers, metals, and metallic oxides to organic resins. The adhesion mechanism is due to two groups in the silane structure. The $Si(OR)_3$ portion reacts with the inorganic reinforcement, while the organofunctional (vinyl-, amino-, epoxy-, methoxy-) group reacts with the resin. When used as a coupling agent, the silane is defined as chemical substances capable of reacting with both the reinforcement and the resin matrix of a composite material. It may also bond inorganic fillers or fibers to organic resins, which promotes a stronger interface bonding. Then, the agent acts as interface between the resin and glass fiber to form a chemical bridge between the resin and fiber.[22]

The present study showed that RelyX Ceramic Primer presented significantly lower bond strength values when air-drying at room temperature was applied. On the other hand, when warm air-drying at 60°C was used to evaporate the solvents, the bond strength values was similar to the other groups. The solvents of this material are ethyl alcohol and water, which is more difficult to be evaporated, and it probably increased the bond strength.[11] Higher temperatures of warm air stream may facilitate water removal and increase bond strength.[8]

The two-component Silane Coupling Agent (Dentsply) has been proven to be more sensitive to heating.[23] A rise in air-drying temperature may improve solvent evaporation, but its extent is related to the volatile nature of the specific solvent.[24] Ethanol, the solvent that is present in the two-bottle silane systems evaluated in this study, evaporates easily. It probably explains the results achieved in this group, in which the values decreased after application of heating. On the other hand, the presence of acetic acid in the 3-MPS silane solution may guarantee pH stability and their effectiveness, justifying the higher values without heating.[25]

The bond strength values of control group, which no treatment was realized, presented no significant difference to the other groups, except for Silane Coupling Agent (Dentsply) with air-drying at 23°C. These results are supported for other studies that showed no chemical bond between the composite resin core materials and the FRC posts, indicating no benefit from the preliminary application of silane coupling agents.[3] Bond strength between the composite build-up and the FRC posts consists mainly of a combination of micromechanical interlocking and sliding friction. This can be evidenced by Figure 3; after debonding, post surface is partially covered with core material. However, it is important to consider that although there was no significant difference between almost all groups compared to control group, the numerical values of all groups were higher than the control group.

Current FRC posts are composed of unidirectional fibers embedded in a resin matrix in which reinforcing quartz or glass fibers are immersed.[17] The glass fiber posts used in this study are purported by the manufacturer to contain 87% glass fiber and 13% epoxy resin, which is highly cross-linked and less reactive.[26] Therefore, this posts system has little functional groups to react with the silane solution.[17] Additionally, there is no chemical union between the methacrylate-based resin composites and the epoxy resin matrix of fiber posts.[27] This result seems to justify the similarity of values obtained among the control group with other groups in which silane was used.

Since MPS silane does not bond well with the epoxy matrix, it might be possible that the silane effect of enhancing the post-core bond strength is increased when FRC posts with more superficial parts of fibers are used.[3] When the surface of the post is conditioned with different protocols,[27] more reactive surface is generated because the superficial layer of epoxy resin is removed; this creates chemical and micromechanical retention. More epoxy resin matrix can be exposed on both glass and quartz fiber post surfaces with the application of hydrogen peroxide.[26,28] Thus, conditioning of the surface of the FRC posts followed by silanization may enhance bond strengths to composite build-up.[28] Although the silane coupling agents are capable of forming bonds with both inorganic and organic structures, your application without a prior surface preparation seems to be insufficient to improve the bond strength.[22]

Conclusions

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

- The use of warm air-drying after silane application produced no increase in the bond strength between FRC post and composite core. Additionally warm air-drying after the two-component Silane Coupling Agent (Dentsply) resulted in significantly lower bond strength;
- The two-component Silane Coupling Agent (Dentsply) performed higher bond strength than all pre-hydrolyzed silanes when it was used air-drying at room temperature 23^{o}C.

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Table 1. Features of materials used in the study.

Material	**Composition**	**Solvent**	**Manufacturer / Bath**	**Application mode following manufacture instructions**
Silano	X-R-Si(OR)3n X-Organofunctional group; R-methylene group;OR-hydrolizable group; Si-Silicon	Ethanol	Angelus, Petropolis, RJ, Brazil/ 10975.T	Clean with alcohol and dry with air. Apply silane with a brush, wait for 1 minute, and gently dry with air.
Prosil	3methacryloxypropyltrimethoxysilane (MPS)	Ethanol, water	FGM, Joinville, SC, Brazil/ 2010.OCT	Clean with alcohol and dry with air. Apply silane with a brush, wait for 1 minute, and gently dry with air.
RelyX Ceramic Primer	3methacryloxypropyltrimethoxysilane (MPS)	Ethyl Alcohol, Water	3M ESPE, St. Paul, MN, USA/ 8YP	Clean post with alcohol, air dry, apply silane with a brush, and gently dry with air.

