

# UNIVERSIDADE DE RIBEIRÃO PRETO PROGRAMA DE PÓS-GRADUAÇÃO EM ODONTOLOGIA

# Efeitos do tempo de fotoativação no reforço de raízes fragilizadas experimentalmente e restauradas com resina composta e pinos de fibra

**CLEONICE DA SILVEIRA TEIXEIRA** 

Orientador: Prof. Dr. Manoel D. Sousa-Neto

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# **CLEONICE DA SILVEIRA TEIXEIRA**

# Efeitos do tempo de fotoativação no reforço de raízes fragilizadas experimentalmente e restauradas com resina composta e pinos de fibra

Tese apresentada ao Programa de Pós-Graduação em Odontologia da Universidade de Ribeirão Preto como parte dos requisitos para obtenção do título de Doutor em Odontologia, área de concentração: Endodontia.

Orientador: Prof. Dr. Manoel D. Sousa-Neto

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O objetivo desta pesquisa foi avaliar ex vivo os efeitos do tempo de fotoativação no reforço de raízes fragilizadas experimentalmente e restauradas com resina composta (RC) e pino de fibra de quartzo fototransmissor (DT). Com esta finalidade buscou-se verificar: 1) a dureza Vickers (HV) da resina composta; 2) a resistência de união (RU) ao cisalhamento por extrusão e o tipo de falha ocorrida após teste de *push-out* e, 3) sob Microscopia Eletrônica de Varredura (MEV), a interface dentina/adesivo/resina/cimento/pino resultante. As coroas de 60 incisivos superiores de humanos foram cortadas e o remanescente dental padronizado em 17 mm de comprimento. Após 24 h do tratamento endodôntico, os canais foram esvaziados em 12 mm e divididos em 4 grupos (n=15): 1 controle (GC) e 3 fragilizados, nos quais o espaço do canal foi ampliado com pontas diamantadas, originando folga de 1 mm entre o pino DT Light Post e a dentina. Para o reforço das raízes, após a aplicação do sistema adesivo All Bond 2, a RC (Light Core) foi fotoativada através do pino por 40 (G1), 80 (G2) ou 120 s (G3). Depois de 24 h da cimentação dos pinos, os espécimes foram seccionados transversalmente em 6 fatias de 1 mm de espessura. Para a avaliação da dureza da resina (G1, G2 e G3, n=15) e posterior análise em MEV (G1, G2, G3 e GC, n=3) fatias correspondentes às regiões cervical, média e apical foram incluídas em resina epóxica. Após o polimento, a dureza da RC foi avaliada pela média de 3 indentações em cada região, nas distâncias laterais de 50, 200 e 350 µm a partir do cimento/pino. Nas demais fatias (G1, G2, G3 e G4, n=15), a RU foi avaliada pelo teste de *push-out* e as falhas após desunião observadas em estereoscópio. Após preparo para MEV, avaliou-se a interface adesiva de forma qualitativa nominal e ordinal pelo estabelecimento e determinação de escores. A análise de variância de 3 vias (a=0,05) indicou que os fatores tempo, região e distância influíram na dureza e que a interação tempo X região foi significativa. O teste de Tukey revelou que as médias dos valores de HV obtidos em G1 e G2 foram diferentes (p<0,05) de G3. Após o teste de *push-out*, a análise (2, way-ANOVA) das médias de RU (MPa) indicou diferença significativa apenas entre os grupos fragilizados e o controle, mas não entre as regiões. Em todos os grupos foi verificado maior percentual de falhas adesivas e que as falhas coesivas ocorreram apenas nos espécimes fragilizados/reforçados. Em MEV, a análise qualitativa mostrou formação de camada híbrida, tags de resina e ramificações laterais em todas as regiões analisadas, tanto nos grupos fragilizados quanto no controle. A análise qualitativa ordinal (Kruskal-Wallis, a=0,05) indicou que a região do reforço da raiz não influenciou significativamente na densidade de tags formados. O reforço com RC e pino DT proporcionou RU nos grupos fragilizados semelhante (G2) ou superior (G1 e G3) à RU verificada no grupo controle. A região do reforco influenciou a dureza da RC, mas não na RU alcancada. Em regiões mais profundas e em áreas laterais distantes do pino, a dureza da RC foi mais baixa. Com relação ao fator tempo, a fotoativação por 120 s proporcionou valores de HV maiores do que os tempos de 40 e 80 s, mas não influenciou na RU alcançada ou na interface de união formada ao longo do canal radicular.





The aim of this study was to evaluate ex vivo the effect of the light exposure time in the root reinforcement of weakened experimentally roots restored with composed resin (CR) and guartz fiber post. With this purpose were verified: 1) the Vickers hardness (HVN) of the composite resin; 2) the shear bond strength (BS) to dentin and the type of occurred failure after push-out test, and 3) in Scanning Electronic Microscopy (SEM), the dentin/resin/cement/post interface. The crowns of 60 upper maxillary incisors were removed and the length of roots standardized in 17 mm. After 24 h of the root canal filling, the canals were prepared into 12 mm and had formed 4 groups (n=15): 1 control (GC); and 3 weakened with diamond tips, originating recess of 1 mm between the post and the dentine. After application of the adhesive system All Bond 2, the CR (Light Core) was curing light through DT Light Post for 40 (G1), 80 (G2) or 120s (G3). After 24 h of the post cementation, the specimens had been sectioned perpendicularly to its long axle in 6 slices, with 1 mm of thickness. For hardness evaluation of the composite resin (G1, G2 and G3, n=15) and posterior analysis by SEM (G1, G2, G3 and GC, n=3) the selected slices had been enclosed in epoxy resin cylinders and after polishing, the CR hardness were evaluated in each region, at the lateral distances of 50, 200 and 350 µm from the cement/post. The bond strength was performed by the push-out test and, after dislodgments the failures were observed in the stereomicroscope. The specimens were processed for SEM analysis to observe bonding interface formation, hybrid layer quality and resin tag density using a four-step scale method. The 3-way ANOVA was performed (a=0,05) and indicated that the factors: light-exposure time, region and lateral distance from de post had influenced in the HV and the interaction exposure time and region was significant. The Tukey test disclosed that the HV values in G1 and G2 had been different (p < 0.05) from G3. After the push-out test, the 2-way-ANOVA of the BS means (MPa) indicated difference enters the groups but not between the regions. In the weakened groups, it was verified that the light exposure time did not influence the results. The adhesive failures were frequent in the weakened groups as either in the control group. The cohesive failure occurred only in flared/reinforced specimens. In SEM, the qualitative analysis showed formation of hybrid layer, resin tags and lateral branches in all the analyzed regions, independently of the group. The quantitative analysis (Kruskal-Wallis test, a =0,05) indicated that the reinforcement region did not influence in the hybrid layer and resin tags formation. The reinforcement with CR and guartz fiber post provided, in the weakened groups, higher bond strength than in the control group. The reinforcement region influenced the CR hardness, but did not influence in the BS results. In deeper regions and lateral distant areas of the post, the hardness of the CR was lower. With regard to the factor time, the light-exposure for 120 s provided significantly higher values of HV than 40 or 80 s. However, the light-exposure time did not influence in the BS results or in adhesive interface quality throughout the root canal.





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# Introdução Geral

Após o tratamento endodôntico, muitos dentes apresentam estrutura corono-radicular severamente reduzida, tanto pela prévia presença de cáries extensas, fraturas, trauma em dente permanente jovem, patologias pulpares e iatrogenias, quanto pelo excessivo desgaste realizado durante o próprio tratamento endodôntico, o que os tornam frágeis e propensos à fratura (SCHWARTZ; ROBBINS, 2004; DIETSHI et al., 2007). Dependendo do grau de redução estrutural, a fragilidade do elemento dental pode determinar que, mesmo depois de restaurado, o risco de fratura seja relativamente alto, principalmente quando a porção cervical do dente for submetida às forças de tração, compressão e torção, o que geralmente ocorre durante a função mastigatória (FERRARI et al., 2000b; YOLDAS et al., 2005).

Especial cuidado deverá ter o profissional de Odontologia ao selecionar o tipo de tratamento restaurador a ser realizado, principalmente nos casos onde a perda da estrutura coronária impõe a necessidade de retentores intra-radiculares para suportar a restauração ou a coroa do elemento dental (SCHWARTZ; ROBBINS, 2004; MARCHI et al., 2003; YOLDAS et al., 2005; CHEUNG, 2005).

Atualmente, a utilização de retentor intra-radicular pode ser feita de forma direta, com a utilização de pinos pré-fabricados, ou indireta, pela realização de núcleos metálicos fundidos (LEWGOY et al., 2003; SCHWARTZ; ROBBINS, 2004).

Durante muitos anos, diante da necessidade da colocação de um retentor intra-radicular, pareceu não haver dúvidas de que os núcleos metálicos fundidos, quando bem executados, proporcionassem ótima retenção (MARCHI et al., 2003; SCHWARTZ; ROBBINS, 2004). Porém, não há evidências de que os núcleos da resistência metálicos possibilitem aumento de um dente tratado endodonticamente e fragilizado (DIETSHI et al., 2007). Pelo contrário, a fragilidade da estrutural, quando associada ao efeito cunha desse tipo de restauração, ocasiona maior probabilidade de fratura da raiz (BONFANTE et al., 2007). Isso sugere que o núcleo metálico pode, dependendo do caso e do seu design, comprometer a resistência do dente quando fragilizado (MUSIKANT; DEUTSCH, 2006). Portanto, em tais casos, torna-se necessário buscar alternativas aos sistemas de núcleos metálicos fundidos.

A restauração intracanal com um material com módulo de elasticidade semelhante ao da dentina, de modo a restabelecer a forma e espessura das paredes dentinárias fragilizadas, além de promover o reforço da estrutura radicular, é uma opção mais segura do que o total preenchimento do espaço do canal com núcleos metálicos fundidos (GONÇALVES et al., 2006; WU et al., 2007). Esses núcleos, pelo alto módulo de elasticidade do metal, potencialmente transferem as forças mastigatórias concentrando-as na estrutura dentinária circundante (BATEMAN et al., 2003). Em outras palavras, os pinos e núcleos metálicos ou de outro material de rigidez superior à dentina, ao resistirem às forças de deformação, concentram tensões cujas resultantes podem ocasionar fratura do remanescente dental, principalmente nas áreas de pouca espessura dentinária (ASMUSSEN et al., 1999; BOSCHIAN-PEST et al., 2002; CARVALHO et al., 2005).

LUI (1987) sugeriu uma técnica de reforço radicular, na qual as paredes internas da raiz são condicionadas com ácido e, após a aplicação de adesivo, uma resina quimicamente ativada é inserida no canal. Na seqüência, um pino metálico de geometria paralela e de atuação passiva deve ser cimentado. Em outro estudo, LUI (1994) utilizou resina fotopolimerizável para o reforço intracanal e sugeriu o uso de pinos plásticos transparentes como dispositivo auxiliar para a fotoativação. Esses pinos, ao serem introduzidos no canal juntamente com a resina composta, permitem a transmissão da luz em toda a sua extensão e possibilitam a polimerização do material resinoso em maior profundidade (YOLDAS; ALAÇAN, 2005).

O uso de resina composta fotopolimerizável no reforço intracanal, pela facilidade de manipulação, inserção e controle do tempo de trabalho, apresenta-se como melhor opção do que as resinas quimicamente ativadas (LUI, 1994; GONÇALVES et al., 2006). No entanto, mesmo com o uso do pino fototransmissor, ainda se observam valores de dureza mais baixos em regiões da resina mais distantes da fonte de luz (ROBERTS et al., 2004; YOLDAS; ALAÇAN, 2005).

A polimerização deficiente do material resinoso pode determinar alterações nas suas propriedades físicas e mecânicas, tais como dureza, resistência estrutural e estabilidade de cor, diminuindo a longevidade da restauração (PEUTZFELDT; ASMUSSEN, 2005; STANSBURY et al., 2005; AGUIAR et al., 2007).

Deve-se ressaltar que, além da distância da ponta do fotoativador, o processo de polimerização do material pode ser afetado por diversos outros fatores, tais como a potência e o tipo de unidade fotoativadora, o tempo de exposição à luz, a cor e a translucidez da resina, bem como a composição, o tamanho, a quantidade e o tipo da partícula de carga (AGUIAR et al., 2005; EMAMI et al., 2005; VISVANATHAN et al., 2007; LAZARCHIK et al., 2007). Esses fatores, de forma direta ou inversamente proporcional, interferem na quantidade de energia que alcança e polimeriza o material resinoso. Por exemplo, o aumento da espessura da resina interfere negativamente na passagem de luz, o que diminui a quantidade de energia em profundidade. No entanto, sabe-se que essa diminuição de energia pode ser compensada, pelo menos em parte, pelo aumento do tempo de fotoativação do material (EMAMI et al., 2005; LINDBERG et al., 2005; AGUIAR et al., 2007).

Por outro lado, é importante que a resistência de união da resina à dentina e a interface adesiva do sistema formado após o reforço (dentina/adesivo/ resina/cimento/pino) sejam avaliadas de acordo com as estratégias de fotoativação empregadas.

Para a avaliação da resistência de união (RU), testes de adesão convencionais, como os testes de cisalhamento e de microtração, têm sido realizados em superfícies planas de dentina ou esmalte (BOUILLAGÜET et al., 2003; GORACCI et al., 2004). No entanto, para a avaliação dos materiais de uso intra-radicular, testes de cisalhamento por extrusão (*push-out* e *micro-push-out*) são considerados mais adequados, pois, além de possibilitarem avaliação regional, podem ser empregados em superfícies confinadas, à semelhança das paredes do canal radicular após o preparo endodôntico (SOUSA-NETO et al., 2005; PERDIGÃO et al., 2006).

Nos estudos de *push-out*, os baixos valores de RU dos pinos de fibra às paredes dentinárias do canal radicular têm sido relacionados à configuração da cavidade endodôntica, às diferenças estruturais da dentina ao longo da raiz preparada para o pino e com as dificuldades de acesso às regiões mais profundas do canal (GORATTI et al., 2004).

Primeiramente, quanto à configuração da cavidade do canal radicular, verifica-se que após a inserção do material restaurador tem-se uma relação entre superfícies unidas/desunidas, ou fator C, bem superior ao observado em restaurações de superfícies planas, ou de cavidades classe II (BOUILLAGUET et al., 2003; PERDIGÃO et al., 2006). A importância de se avaliar o fator C explica-se pelo fato de que quando a restauração possui mais superfícies livres, possibilita maior escoamento do material, resultando em relaxamento e conseqüente diminuição das forças de contração que ocorrem durante a polimerização da resina. Essas forças podem "puxar" a resina para longe das paredes dentinárias,

resultando em formação de falhas interfaciais e perda de adesão (MOREIRA DA SILVA et al., 2007).

Com relação às diferenças estruturais da dentina, sabe-se que a densidade e diâmetro dos túbulos dentinários diminui de cervical para apical (MJÖR; NORDAHL, 1996; FERRARI et al., 2000). Além disso, o conteúdo mineral e orgânico da dentina difere de acordo com a região analisada e com o envelhecimento dos dentes, o que pode afetar e comprometer os procedimentos adesivos (MJÖR et al. 2001).

Outro fator que pode explicar a menor resistência de união nas partes mais profundas do preparo intra-radicular é a dificuldade de acesso a essas regiões, o que, além de dificultar a fotoativação da resina composta, pode tornar mais críticos os procedimentos de condicionamento, aplicação do sistema adesivo e adequada hibridização da dentina intracanal (VICHI et al., 2002). Para a análise desse fator, a microscopia eletrônica de varredura (MEV) é importante instrumento na verificação dos processos de interação dos materiais restauradores adesivos e pino com a dentina intracanal (PATIERNO et al., 1996; FERRARI; MANNOCCI, 2000; MAGNI et al., 2007; PERDIGÃO et al., 2007a; PERDIGÃO et al., 2007b).

Vários autores realizando estudos sob MEV têm observado que o aspecto usual da hibridização entre adesivo e dentina, com formação de interações laterais e *tags* em forma de cones invertidos, comuns na dentina coronária, não são comumente observados na dentina da região apical do preparo para receber o pino (BOSCHIAN-PEST et al., 2002). Isso pode explicar, em parte, a menor resistência adesiva obtida nessa região (GORACCI et al., 2005; SADEK et al., 2006).