Silane Coupling Agent	Silane	Ethanol, Acetic acid	Dentsply, Petropolis, RJ, Brazil/ 133203B	Mix a drop of Silane Primer and a drop of Silane Activator for 10 to 15 seconds. Let stand for 5 minutes. Apply a thin layer on the surface of post and air-dry. Apply a second coat and air-dry.
Filtek™ Z250 Universal Restorative	Inorganic filler (Zirconium/silica) loading is 60% by volume with a particle size range of 0.01 to 3.5 microns. BIS-GMA, UDMA and BIS-EMA.	-	3M ESPE, St. Paul, MN, USA/ 8HE	The material is incrementally placed in layer less than 2.5mm and each layer is cured for 20 seconds.

Table 2. Mean values of push-out bond strengths in MPa and standard deviation (SD).

Temperature	Silane			
	Angelus	*Prosil*	*Relyx Ceramic Primer*	*Silane Coupling Agent*
23°C	20.29 (2.32) Aa	20.70 (2.33) Aa	18.52 (2.84) Ba	21.66 (2.49) Aa
60°C	19.70 (2.88) Aa	19.37 (1.04) Aa	19.91 (2.27) Aa	19.44 (1.32) Ab

* Means with same letter (uppercase in horizontal and lowercase in vertical) are not significantly different at $P > 0.05$.

Table 3. Means and standard deviation (SD) of push-out bond strength values (MPa) comparing negative control group with the others using Dunnett's test.

Group	Mean (SD)
G1, negative group	18.12 (0.82) b
G2, Silano Angelus (23°C)	20.29 (2.32) b
G3, Silano Angelus (60°C)	19.70 (2.88) b
G4, Prosil (23°C)	20.70 (2.33) b
G5, Prosil (60°C)	19.37 (1.04) b
G6, Relyx Ceramic Primer (23°C)	18.52 (2.84) b
G7, Relyx Ceramic Primer (60°C)	19.91 (2.27) b
G8, Silane Coupling Agent Dentsply (23°C)	21.66 (2.49) a
G9, Silane Coupling Agent Dentsply (60°C)	19.44 (1.32) b

* Different letters indicate statistically significant differences.

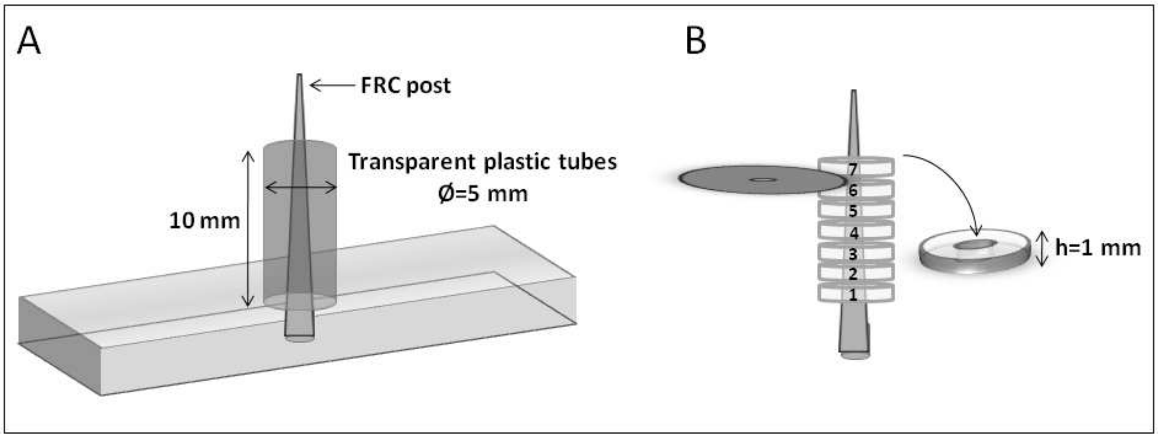

Figure 1. A, Schematic view of the specimen preparation procedure; B, Sectioning of post-and-core specimens for push-out test.