Como observado, existem ainda deficiências nas técnicas restauradoras de dentes tratados endodonticamente e fragilizados, principalmente quanto aos aspectos adesivos. O estudo contínuo da interface adesiva após o reforço intracanal pode elucidar alguns destes aspectos, bem como permitir verificar o tipo de falha ocorrida após a desunião, esclarecendo o processo de adesão ao longo do canal radicular (BOSCHIAN-PEST et al., 2002, PERDIGÃO et al., 2006).

Diante do exposto, no que diz respeito aos aspectos restauradores em dentes fragilizados após tratamento endodôntico, existe a necessidade do estudo e avaliação de procedimentos que maximizem a polimerização da resina composta ao longo do reforço intracanal, tal como o uso de tempos adicionais de fotoativação, avaliando essas estratégias na resistência de união à dentina e na interface adesiva formada.





A proposta do presente estudo foi verificar *ex vivo* os efeitos do tempo de fotoativação no reforço de raízes fragilizadas experimentalmente e restauradas com resina composta e pino de fibra de quartzo fototransmissor. Com essa finalidade, foram realizados três experimentos<sup>1</sup> nos quais se buscou avaliar:

1) a dureza Vickers da resina composta;

2) a resistência de união ao cisalhamento por extrusão, por meio do teste de *push out*, e o tipo de falha ocorrida após desunião e,

3) a interface adesiva resultante formada entre dentina/adesivo/resina/ cimento/pino, por meio de análise quali-quantitativa em MEV.

<sup>&</sup>lt;sup>1</sup> Esta tese foi dividida em capítulos, na forma de artigos, que apresentam os resultados obtidos em cada etapa desse estudo. Cada capítulo foi formatado de acordo com a revista à qual foi submetido.









# **CAPÍTULO 1**

#### Effects of Light Exposure Time on Composite Resin Hardness after Root

Reinforcement Using Translucent Fibre Post\*

#### Artigo submetido à revista:

Journal of Dentistry

\*Artigo formatado de acordo com as normas específicas da revista (acessada em: 1 de outubro de 2007) exceto quanto à disposição das figuras e tabelas.
# Effects of Light Exposure Time on Composite Resin Hardness after Root Reinforcement Using Translucent Fibre Post

Short Title: Composite resin hardness after intracanal reinforcement

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# Effects of Light Exposure Time on Composite Resin Hardness after Root Reinforcement Using Translucent Fibre Post

### ABSTRACT

*Objectives*: The purpose of this *in vitro* study was to evaluate the Vickers hardness (VHN) of a Light Core (Bisco) composite resin after root reinforcement, according to the light exposure time, region of intracanal reinforcement and lateral distance from the light-transmitting fibre post.

*Methods*: Forty-five 17 mm-long roots were used. Twenty-four hours after obturation, the root canals were emptied to a depth of 12 mm and the root dentine was artificially flared to produce a 1 mm space between the fibre post and the canal walls. The roots were bulk restored with the composite resin, which was photoactivated through the post for 40 s (G1 - control), 80 s (G2) or 120 s (G3). Twenty-four hours after post cementation, the specimens were sectioned transversely into three slices at depths of 2, 6 and 10 mm, corresponding to the coronal, middle and apical regions of the reinforced root. Composite VHN was measured as the average of 3 indentations (100 g/15 s) in each region at lateral distances of 50, 200 and 350  $\mu$ m from the cement/post interface.

*Results:* Three-way analysis of variance ( $\alpha = 0.05$ ) indicated that the factors time, region and distance influenced the hardness and that the interaction time x region was statistically significant (p = 0.0193). Tukey's test showed that the mean VHN values for G1 (76.37 ± 8.58) and G2 (74.89 ± 6.28) differed significantly from that for G3 (79.55 ± 5.18).

*Conclusions*: Composite resin hardness was significantly lower in deeper regions of root reinforcement and in lateral areas distant from the post. Overall, a light exposure time of 120 s provided higher composite hardness than the shorter times (40 and 80 s).

**Keywords:** composite resin; hardness; light exposure time; root canal; Vickers Hardness Number.

# 1. Introduction

The restoration of endodontically treated teeth commonly presents a challenge to dentists, especially in cases of extensive crown-root destruction.<sup>1,2</sup> Excessive root canal flaring can result in a large, conical and insufficiently retentive post.<sup>3,4</sup> On the other hand, the use of posts with a smaller diameter than that of the post space leads to the formation of voids that, even if filled with cement, represent potentially weakened areas.<sup>3</sup>

Care should be taken in the post selection because posts with a high module of elasticity, such as metal posts (up to 20 times the value for dentine which is approximately 18 GPa), cause force concentration in areas where the dentine wall is thin, which may increase the incidence of root fractures.<sup>2,5-7</sup> Therefore, root restoration (and reinforcement) with a material that is elastically compatible with dentine, such as composite resin or glass ionomer cement, is considered to be safer than the use of cast metal post-core systems.<sup>8,9</sup>

Due to their ease of handling, incremental insertion and control of working time, light-activated composite resins present a better option for intracanal reinforcement than chemically activated composite resins.<sup>10,11</sup> However, polymerisation and hence the adhesion of light-activated resin materials may be inhibited in the most apical regions of post space because these areas are far from the light source.<sup>12</sup>

The depth of cure is affected by several factors, including the power and type of lightcuring unit (LCU), light exposure time, distance from the LCU tip, composite shade and translucence, as well as the composition, size, amount and type of filler particles.<sup>13-19</sup> The influence of these factors is directly or indirectly proportional to the density of the LCU energy that reaches and polymerises the resin material. For example, as the depth and percentage of cure are influenced by the LCU energy density (E = power x light exposure time),<sup>13,20-22</sup> longer exposure times may compensate for the loss of energy density caused by the increase in the sectional distance and provide a greater percentage of cure.<sup>13,16,23</sup>

In addition to evaluating composite mechanical properties the hardness test also serves as an indirect indicator of its degree of conversion.<sup>18,24,25</sup>

In cases of root reinforcement, the use of light-transmitting fibre posts permits a more effective polymerisation of the composite resin in areas far from the light source compared to conventional photoactivation at the canal entrance.<sup>12</sup> However, Roberts et al.<sup>26</sup> reported that the use of a light-transmitting glass fibre post increased composite hardness values in the apical regions of simulated root canals only very near the post. In addition, even with the use of light-transmitting posts lower hardness values have been reported in composite regions farther from the light source.<sup>12,26</sup> These results emphasize the need for the development of strategies that optimize composite polymerization in more apical regions of intracanal reinforcement.

Since the degree of conversion of resin materials may be related to their mechanical properties and influence the bond strength and longevity of the tooth, the purpose of this study was to investigate the influence of light exposure time, region and lateral distance from the translucent fibre post on the hardness of a composite resin used for the reinforcement of experimentally weakened roots.

### 2. Material and Methods

#### 2.1 Tooth Selection and Preparation

Forty-five extracted noncarious human maxillary central incisors were stored in 0.1% thymol diluted in saline at 4°C and pH 7 and used within 3 months following extraction. The crowns were sectioned transversally at the cementoenamel junction with a double-faced diamond disc No. 6911H (Brasseler Dental Products, Savannah, GA, USA) at low speed under air/water spray coolant and discarded. Root length was standardized at approximately 17 mm. Working length was established at 1.0 mm short of the apical foramen. Root canal preparation was performed according to the crown-down technique using #2 to #4 Gates Glidden drills (Union Broch, York, PA, USA) and rotary instruments (Profile .04/06 tapers; Dentsply/ Maillefer, Tulsa, OK, USA) incrementally up to the working length. The apical stop was enlarged up to a #50 file/.04 taper. During preparation, the canals were irrigated with 2 mL of 1% sodium hypochlorite at each change of instrument. A final flush was performed with 2 mL of deionised water.

The root canals were then dried with absorbent paper points (Dentsply/Maillefer) and filled with gutta-percha main cones and accessory points (Dentsply/Maillefer) and a resinbased endodontic sealer (AH Plus; Dentsply De Trey, Konstanz, Germany) (Table 1). Root canal obturation was carried out according to Tagger's<sup>27</sup> hybrid technique. After vertical compaction and placement of provisional restorations (Citodur; Septodont, Switzerland), the roots were stored in 100% relative humidity at 37°C for 24 h.

Product	Composition	Batch	
(Manufacturer)			
AH Plus <sup>TM</sup> (Dentsply DeTrey, Konstanz, Germany)	<i>AH Plus Paste A:</i> Bisphenol-A epoxy resin; Bisphenol-F epoxy resin; Calcium tungstate; Zirconium oxide; silica; iron oxide pigments.	0603002042	
	AH Plus Paste B: Dibenzyldiamine; aminoadamantane; Tricyclodecane-diamine; Zirconium oxide; silica; silicone oil.		
All-Bond 2 (Bisco Inc., Schaumburg, IL, USA)	<i>UNI-ETCH</i> : 32% Phosphoric acid, Benzalkonium Chloride and xanthum gum thickener	0600001033	
	Primer A: NTG-GMA, acetone, ethanol and water	0600001076	
	<i>Primer B</i> : BPDM, acetone, ethanol and photoinitiator	0600001077	
	D/E Resin: Bis-GMA, UDMA,	060000717	
	HEMA, photoinitiator (CQ) and amine activator		
Light-Core <sup>TM</sup> (Bisco Inc., Schaumburg, IL, USA)	Bis-GMA, Ethoxylated Bisphenol A Dimethacrylate, glass frit (>60%)	0600004829	
Duolink (Bisco Inc., Schaumburg, IL, USA)	Base: Bis-GMA, TEGDMA, UDMA, glass filler	0600004680	

Table 1 - Materials used in the experimental procedures, with the respective manufacturer, composition and batch number.

*NTG-GMA*, Na-N-tolylglycine glycidylmethacrylate; BPDM, Biphenyl dimethacrylate; *Bis-GMA*, bisphenol A diglycidyl methacrylate; *UDMA*, urethane dimethacrylate; *HEMA*, 2-Hydroxyethyl methacrylate; *TEGDMA*, Triethylene glycol dimethacrylate; *CQ*, camphorquinone.

### 2.2 Root Dentine Flaring and Post Space Preparation

Twenty-four hours after the root canal filling the gutta-percha was removed from the root canal to a depth of 12 mm using Gates-Glidden drills No. 4 and 3 (Dentsply/Maillefer), specific burs supplied with the fibre post system (DT; Bisco Inc., Schaumburg, IL, USA) and heated digital compactors (Dentsply/Maillefer), maintaining 4 mm of filling material in the apical third.

The thickness of the root canal dentine walls was reduced using high-speed diamond burs Vortex No. 4137 (Vortex Ind. e Comércio, São Paulo, SP, Brazil) and KG No. 717 (KG Sorensen, São Paulo, SP, Brazil) under air/water spray coolant, in such a way as to produce a circumferential space of approximately 1.0 mm between the fibre post (DT Light Post #2; coronal diameter = 1.8 mm and apical diameter = 1.0 mm; Bisco Inc., Schaumburg, IL, USA) and the circumjacent dentine walls. Each post was fitted and sectioned at the coronal third perpendicularly to its long axis, 4 mm above the coronal border of the root using a doubled-faced diamond disc (Brasseler No. 911H) at low speed under air/water spray coolant.

# 2.3 Intracanal Restoration with Composite Resin

Prior to composite resin restoration, the canals were irrigated with 10 mL of deionised water and dried with absorbent paper points. The intracanal dentine was etched with 32% phosphoric acid (Uni-Etch; Bisco Inc.) (Table 1) for 15 s, rinsed with deionised water for 30 s and gently dried with absorbent paper points (Dentsply/Maillefer). A 3-step "etch-and-rinse" adhesive system (All Bond 2; Bisco Inc.) (Table 1) was applied to the slightly moist dentine with disposable microbrush tips (3M/ESPE, St. Paul, MN, USA), according to the

manufacturer's instructions. The material was photoactivated by positioning the tip of the light-curing unit (Curing Light 2500; 3M/ESPE; 500 mW/cm<sup>2</sup> light intensity) at the canal entrance for 20 s.

For the intracanal restoration the canal space was bulk filled with a translucent composite resin (Light Core; Bisco Inc.) (Table 1) using digital compactors (Dentsply/Maillefer). In each canal a fibre post (DT Light Post; Bisco Inc.) coated with a thin layer of petroleum jelly (Vimak, São Paulo, SP, Brazil) was centrally inserted into the resin mass along the whole post space extension. After removal of the resin excess the tip of the LCU was placed over the post and the device was activated according to the light exposure times established for each group of 15 specimens: 40 s (group 1); 80 s (group 2) and 120 s (group 3). After composite resin polymerisation, the post was clamped with needle-nose pliers and removed from the canal.

### 2.4 *Post Cementation*

The root canals of all groups were reprepared with the bur supplied with the kit of the post system used (#2 DT Light Post, Bisco Inc.), washed with 10 mL of deionised water and dried with absorbent paper points. After removal of the petroleum jelly coating the posts were rinsed with water and dried in a mild air stream. Primer B (AB, Bisco Inc.) was applied to the post surface, gently air thinned and light cured for 10 s. The root canals were etched with phosphoric acid (Uni-Etch; Bisco Inc.), applied with disposable microbrush tips (3M/ESPE), and left for 15 s, rinsed with deionised water for 30 s and gently dried with absorbent paper points. Two consecutive drops of primer B were applied and the excess was removed with absorbent paper points. Equal amounts of Duo Link<sup>TM</sup> resin cement

(Bisco Inc.) were mixed and the material was taken to the canal with a lentulo spiral. Each post was seated into the respective canal with gentle pressure, excess cement was removed with a microbrush tip and the material was light activated for 40 s. After 4 min, the specimens were placed in black light-proof receptacles and stored in 100% relative humidity at 37°C for 24 h.

The root specimens were individually placed on a precision cutting machine (Isomet 1000; Buehler, Lake Forest, IL, USA) with a water-cooled diamond saw (South Bay Technology, San Clement, CA, USA) and sectioned perpendicularly to the long axis of the posts. Three slices were obtained from each specimen, representing the coronal, middle and apical regions of the post space preparation (one slice per region), at depths of 2, 6 and 10 mm, respectively, from the coronal border of the root.

### 2.5 Hardness Test

The root sections were embedded in epoxy resin (Paladur; Heraeus Kulzer, Wertheim, Germany) with the test surface facing upward and polished in a polishing machine under water coolant with silicon carbide paper of decreasing abrasiveness (from 600 to 1500 grit) and complemented with 3  $\mu$ m, 1  $\mu$ m, 0.5  $\mu$ m and 0.03  $\mu$ m (Buehler Ltda, Lake Bluff, IL, USA) diamond polishing pastes for 1 min each. The specimens were copiously rinsed in tap water at each change of abrasive paper or paste.

After polishing, the specimens were submitted to a hardness test in a Shimadzu HMV2 hardness tester (Newage Testing Instruments, Inc., Southampton, PA, USA) with a square base diamond pyramid Vickers hardness indenter. In the coronal, middle and apical regions of root reinforcement, hardness was calculated at three different lateral areas of the

composite resin, determined at distances of 50  $\mu$ m (p – close to post), 200  $\mu$ m (m – middle region) and 350  $\mu$ m (d – close to dentine) in relation to the cement/post interface (Fig. 1a, b). At each lateral distance (p, m and d) hardness was calculated as the average of the three measurements, totalling nine indentations in each region (Fig. 2a, b). Each indentation was made using a 100 g static load for 15 s. Vickers hardness was calculated based on the average of the diagonals measured under examination with an optical microscope (Shimadzu HMV2, ×400 magnification; Fig. 2b). All measurements were calculated and recorded as Vickers hardness number (VHN) using Newage C.A.M.S software (Computer Assisted Measurement System; Newage Testing Instruments, Inc., Southampton, PA, USA). Representative images of each third were photographed at ×100 and ×400 magnification for analysis and illustration of the results.



Fig. 1 - (A) Optical microscopy image ( $\times 100$ ) of the apical region of root reinforcement in a specimen submitted to a 120-s light exposure time (Group 3), showing the interface between the DT Light Post (P), Duo Link resin cement (RC) and Light Core composite resin (R). (B) Higher magnification of Fig. 1a (400x). Quartz fibres (circular sections) wrapped in epoxy resin (arrows) can be observed in the post (P). A large amount of filler particles of different sizes and shapes is observed in the composite resin (R) (\*), interwoven within the resin matrix.