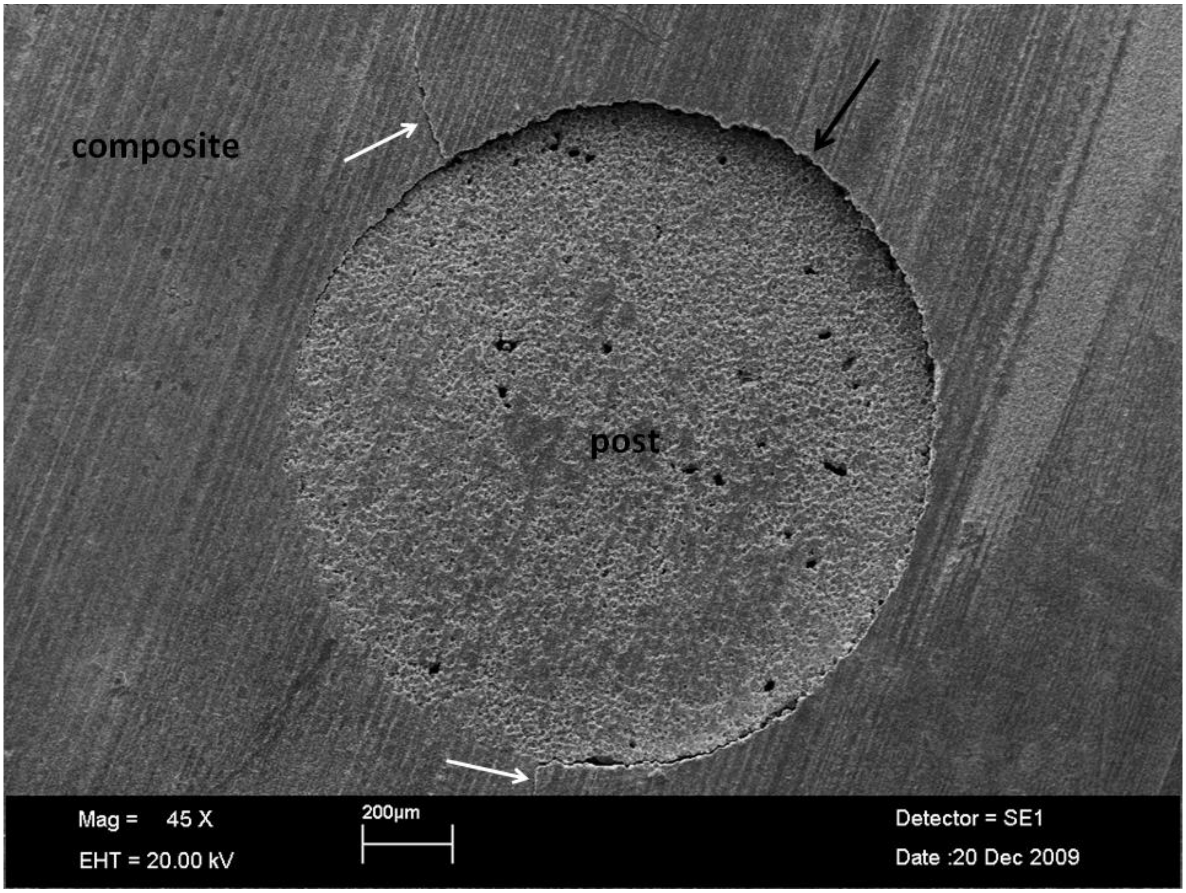

Figure 2. Illustrative SEM image of the slice after being tested. Black arrow in figure suggest the FRC post being extruded of the composite build-up that represents partial adhesive failure. White arrows suggest the cohesive fracture of the composite resin core (Original magnification ×45).

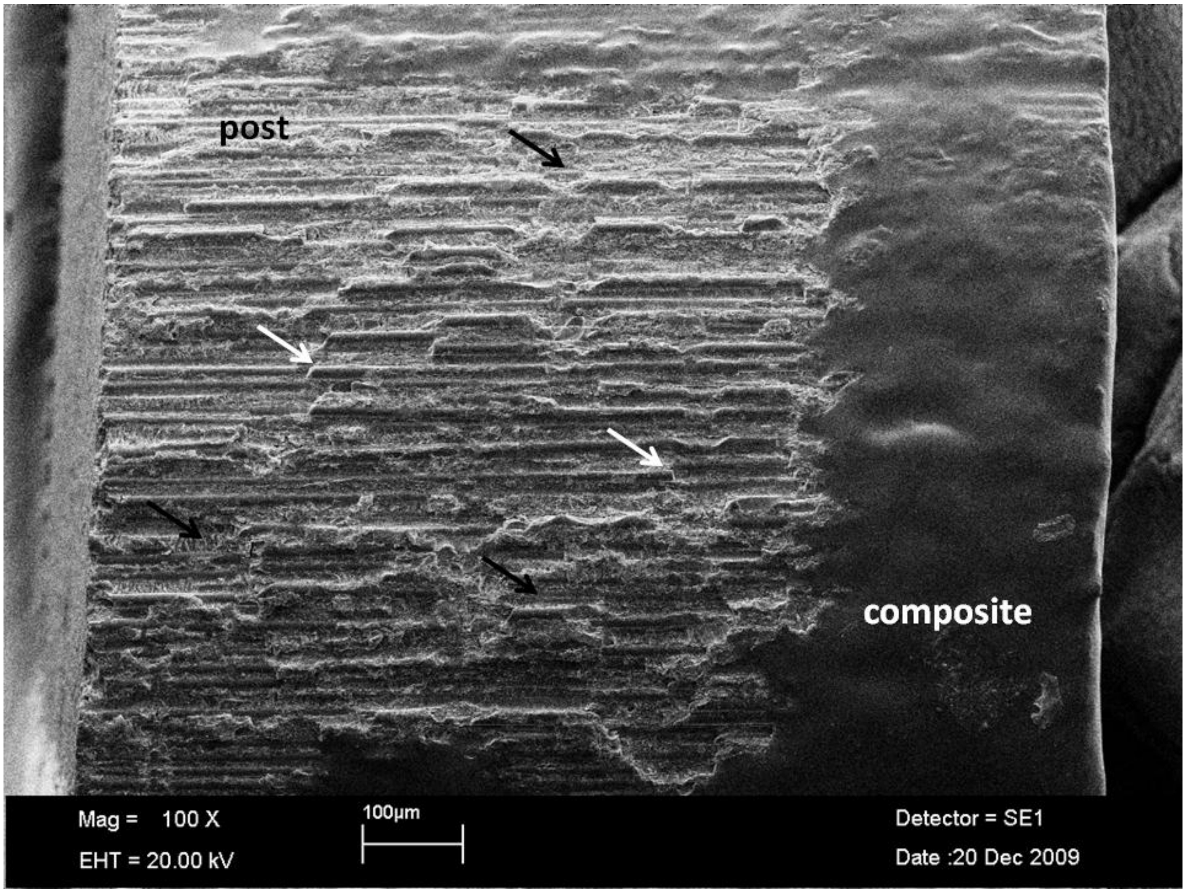

Figure 3. SEM image of a post surface after fracture, illustrating the mixed nature of the failure mode. Resin composite remnants are partially evident. Black arrow suggest the epoxy resin exposed on the most of post surface. White arrow suggest the amount of the glass fiber exposed (×100).

CONSIDERAÇÕES GERAIS

A avaliação das propriedades mecânicas de materiais restauradores é imprescindível para o conhecimento e adequada indicação e aplicabilidade clínica, ao mesmo tempo em que se faz necessária a avaliação das metodologias empregadas para a análise destes materiais dentários, com o intuito de melhorar a interpretação e comparação de resultados.

Uma vez que não existe norma específica para análise dos materiais reforçados com fibras, os pinos pré-fabricados são freqüentemente testados usando as mesmas configurações do teste de flexão de três pontos especificado pela ISO para os materiais de base polimérica. O objetivo no Capítulo 1 foi investigar e discutir as complicações e a validade do uso do ensaio de flexão de três pontos para testar pinos pré-fabricados reforçados com fibras, por meio do método de elementos finitos. Este é um método numérico para investigar sistematicamente as variáveis experimentais, possibilitando assim, sugerir alterações geométricas de corpos-de-prova e/ou dispositivos de ensaios experimentais.

A preocupação de usar pinos pré-fabricados em testes de flexão de três pontos é que muitos apresentam geometria com secção cônica, diferente dos espécimes preconizados pela Organização Internacional para Padronização (ISO). A principal dúvida é de como esses espécimes cônicos devem ser posicionados sobre os suportes e se eles devem ser nivelados. Os resultados deste estudo mostraram que o nivelamento dos pinos no momento do ensaio teve pouco efeito sobre a flexão e valores de tensão máxima. No entanto, a presente análise mostrou que o efeito da conicidade presente em pino pré-fabricado reforçado com fibra, no ensaio mecânico de flexão, influencia os resultados obtidos. Quando metade da extensão do pino envolvida na simulação do teste era cônica, a flexão aumentou substancialmente em relação ao modelo de pino cilíndrico, considerado como modelo de referência. No entanto, quando apenas 20% da conicidade do pino foi envolvida no momento do teste, o efeito sobre a flexão não foi substancial. Os valores de tensão máxima correspondente mostraram uma tendência similar, embora tenham sido menos pronunciados.

Outra preocupação é que as fórmulas matemáticas empregadas para o cálculo da flexão e tensão máxima padronizadas pela ISO para materiais poliméricos consideram o material como homogêneo e isotrópico. A análise de elementos finitos, ao contrário, calculou a flexão e tensões máximas nos pinos em relação a sua real geometria e propriedades (ortotropia). Comparando a teoria de flexão de engenharia e a análise por elementos finitos, foi possível perceber que ao considerar a propriedade de ortrotopia dos pinos, uma grande diferença pôde ser observada para os valores de flexão, enquanto os valores de tensão máxima foram relativamente próximos. Esta diferenciação não foi detectada quando se considerou o pino sendo isotrópico, constatando uma aproximação dos valores de flexão tanto no cálculo matemático da expressão de engenharia, como pela análise de elementos finitos.

Além da conicidade e das propriedades isotrópica e ortotrópica do pino, a distância entre os dois suportes inferiores no qual a amostra é posicionada no momento do teste, também afeta a flexão e tensão máxima. Neste estudo, duas distâncias entre os suportes (10 e 6 mm) foram modeladas. A distância de 6 milímetros foi escolhida para verificar se para esta situação, no qual o teste de flexão poderia ser realizado somente na secção cilíndrica do pino, se evitaria as complicações decorrentes da secção cônica. Comparação foi feita entre este modelo de pino cilíndrico-cônico, cuja distância entre os suportes era de 6 mm, sendo somente a porção cilíndrica do pino posicionada sobre os suportes no momento do teste, com um modelo que seria o modelo de referência (M6), simulando um pino totalmente cilíndrico, mas com distância de 10 mm entre os suportes. Os resultados encontrados entre os dois modelos foram consistentes.

Os resultados do Capítulo 1, portanto, suportam que o teste de resistência à flexão para pinos pré-fabricados reforçados com fibra, é um procedimento adequado para mensuração das propriedades destes materiais. Os valores de tensão máxima dos pinos são condizentes quando se utiliza metodologia semelhante aos padrões ISO para materiais poliméricos, desde que o envolvimento da porção cônica do pino entre os suportes no momento do teste seja minimizada. Metodologia padronizada pela ISO para determinação do módulo elástico, porém, não deve ser usada porque o cálculo de flexão na expressão matemática da engenharia não é preciso para secções cônicas e propriedades não isotrópicas.