# 2.6 Statistical Analysis

Data were analysed statistically using SPSS 13.0 for Windows (SPSS Inc, Chicago, IL, USA) statistical software. Means and standard deviations were calculated and data homogeneity and normality were tested by Levene's and Kolmogorov-Smirnov's tests, respectively. Three-way analysis of variance (ANOVA) was used to compare the variables: light exposure time, reinforcement region and lateral distance from the light-transmitting post. Post-hoc tests were calculated using the Tukey's multiple-comparison test. A confidence interval of 95% was set for all tests ( $\alpha = 0.05$ ).



Fig. 2 - (A) Optical microscopy image ( $\times 100$ ) of the apical region of root reinforcement in a specimen submitted to an 80-s light exposure time (G2). Three indentations were made for each lateral distance from the post (p, m and d). The number in the upper right corner (VHN=76.8) is the hardness value corresponding to the last indentation made after image acquisition. (B) Optical microscopy image ( $\times 400$ ) of the coronal region of root reinforcement in a specimen submitted to a 40-s light exposure time (G1) at the moment at which the diagonals used to calculate Vickers hardness (VHN=83.7) were measured. Despite the high filler content, the image obtained after indentation allowed adequate observation of both diagonals.

### 3. Results

The results of the hardness test are shown in Tables 2-4 and Figures 3-4. A total of 405 VHN means were obtained (135 per group, 45 in each root region and 15 for each lateral distance) resulting from 1,215 indentations. All analysed factors (light exposure time, reinforced root region and lateral distance from the post) had a statistically significant influence on the composite resin hardness (Table 2, three-way ANOVA). The analysis of factor interactions showed that only the interaction light exposure time x root reinforcement region had a statistically significant effect on the results obtained (p = 0.0193).

Variation Source	Sum of Squares	Degrees of Freedom	Mean Squares	F value	p value*
Light exposure time	1528	2	764	24.17	< 0.001
<b>Reinforced root region</b>	4485	2	2243	70.96	< 0.002
Lateral distance from the post	1224	2	612	19.37	< 0.003
Time x Region	376	4	94	2.97	0.0193
Time x Lateral distance	270	4	67	2.14	0.0758
<b>Region x Lateral distance</b>	72	4	18	0.57	0.6828
Time x Region x Lateral distance	370	8	46	1.46	0.1696
Error	11947	378	32		

Table 2: Analysis of variance of the factors light exposure time, reinforced root region and lateral distance from the post (p < 0.05).

\*Significance level was set at 5%.

Group	Light exposure time	<b>Reinforced region</b>	Microhardness (VHN) (SD)
	40 s	coronal	81.89 (6.99) a
1	40 s	middle	76.15 (8.70) b
	40 s	apical	71.07 (6.28) c
	80 s	coronal	78.60 (5.30) a
2	80 s	middle	75.52 (4.73) a
	80 s	apical	70.56 (6.01) b
3	120 s	coronal	81.78 (4.02) a
	120 s	middle	80.55 (4.55) a
	120 s	apical	76.31 (5.30) b

Table 3: Mean microhardness values generated by the interaction of the factors light exposure time and root reinforcement region within each group.

\*Different letters (a-c) indicate statistically significant difference at 5% level between the reinforced root region only within each group (Tukey's test; p < 0.05).

Overall, the mean VHN values for G1 (76.37 ± 8.58) and G2 (74.89 ± 6.28) differed significantly (p < 0.05) from that for G3 (79.55 ± 5.18). However, the analysis of the interaction light exposure time x root reinforcement region within each group (Table 3, Figure 3) showed no statistically significant difference (p > 0.05) between the coronal and middle regions when the 80 s (G2) and 120 s (G3) exposure times were used. On the other hand, the apical region of the root reinforcement differed significantly (p < 0.05) from the others and had lower VHN means. Furthermore, in G1 (40-s light exposure time), there was a statistically significant difference (p < 0.05) between all root reinforcement regions, the coronal region having higher hardness values.

Time	<b>Reinforced Region</b>	Lateral distance	Vickers hardness (VHN)*
		from the post	
80 s	apical	d	68.71 a
80 s	apical	m	69.04 a
40 s	apical	d	69.29 ab
40 s	middle	d	71.42 abc
40 s	apical	р	71.75 abcd
40 s	apical	m	72.17 abcd
120 s	apical	d	73.08 abcde
80 s	middle	m	73.85 abcdef
80 s	middle	d	73.89 abcdef
80 s	apical	р	73.94 abcdef
80 s	coronal	d	75.50 abcedfg
120 s	apical	m	76.71 bcdefgh
40 s	middle	m	76.80 bcdefgh
80 s	middle	р	78.81 cdefgh
80 s	coronal	m	79.06 defgh
40 s	coronal	d	79.15 defgh
120 s	apical	р	79.16 defgh
120 s	middle	р	80.20 efgh
40 s	middle	р	80.23 efgh
120 s	coronal	d	80.40 efgh
120 s	middle	m	80.43 efgh
120 s	middle	d	81.03 fgh
80 s	coronal	р	81.26 fgh
120 s	coronal	р	81.74 gh
40 s	coronal	m	82.94 gh
120 s	coronal	m	83.20 h
40 s	coronal	р	83.60 h

Table 4: Results for Tukey's multiple-comparison *post-hoc* test arranged in an increasing sequence of hardness means.

\*Different letters (a-h) indicate statistically significant difference at 5% level (Tukey's test; p < 0.05). p, m, d: distances from cement/post interface (p - close to post - 50 µm; m - middle region - 200 µm; d - close to dentine - 350 µm).



Fig. 3 - Composite VHN means and confidence interval for the different lateral distances of the composite root reinforcement in each region analyzed according to the light exposure time.



Fig. 4 - Composite VHN means and confidence interval for the different root reinforced regions within the lateral distances analyzed and according to the light exposure time.

Comparing the lateral distances (p, m and d) in each region analysed and according to each light exposure time a statistically significant difference (p < 0.05) was observed only in the middle region of root reinforcement in Group 1 specimens, when the light exposure time was 40 s (Figure 4). In this case, the mean composite hardness at distance d (the farthest laterally from the light-transmitting post) differed significantly from the mean hardness of lateral distance p, the closest to the post.

Table 4 presents the results of Tukey's multiple-comparison post-hoc test arranged by increasing VHN means. It is possible to observe that the highest VHN means were obtained in the coronal and middle regions of root reinforcement.

### 4. Discussion

Several direct and indirect methods have been used to evaluate or estimate the degree of polymerisation of resin materials. Spectroscopic techniques (e.g. Fourier transform infrared spectroscopy - FTIR and Fourier transform Raman spectroscopy -FT-Raman) and electronic resonance may be use to directly quantify the percentage of cure of different composites.<sup>23,28</sup> However, due to the complexity and high cost of these methods, hardness measurements may be used to assess structural changes and predict, though indirectly, the degree of conversion of the resin materials.<sup>25,29</sup>

Composite hardness correlates directly with the module of elasticity and may serve as an indirect parameter to evaluate the percentage of composite monomer conversion.<sup>18,30</sup> As the organic matrix of the dimethacrylate system undergoes crosslinking substantial changes occur in its internal molecular organisation and hence in its mechanical properties.<sup>31,32</sup> This polymerisation crosslinking causes a progressive increase in the elasticity module of the resin material and, consequently, in its hardness.

The hardness tests may be used to assess the depth of cure within the root canal<sup>33</sup> or in simulated canals<sup>12,34</sup> particularly in root reinforcement techniques with composite resin, in which the LCU may have an insufficient intensity or might not be able to transmit light through the material layer.