O conhecimento das propriedades físicas dos pinos reforçados com fibras é muito importante para prever o seu comportamento e desempenho quando estão inseridos nos canais radiculares e sob carregamento na cavidade oral (Papadogiannis *et al.*, 2009). Considerando-se que o ensaio mecânico de flexão é aceitável para análise das propriedades mecânicas de pinos pré-fabricados, e que a distância entre os suportes de 6 mm empregada no momento do teste é satisfatória, o Capítulo 2 deste estudo avaliou a resistência à flexão e módulo de flexão de diferentes sistemas de pinos reforçados com fibra, por meio do ensaio de flexão preconizado no Capítulo 1, assim como correlacionou os valores obtidos no ensaio experimental com a análise de imagens feitas em microscópio eletrônico de varredura, por meio de programa de processamento de imagem.

Sendo assim, os resultados obtidos no Capítulo 2 mostraram que as características estruturais afetam significativamente as propriedades mecânicas de pinos de fibra. A resistência à flexão é linearmente relacionada com a relação fibra/matriz (%), enquanto o módulo de flexão é inversamente relacionado com o número de fibras por mm^2 do pino.

Espera-se que pinos que apresentam uma maior relação fibra/matriz possuam maior resistência à fratura (Grandini *et al.*, 2005; Seefeld *et al.*, 2007; Papadogiannis *et al.*, 2009), uma vez que a adição de fibras a matriz de polímero leva a um aumento significativo nas propriedades físicas dos materiais de base polimérica. O maior conteúdo de fibra (68%) registrado para o pino Exacto Cônico poderia explicar os maiores valores de resistência à flexão apresentados por este grupo. Valores altos de resistência à flexão são importantes para que o pino apresente resistência sem sofrer elevada deformação, pois esta deformação poderia levar à falha do procedimento restaurador. Esta falha provavelmente aconteceria devido às tensões que seriam geradas na interface de união entre pino/ cimento, ou entre pino/ material de preenchimento, uma vez que o material a qual ele está unido não deformaria de maneira semelhante do pino (Debnath et al., 2004; Lassila et al., 2004).

Por outro lado, o menor valor foi obtido pelo Superpost Cônico estriado, que apresentou conteúdo de fibra de 56%. Dois dos onze grupos avaliados: RelyX Fiber Post (54%) e Para Post Fiber White (33%), não apresentaram linearidade dos valores médios de resistência à flexão com a densidade de fibras, e esse fato provavelmente pode ser explicado pelas propriedades individuais das fibras contidas em cada tipo de pino. Portanto, a densidade de fibras contribui para o desempenho mecânico dos pinos, mas de maneira parcial (Cheleux & Sharrock, 2009).

O presente estudo ainda mostrou correlação inversamente proporcional entre o módulo de flexão e o número de fibras por área total de pino. O maior valor de módulo de flexão foi obtido por White Post DC, que ao mesmo tempo apresentou a menor quantidade de fibras. A correlação correspondente foi semelhante para o Reforpost, com menor módulo de flexão e elevado número de fibras. O módulo de flexão define a flexibilidade de uma amostra, no qual maiores valores indicam maior rigidez, enquanto que valores menores indicam uma maior flexibilidade (Plotino *et al.*, 2007). Desta forma, quando a ligação interfacial entre as fibras e a matriz do pino é inadequada, menores valores de rigidez do material podem ser esperados (Seefeld *et al.*, 2007).

Variáveis da fibra e matriz dos pinos reforçados com fibras, como volume de fibras, orientação e espessura, união à matriz de resina, tensões induzidas pela polimerização, processo de fabricação, integridade global dos pinos e propriedades intrínsecas das fibras e matriz, são importantes para compreensão de suas propriedades mecânicas (Grandini *et al.*, 2005; Grandini *et al.*, 2008). Possíveis áreas de enfraquecimento em pinos reforçado com fibras podem ser devido aos vazios presentes na resina ou nas descontinuidades ao longo das interfaces entre as fibras e a matriz (Grandini *et al.*, 2005), como verificado nos grupos Glassix Radiopaque, FRC Postec Plus, Superpost Cônico Estriado e Reforpost, sendo provavelmente a razão para os menores valores de resistência à flexão destes sistemas.

Após avaliação das propriedades de diferentes sistemas de pinos e a relação com suas características estruturais no Capítulo 2, o estudo dos fatores relacionados ao processo de união dos pinos na estrutura dentária se torna importante. A combinação do pino de fibra com uma restauração adesiva é capaz de criar distribuição homogênea das tensões e diminuir fraturas catastróficas da raiz, com possibilidade de substituição e/ou reparo da restauração. No entanto, as fraturas do núcleo de preenchimento em resina ou do pino são as falhas que mais afetam este tipo de procedimento clínico (Santos-Filho *et al.*, 2008), justificando a avaliação de fatores relacionados à união entre pino pré-fabricado reforçado com fibra e núcleo de preenchimento em resina composta no Capítulo 3.

Silanos têm a capacidade de unir materiais inorgânicos às resinas orgânicas (Goyal, 2006). Apresentam nas suas formulações solventes que devem ser evaporados a fim de não interferir no processo de polimerização dos agentes de cimentação (Barghi *et al.*, 2000). No entanto, este estudo mostrou que o emprego de ar quente após silanização de pino de fibra com intuito de evaporar os solventes não melhorou os valores de resistência de união ao núcleo de preenchimento em resina composta à superfície não condicionada de pinos de fibra de vidro.

O sistema de dois componentes Silane Coupling Agent (Dentsply) provou ser mais sensível ao calor (Hooshmand *et al.*, 2002). Um aumento na temperatura do ar para secagem pode melhorar a evaporação do solvente, mas este aumento está relacionado com a natureza volátil específica para cada solvente (Papacchini *et al.*, 2007). Etanol, o veículo que está presente nos sistemas de silano de dois componentes avaliado neste estudo, se evapora facilmente. Isso provavelmente explica os resultados obtidos nesse grupo, em que os valores diminuíram após a aplicação de calor (Olmos *et al.*, 2003).

Os valores de resistência de união do grupo controle, no qual nenhum tratamento foi realizado, não apresentou diferença significativa com os demais grupos, exceto para o silano (Dentsply) com secagem com ar a 23°C. Os atuais pinos pré-fabricados são constituídos por fibras unidirecionais embebidas em uma matriz de resina no qual o reforço de quartzo ou vidro são imersos (Perdigão *et al.*, 2006). A informação do fabricante é que o pino de fibra de vidro utilizado neste estudo contém 87% de fibra de vidro e 13% de resina epóxi, que é altamente densa e menos reativa (Yenisey & Kulunk, 2008). Portanto, este sistema de pinos possui poucos grupos funcionais para reagir com a solução de silano (Perdigão *et al.*, 2006). Além disso, não existe união química entre a resina composta à base de metacrilato e a matriz de resina epóxi dos pinos de fibra (Monticelli *et al.*, 2006). Este resultado parece justificar a similaridade dos valores obtidos entre o grupo controle com os outros grupos em que o silano foi utilizado.

Com isso, estudos futuros devem ser realizados com a finalidade de analisar protocolos de tratamento de superfície de pinos para melhorar a integração dos pinos pré-fabricados com o material de preenchimento, e ainda com o material de cimentação. Outro fator que deve ser também avaliado é a influência dos fatores de envelhecimento nas propriedades mecânicas dos pinos.

CONCLUSÃO

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

1. A análise pelo método de elementos finitos determina que o teste de flexão de três pontos é válido para determinar tensões de flexão de pinos reforçados com fibra, independentemente do seu nivelamento, desde que a porção cônica seja limitada no momento do ensaio;
2. As características estruturais afetam significativamente as propriedades mecânicas dos pinos de fibra. A resistência à flexão é diretamente correlacionada com a razão fibra/ matriz, enquanto o módulo de flexão é inversamente correlacionado com o número de fibras por mm^2 do pino;
3. Módulo de flexão e resistência à flexão não são afetados por diferenças no diâmetro das fibras presentes nos pinos pré-fabricados;
4. O uso de ar quente após aplicação do silano não produziu aumento da resistência de união entre o pino reforçado com fibra e o núcleo de preenchimento em resina composta. Além disso, o ar quente após aplicação do sistema de dois componentes Silane Coupling Agent (Dentsply) resultou em menor resistência de união;
5. O sistema silano de dois componentes Silane Coupling Agent (Dentsply) apresentou melhores valores de resistência de união do que os silanos pré-hidrolizados na temperatura ambiente de secagem de 23°C.

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* De acordo com a norma da UNICAMP/FOP, baseada na norma do International Committee of Medical Journal Editors – Grupo de Vancouver. Abreviatura dos periódicos em conformidade com o Medline.