In this study the Vickers hardness (VHN) test was used to evaluate the effects of light exposure time, root reinforcement region and lateral distance from the light-transmitting post on the hardness of the composite resin used to reinforce weakened roots. It was observed that the region of root reinforcement affected significantly the composite hardness with a progressive decrease moving from the coronal to the apical region, with all light exposure times. There was a significant difference between the hardness values recorded in the coronal and middle regions and those of the apical root region, within each experimental group. These results were predictable and may be attributed mainly to the distances of 2, 6 and 10 mm, respectively, in relation to the cervical border of the root. It is known that light passage is attenuated with an increase in the sectional distance from the irradiated surface, due to the absorption and reflection caused by load particles and other additives present in the resin materials, decreasing the depth of cure.<sup>17,31</sup>

For light-activated composite resins the presence of a light source with adequate intensity and exposure time is essential for the complete curing of the material. If the composite resin does not receive sufficient light exposure at the correct wavelength, the degree of conversion of monomers is considerably reduced.<sup>26</sup> As the adequate polymerisation of resin materials in deeper intracanal regions is sometimes dubious, in

clinical practice the lower energy density delivered to these areas might be compensated for by increasing the light exposure time,<sup>13</sup> choosing more intense light sources or using materials that enhance energy transmission.<sup>19,35</sup>

The light exposure time influenced significantly the VHN means recorded in the present study, although the times of 40 and 80 s (G1 and G2) provided similar results in all regions of the composite-reinforced roots. It should be emphasised that post cementation with Duo Link resin cement yielded an additional photoactivation time of 40 s, which might have contributed to the lack of significant differences between these groups. However, the specimens with a light exposure time of 120 s had significantly higher VHN means than those of the other groups, especially in the apical region.

A translucent composite resin (Light Core) was used in this study in order to maximize light irradiation along the canal length, as the composite shade may interfere with its transmission and, consequently, the degree of conversion of monomers and composite hardness.<sup>15</sup> In a recent study, Lazarchik et al.<sup>19</sup> observed that, unlike other shades, translucent composite resins had Knoop hardness values at the top surface similar to those observed at a depth of 3 mm, regardless of the photoactivation technique (incremental or bulk). Due to its translucency, attributed to the absence of opacifying components and presence of quartz load particles, it may be assumed that this type of resin optimises light transmission. Additionally, according to the manufacturer, this material may be adequately polymerised to a depth of 5 mm with a light exposure time of only 20 s, which is shorter than the shortest exposure time used in this study.

In this investigation, the use of a translucent fibre post (DT Light Post) might also have allowed a greater depth of cure of the composite resin. Lui<sup>10</sup> et al. reported that it is possible to reach a depth of cure of the intraradicular composite resin reinforcement of 11 mm using the Luminex system (Light Transmitting Plastic Post) with a light exposure time of 40 s. In another experiment in simulated root canals, Yoldas & Alaçan<sup>12</sup> verified that composite photoactivation with the use of light-transmitting plastic posts or composite posts reinforced by fibre posts was superior to that obtained with conventional photoactivation. Additionally, these authors observed that resin polymerisation occurred up to a depth of 14 mm, with a light exposure time of 90 s. Nevertheless, as observed in the present study, even with the use of light-transmitting posts, hardness measurements were comparatively lower in regions farther from the LCU.

According to Teixeira et al.,<sup>36</sup> although light-transmitting posts are capable of transmitting light to considerable depths, 10 mm in the case of DT Light Post, the amount of transmitted light is lower than 40% of the incident light. This may explain the fact that in the study here reported lower hardness means were recorded in the apical region of root reinforcement even with the use of longer light exposure times. Furthermore, the irradiation time necessary to provide an adequate polymerisation seems to increase exponentially with an increase in the thickness of the material to be polymerised.<sup>35</sup>

In the present study, the hardness of the most superficial layer was measured on the base of the first slice, which corresponded to a depth of 2 mm from the top surface of the composite. The longer light exposure time together with the use of a translucent resin and a translucent fibre post seem to have been determinant factors in obtaining composite resin VHN means in the middle and apical reinforcement regions higher than 80% of the VHN means recorded in the coronal region. While these results are promising, they cannot be considered as ideal because the reference parameter was not the hardness on the top surface

of the material. This minimum conversion percentage (at least 80% of top hardness) is usually determined<sup>37</sup> and accepted as appropriate in tests for the verification of depth of cure.<sup>18,38</sup> However, even in studies in which the composite hardness of the top surface was evaluated,<sup>14,15,22,26</sup> assuming that the maximum possible hardness was reached on the top surface of the material, the determination of 80% as adequate for bottom hardness cannot be considered sufficient from a scientific standpoint.<sup>35</sup> According to Musange & Darvell,<sup>35</sup> the maximum percentage of cure of a composite resin, if well polymerised and evaluated on its top surface, is around 65%, which would represent conversion values significantly lower at the depth corresponding to 80% of top surface hardness. This raises doubts about the longevity and biological compatibility of the material in these regions.

Due to the lack of studies carried out to investigate composite hardness within the root canal, the findings of this study cannot be considered as definitive or compared to those of other materials because of the structural differences between the several types of composites and fibre posts in use. In addition, other properties of the materials used for root reinforcement should be investigated, as well as bond strength and the adhesive interface formed by the dentine/adhesive/resin/cement/post set, including material-aging processes such as thermocycling.

# 5. Conclusions

Based on the data obtained in this *in vitro* study, it may be concluded that: 1. Light exposure time, root reinforcement region and lateral distance from the post influenced the hardness values obtained. 2. Overall, composite resin hardness was significantly lower in

the deeper regions of root reinforcement and in lateral areas far from the fibre post. 3. The interaction light exposure time and reinforcement region was significant, which means that in deeper intracanal regions composite hardness increased with an increase in light exposure time. 4. The comparison of the different photoactivation times showed that a light exposure time of 120 s provided higher composite hardness than the shorter times (40 and 80 s), especially in the apical region.

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# **CAPÍTULO 2**

# Bond Strength of Fiber Posts to Experimentally Flared Root Canal Dentin

# Reinforced With Composite Resin: Effect of Light-Exposure Time\*

# Artigo submetido à revista:

# Operative Dentistry

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# Bond Strength of Fiber Posts to Experimentally Flared Root Canal Dentin Reinforced With Composite Resin: Effect of Light-Exposure Time

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Running Title: Adhesion of fiber posts to weakened/resin-reinforced root dentin

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### **CLINICAL RELEVANCE**

Intracanal restoration with adhesive resin materials and fiber posts may be indicated for structural reinforcement of roots with thin dentin walls. Photoactivation of translucent composite resins through light transmitting posts may provide, depending on the employed light-exposure time, similar or higher bond strength to dentin than that obtained in roots with thicker dentin walls, even in regions distant from the light-curing source.

# ABSTRACT

**Purpose:** This *ex vivo* study evaluated the influence of different light-exposure times on the interfacial bond strength of DT Light Post (Bisco Inc.) fiber posts to experimentally weakened root dentin restored with Light Core (Bisco Inc.) composite resin. Methods & Materials: Sixty 17-mm long maxillary incisor roots were used. Twenty-four hours after obturation, the root canals were emptied up to a depth of 12 mm and 4 groups (n=15) were formed at random. In the 3 experimental groups (G1, G2 and G3), root dentin was flared to produce a 1-mm space between the fiber post and the canal walls. In the control group (G4), the roots were not experimentally weakened. The roots in the experimental groups were bulk restored with Light Core composite resin, which was light-activated through the DT Light Post for either 40 s (G1), 80 s (G2) or 120 s (G3). The posts were cemented (Duo Link-Bisco Inc.) and, after 24 h, the roots were sectioned transversely at the coronal, middle and apical regions producing 1-mm-thick slices ( $\pm$  0.1 mm). Push-out tests were performed (0.5 mm/min, Instron 4444) and failure modes were observed under stereomicroscopy. **Results:** Means in MPa ( $\pm$ SD) were: GC=7.94  $\pm$  2.78; G1=10.36  $\pm$  2.99; G2=9.03  $\pm$  2.69 and G3=10.28  $\pm$  3.16. Two-way ANOVA ( $\alpha$ =0.05) indicated statistically significant difference among the groups (p<0.001), but not among the post regions (p>0.05). Comparing the weakened/reinforced groups, composite light-exposure time did not influence the results (p>0.05). There was a higher percentage of adhesive failures (in the post or dentin) in the control (73.33%) and experimental groups (85.18%). Cohesive failures occurred only in the weakened/reinforced roots (100%). **Conclusion:** Root reinforcement with composite resin and light transmitting posts provided higher bond strength to dentin than the control group, independently of the composite light-exposure time and analyzed region.