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ANEXOS

1. Materiais e Métodos do Capítulo 2

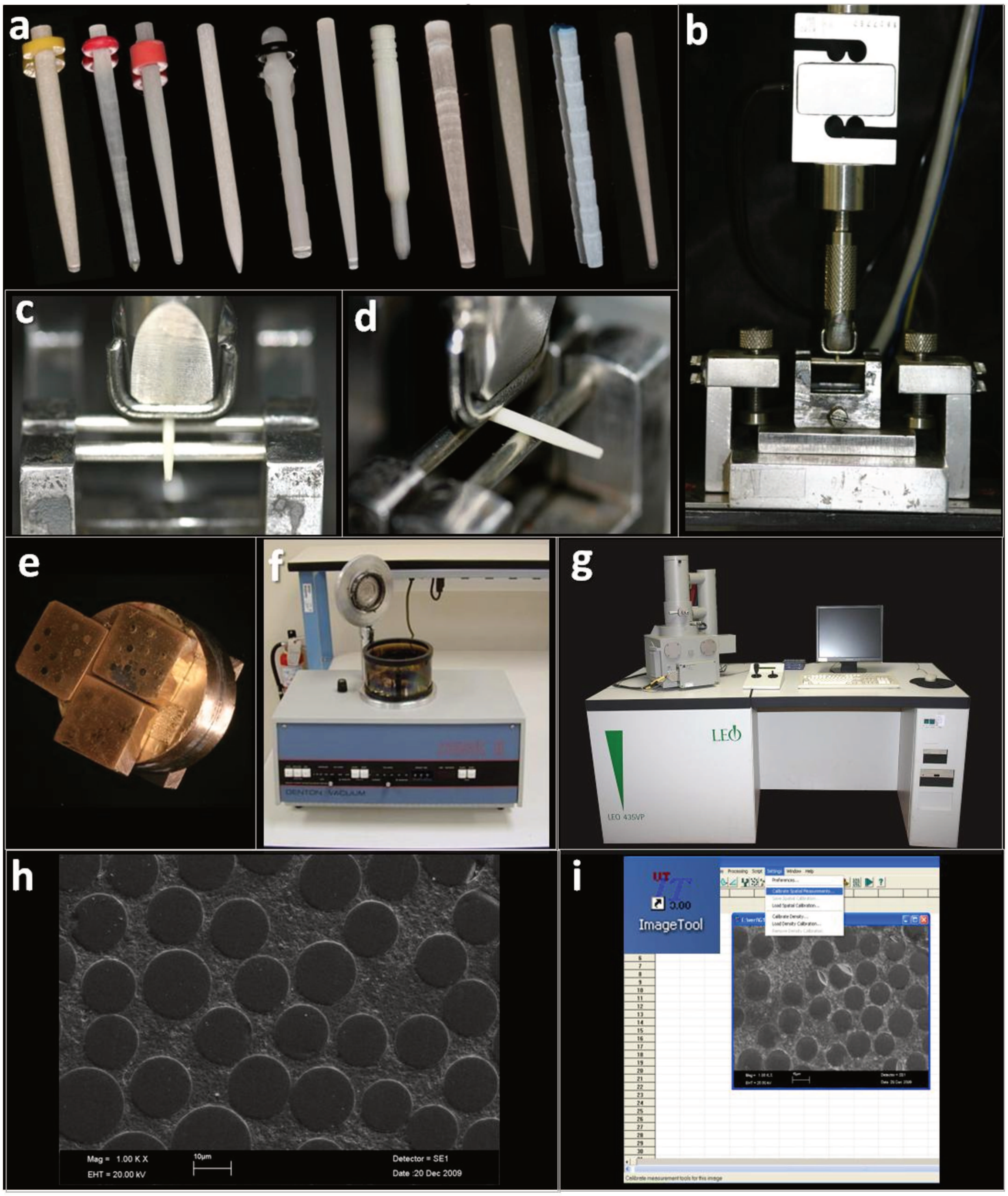

Figura 1. Metodologia utilizada no Capítulo 2: a, sistemas de pinos pré-fabricados reforçados com fibra testados no estudo - Exacto Cônico (Angelus), DT Light SL (VDW), Relyx Fiber Post (3M-Espe), Glassix Radiopaque (Nordim), Para Post Fiber White (Coltène), FRC Postec Plus (Ivoclar), Aestheti-Plus Post (Bisco), Superpost Cônico Estriado (Superdont), Superpost Ultrafine (Superdont), Reforpost (Angelus), e White Post DC (FGM), respectivamente; b, ensaio mecânico de flexão; c, dispositivo utilizado no ensaio mecânico, com regulagem para adaptação da distância entre os suportes inferiores; d, força aplicada somente na porção cilíndrica do pino; e, metalização após o seccionamento dos pinos, na região em que a força foi aplicada, e posterior polimento; f, Metalizador (Baltec SCD 050); g, análise em microscópio eletrônico de varredura; h, micrografia com aumento padronizado (X1000); i, micrografias em programa de processamento de imagen (Image Tool).