#### **INTRODUCTION**

Endodontically treated teeth may be compromised due to caries, excessive hard tissue removal or previous restorations, which result in loss of coronal structure and, many times, intra-radicular structure. Metallic intracanal posts have been commonly used in these clinical situations to provide the required retention for subsequent prosthetic rehabilitation.<sup>1</sup> Nevertheless, dentin removal at the coronal root third may produce canals with thin tapered walls, where conventional restorations frequently lead to irreversible root fractures.<sup>2</sup> Therefore, these teeth demand the use of restorative techniques that reinforce the weakened root structure.<sup>3</sup>

Intracanal restoration with resin materials followed by cementation of metallic or fiber posts has been considered an effective technique for root reinforcement.<sup>2-4</sup> The goal of using adhesive restorative techniques is to reinforce the root by increasing its internal thickness. The adhesion of a resin material, which is elastically compatible with dentin,

may increase the resistance to fracture.<sup>3,5</sup> In addition, it has been observed that the use of intraradicular posts adhered both to the dentin and to the coronal core provide better distribution of forces along the canal, thus contributing to reinforce the tooth structure.<sup>2,6</sup>

Although some studies have suggested the cementation of metallic posts after root reinforcement<sup>2,3</sup>, the use of esthetic resin fiber posts, such as glass fiber and quartz prefabricated posts, has been referred to as particularly advantageous. Fiber posts not only contribute to generate a more uniform stress distribution to the remaining tooth structure<sup>7</sup> and minimize the risks of irreparable root fractures,<sup>6</sup> but also enhance the esthetic results of the coronal restoration due to its light transmission property.<sup>8</sup> The use of light-transmission fiber posts allow composite resin photoactivation to be performed with the same post that will be further cemented into the root canal.<sup>9</sup>

The push out test has been used for evaluation of the adhesion between post/composite resin/resin cement and root canal dentin using different types of posts and bonding protocols.<sup>10-14</sup> This type of test may be used to determine the regional adhesive forces after restoration and post cementation in weakened roots. Several studies that investigate regional bond strength have found lower values at the middle and apical root canal regions.<sup>10,12-14</sup> On the other hand, other authors<sup>15,16</sup> have reported higher bond strength at more apical canal regions. Although methodological differences may explain these divergent results, several issues referring to adhesion to root canal dentin remain unclear. In spite of the high-quality esthetic outcomes and the favorable laboratorial and clinical results, even with the advent of adhesive cementation, the loss of adhesion of fiber posts to root canal dentin is still the main cause of failure of fiber post-retained restorations.<sup>17,18</sup>

Light-activated composite resins have better handling characteristics than self-cured ones, mainly because the former allow better setting time control, which facilitates root canal filling during the resin reinforcement procedures.<sup>9</sup> However, when light-activated composite resins are placed at more deeper regions or regions with thickness greater than 2-3 mm, polymerization may be undermined and affect the material's hardness.<sup>19</sup> It has been reported that composite resin polymerization depth is directly related to its thickness and is influenced by the curing light intensity.<sup>20</sup> The access to the resin material, the distance to the light-curing source and the length of exposure to light may affect composite resin hardness<sup>9,19-21</sup> and affect negatively some of its mechanical properties.<sup>22</sup> On the other hand, some authors<sup>23,24</sup> have observed that a longer light-exposure time may compensate a lower light intensity and promote adequate polymerization. However, the influence of the light-exposure time on the mechanical properties of composite resins used for root reinforcement, more specifically bond strength to dentin, has not yet been evaluated.

The purpose of this *ex vivo* study was to evaluate the influence of different lightexposure times on the interfacial bond strength of DT Light Post (DT-Bisco Inc.) quartz fiber posts to experimentally weakened root dentin restored with Light Core (LC-Bisco Inc.) composite resin.

### **METHODS & MATERIALS**

#### **Tooth Selection and Preparation**

Sixty extracted noncarious human maxillary central incisors were stored in 0.1% thymol diluted in saline at 4°C, pH=7 and used within 3 months following extraction. The

crowns were sectioned transversally at the cementoenamel junction with a double-faced diamond disc (#6911H; Brasseler Dental Products, Savannah, GA, USA) at low speed under air/water spray coolant and discarded. Root length was standardized in 17 mm. Working length was established at 1 mm short of the apical foramen. Root canal preparation was performed according to the crown-down technique using #2 to #4 Gates Glidden drills (Union Broch, York, PA, USA) and rotary instruments (Profile .04/06 tapers; Dentsply/ Maillefer, Tulsa, OK, USA) incrementally up to the working length. The apical stop was enlarged up to a #50 file/.04 taper. During preparation, the canals were irrigated with 2 mL of 1% sodium hypochlorite (Dermus, Florianópolis, SC, Brazil) at each change of instrument. A final flush was performed with 2 mL of deionized water.

Thereafter, the root canals were dried with absorbent paper points (Dentsply/Maillefer) and filled with gutta-percha main cones and accessory points (Dentsply/Maillefer) and a resin-based endodontic sealer (AH Plus; Dentsply De Trey, Konstanz, USA) (Table 1). Root canal obturation was done according to Tagger's<sup>25</sup> hybrid technique. After vertical compaction and placement of provisional restorations (Citodur; Septodont, Switzerland), the roots were stored in 100% relative humidity at 37°C for 24 h.

# **Post Space Preparation**

After 24 h, the gutta-percha was removed from the root canal up to a depth of 12 mm using #3 to #4 Gates-Glidden drills (Union Broch) and heated digital compactors (Dentsply/Maillefer), maintaining at least 4 mm of filling material in the apical third. Fiber post (#2 DT Light Post; Bisco Inc., Schaumburg, IL, USA; coronal diameter = 1.8 mm and

apical diameter = 1.0 mm) fitting to the canal space was tested. Each post was sectioned at the coronal third perpendicularly to its long axis, 4 mm above the coronal border of the root using a doubled-faced diamond disc (#6911; Brasseler) at low speed under air/water spray coolant.

The specimens were randomly assigned to 4 groups (n=15). In the 3 experimental groups (G1, G2 and G3), the thickness of root canal dentin walls was reduced using high-speed diamond burs (#4137; Vortex Ind. e Comércio, São Paulo, SP, Brazil and KG 717; KG Sorensen, São Paulo, SP, Brazil) under air/water spray coolant, in such a way to produce a circumferential space of approximately 1.0 mm between the fiber post and the circumjacent dentin walls. In the control group (G4), the roots were not experimentally weakened.

#### **Composite Resin Reinforcement**

Prior to composite resin reinforcement, the canals of the flared roots were irrigated with 10 mL of deionized water and dried with absorbent paper points. Intracanal dentin was etched with phosphoric acid (Uni-Etch; Bisco Inc.) for 15 s, rinsed with deionized water for 30 s and gently dried with absorbent paper points. A 3-step total-etch adhesive system (All Bond 2; Bisco Inc.) was applied to the slightly moist dentin with disposable microbrush tips (3M/ESPE, St. Paul, MN, USA), according to the manufacturer's instructions. The material was photoactivated by positioning the tip of the light-curing unit (Curing Light 2500; CL, 3M/ESPE; 500 mW/cm<sup>2</sup> light intensity) at canal entrance for 20 s.

Product (manufacturer)	Composition	Batch #
AH Plus <sup>™</sup> (Dentsply DeTrey, Konstanz, Germany)	<i>AH Plus Paste A:</i> Bisphenol-A epoxy resin; Bisphenol-F epoxy resin; Calcium tungstate; Zirconium oxide; silica; iron oxide pigments	0603002042
	AH Plus Paste B: Dibenzyldiamine; aminoadamantane; Tricyclodecane-diamine; Zirconium oxide; silica; silicone oil.	
All-Bond 2 (Bisco Inc, Schaumburg, IL, USA)	<i>UNI-ETCH</i> : 32% Phosphoric acid, Benzalkonium Chloride and xanthum gum thickener	0600001033
	Primer A: NTG-GMA, acetone, ethanol and water	0600001076
	PrimerB: BPDM, acetone, ethanol and photoinitiator	0600001077
	<i>D/E Resin</i> : Bis-GMA, UDMA, HEMA, photoinitiator (CQ) e amine activator	0600000717
Ligtht-Core <sup>TM</sup> (Bisco Inc, Schaumburg, IL, USA)	Bis-GMA, Ethoxylated Bisphenol A Dimethacrylate, glass frit (>60%)	0600004829
<b>Duolink</b> (Bisco Inc, Schaumburg, IL, USA )	Base: Bis-GMA, TEGDMA, UDMA, glass filler	0600004680

**Table 1.** Materials used in the experimental procedures, with the respective manufacturer information, composition and batch number.