2. Materiais e Métodos do Capítulo 3

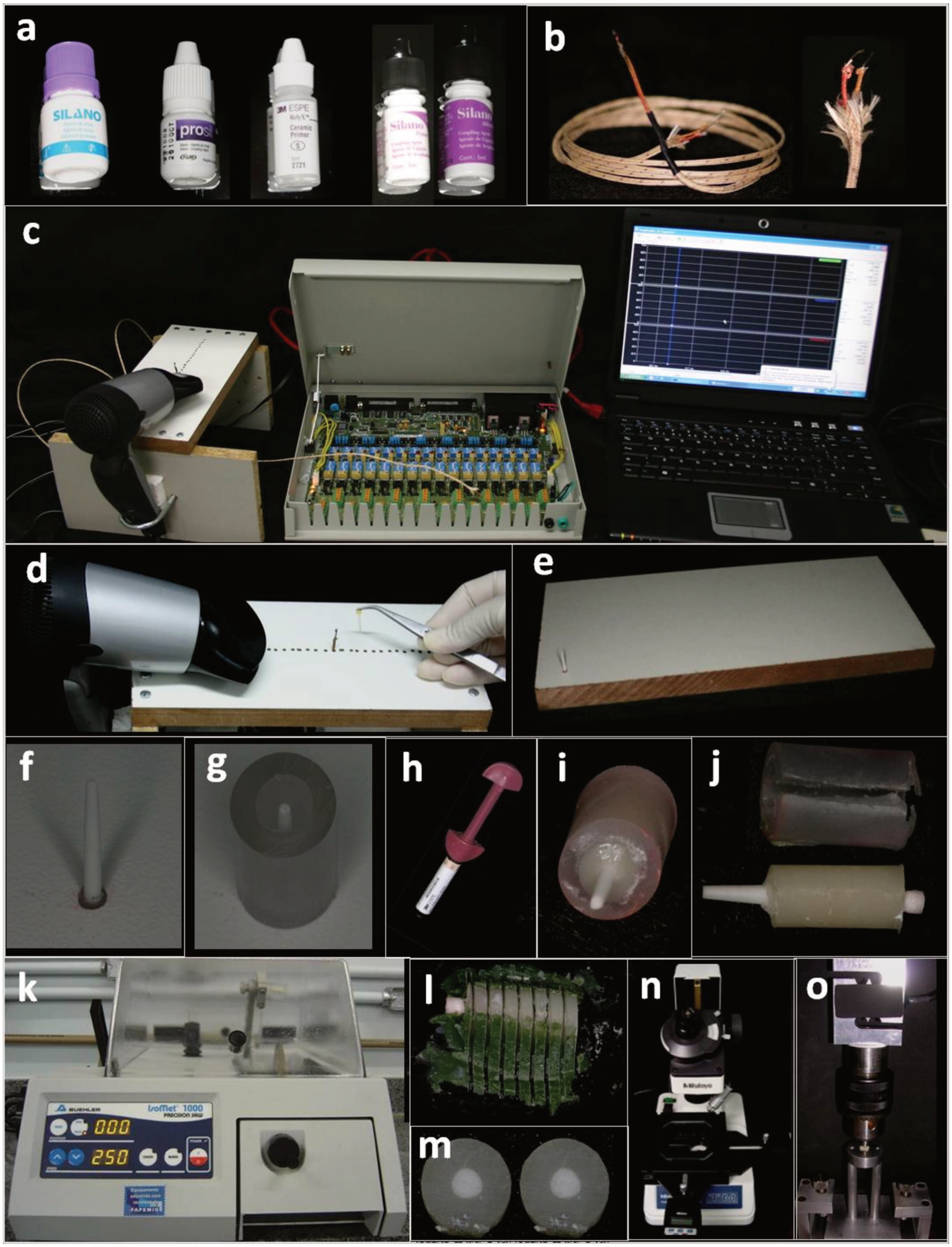

Figura 2. Metodologia utilizada no Capítulo 3: a, agentes silano utilizados no estudo - Silano (Angelus), Prosil (FGM), RelyX Ceramic Primer (3M ESPE), Silane Coupling Agent (Dentsply), respectivamente; b, termopar Tipo T (Cobre-Constatan); c, Secador acoplado em mesa com termopar, este conectado a placa de aquisição de dados e computador com software para controle de temperatura; d, aplicação de ar quente na superfície do pino coberta com silano; e, f, posicionamento do pino em dispositivo; g, posicionamento do tubo plástico transparente com dimensões de 5 mm de diâmetro e 10 mm de altura; h, resina composta utilizada como material de preenchimento; i, após o preenchimento da resina em volta do pino; j, seccionamento do tubo e obtenção da amostra; k, cortadeira de precisão (IsoMet 1000); l, amostra imediatamente após o seccionamento em fatias; m, fatias de 1 mm de espessura; n, Microscópio de mensuração utilizado para medição do diâmetro dos pinos após o seccionamento das amostras; o, ensaio mecânico de micropush-out.

3. Comprovante de envio de trabalho – Artigo do Capítulo 1

ENC: International Endodontic Journal - Manuscript ID IEJ-10-00379

De: **Carlos** (carlosjsoares@umuarama.ufu.br)
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19-Sep-2010

Dear Prof. Soares

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4. Comprovante de envio de trabalho – Artigo do Capítulo 3

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Detailed Status Information

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Corresponding Author	Carlos Soares (School of Dentistry of Federal University of Uberlândia)
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Abstract	This study evaluated the effect of air-drying temperature and different silane coupling agents on the bond strength between glass fiber posts and composite resin core. The post surface was cleaned with alcohol and treated with different silane coupling agents, three prehydrolyzed silanes [Silano (Angelus), Prosil (FGM), RelyX Ceramic Primer (3M ESPE)] and one two-component [Silane Coupling Agent (Dentsply)]. Two different post-silanization air-drying temperatures, 23{degree sign}C and 60{degree sign}C, were applied. A cylindrical plastic matrix was placed around the silanized post and filled with resin composite. Each bonded post provided seven slices for push-out testing. Each slice was loaded to failure under compression at a cross-head speed of 0.5mm/min. Data were analyzed by two-way ANOVA and Scott-Knott tests (α = 0.05). To compare mean of control group to each of the experimental groups, Dunnett's test was used. Scanning electron microscopy (SEM) was used to evaluate the interface of the fractured slices. For the 23{degree sign}C air-drying temperature, the RelyX Ceramic Primer resulted in significantly lower bond strength than the other silane coupling agents, while Silane Coupling Agent (Dentsply) was higher than the other groups. Only for the Silane Coupling Agent,

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