NTG-GMA, Na-N-tolylglycine glycidylmethacrylate; BPDM, Biphenyl dimethacrylate; Bis-GMA, bisphenol A diglycidyl methacrylate; UDMA, urethane dimethacrylate; HEMA, 2-Hydroxyethyl methacrylate; TEGDMA, Triethylene glycol dimethacrylate; CQ, camphorquinone.
For root reinforcement, each canal was bulk filled with a translucent composite resin (Light Core; Bisco Inc.) (Table 1). In each canal, a #2 DT Light Post (Bisco, Inc.) coated with a thin coat of petroleum jelly (Vimak, São Paulo, Brazil) was centrally inserted into the resin mass along the whole post space extension. After removal of resin excesses, the tip of the light-curing unit was placed over the post and the device was activated according to the light-exposure times established for each group: G1 = 40 s; G2 = 80 s and G3 = 120 s. After composite resin polymerization, the post was clamped with needle-nose pliers and removed from the canal.

# **Post Cementation**

The root canals of all groups (weakened/reinforced and control) were reprepared with the bur supplied with the kit of the post system used (#2 DT Light Post, Bisco Inc.), washed with 10 mL of deionized water and dried with absorbent paper points. After removal of the petroleum jelly coating, the posts were water rinsed and dried with a mild air stream. Primer B (AB, Bisco Inc.) was applied to post surface, gently air thinned and light cured for 10 s. The root canals were etched with phosphoric acid (Uni-Etch; Bisco Inc.), applied with disposable microbrush tips (3M/ESPE) and left for 15 s, rinsed with deionized water for 30 s and gently dried with absorbent paper points. Equal amounts of Duo Link<sup>™</sup> resin cement (Bisco Inc.) were mixed and the material was taken to the canal with a lentulo spiral. Each post was seated into the respective canal with gentle pressure, cement excesses were removed with a microbrush tip and the material was light

activated for 40 s. After 4 min, the specimens were placed in black light-proof receptacles and stored in 100% relative humidity at 37°C for 24 h.

# Push Out Test - Specimen Preparation, Post Dislodgment and Failure Pattern Analysis

The roots were individually taken to a precision cutting machine (Isomet 1000; Buehler, Lake Forest, IL, USA) with a water-cooled diamond saw (South Bay Technology, San Clement, CA, USA) rotating at 325 rpm with 75 g load and serially sectioned in a mesiodistal direction (perpendicular to post long axis). Approximately 1.0-mm-thick ( $\pm$  0.1 mm) slices were obtained from the coronal, middle and apical regions of the post/canal. One slice of each region was selected at 2-, 6- and 10-mm depths, respectively.

The cuts were fixed in a stainless steel base (Fig. 1) with a 2.5-mm-diameter central hole attached to the inferior portion of an Instron machine (Model 4444; Instron, Canton, MA, USA). The root canal containing the DT Light Post was positioned in the same direction of the metallic base hole with its coronal border turned downwards. A metallic shaft with a 0.6-mm active tip attached to the superior portion of the Instron machine running at a crosshead speed of 0.5 mm/min until post dislodgment.

The maximum stress was calculated from the recorded peak load divided by the computed surface. The force (F) required to displace the post was recorded in kN, transformed into N and divided by the post lateral area in  $mm^2$  (S<sub>L</sub>) to determine the bond strength (BS) in MPa, using the following equation: BS=F/S<sub>L</sub>.

To calculate the exact bonding surface, the tapered design of the posts with regard to the respective part of the post was considered. The height of each specimen was measured with digital caliper (Mitutoyo Messgerate GmbH, Neuss, Germany; accurate to 0.001 mm) and the bonding surface was calculated using the formula of a conical frustum following equation:  $S_L = \pi (R + r) \sqrt{h^2 + (R - r)^2}$ 

where SL is the post lateral area, R = post radius at the coronal portion; r = post radius at the apical portion; h = post height/thickness.



Figure 1: Specimen adapted to the Instron machine device at the moment of the push out test. A: Instron machine support; B: Metallic base; C: 0.6-mm-diameter metallic post; D: Specimen.

For the fracture analysis, a careful visual examination was first performed at x4 magnification (Illuminated magnifying glass, Tokyo, Japan) and the debonded area was examined with a stereomicroscope at x20 to x40 magnifications (SZ60, Olympus Tokyo, Japan). Failure was considered: adhesive in the post - if resin/cement set was displaced from post; adhesive in the dentin - if the composite resin was displaced from dentin; mixed – when occurred a mixture of adhesive in dentine and post failure; cohesive in the resin - if the fracture occurred only in resin; and cohesive in dentin - if the fracture occurred in dentin. Failures modes were recorded as percentages.

Data were analyzed by using analysis of variance (ANOVA) with SPSS 13.0 for Windows (SPSS Inc, Chicago, IL, USA) statistical software. Post hoc tests were calculated using the Tukey's multiple comparison test at  $\alpha = 0.05$ .

#### RESULTS

Bond strength means and standard deviation displayed in Table 2. Two-way ANOVA showed that only the factor group was statistically significant (p<0.001). Neither the factor region nor the interaction between the factors group and region had significant influence of the results (p>0.05).

Comparison of all groups (Table 2) showed that the groups with root reinforcement had statistically similar (G2) or significantly higher (G1 and G3) bond strength means compared to the control group (G4).

C**	Pos	T-4-1		
Group**	Coronal	Middle	Apical	lotai
G1	10.39 (3.41) <sup>ABC</sup>	11.15 (2.89) <sup>AB</sup>	9.54 (2.61) <sup>ABC</sup>	10.36 (2.99) <sup>a</sup>
G2	8.91 (2.37) <sup>ABC</sup>	8.75 (2.76) <sup>ABC</sup>	9.44 (3.04) <sup>ABC</sup>	9.03 (2.69) <sup>ab</sup>
G3	11.77 (2.90) <sup>A</sup>	9.13 (2.93) ABC	9.95 (3.24) <sup>ABC</sup>	10.28 (3.16) <sup>a</sup>
G4	8.35 (2.52) <sup>BC</sup>	7.78 (2.28) <sup>C</sup>	7.679 (3.49) <sup>C</sup>	7.94 (2.78) <sup>b</sup>

**Table 2**. Bond strength means (MPa) and standard deviation for the different groups and post/canal regions.\*

\* Different superscript letter indicate statistically significant difference at 5%. \*\* G1, G2 and G3 are weakened/reinforced groups with different composite resin light-exposure times (40 s, 80 s and 120 s, respectively). G4 is the nonflared/non-reinforced control group.

Comparing the experimentally weakened/resin-reinforced groups (G1, G2 and G3), it was observed that the increase in composite light-exposure time did not result in statistically significant difference in bond strength (p>0.05). Likewise, when the post/canal regions (coronal, middle and apical) were compared to each other, the use of different light-exposure times had no statistically significant effect on bond strength (p>0.05).

Table 3 displays the failure modes observed in each group and post/canal region. There was a predominance of adhesive failures (in post or dentin) in the weakened/reinforced groups (85.18%) as well as in the control group (73.33%). In the resin-reinforced specimens, either the cement displaced completely from post (54.81%) or the post/cement/resin set displaced completely from dentin (30.37%). In control group, adhesive failures in post were the most frequent (68.89%) followed by mixed failures (26.66%). Cohesive failures predominated in the apical region and were observed only in weakened/reinforced groups.

Group		Adhesive	Adhesive	Mixed failure (%)	Cohesive	Cohesive
	Regions	failure/	failure/		failure/	failure/
		post (%)	dentin (%)		dentin (%)	resin (%)
G1 (40 s)	Coronal	66.67	20.00	_	13.33	_
	Middle	66.67	26.66	_	6.67	_
	Apical	60.00	_	_	33.33	6.67
G2 (80 s)	Coronal	46.67	53.33	_	_	_
	Middle	60.00	40.00	_	_	_
	Apical	40.00	33.33	_	20.00	6.67
G3 (120 s)	Coronal	53.33	40.00	_	6.67	-
	Middle	53.33	40.00	_	6.67	_
	Apical	46.67	20.00	6.67	13.33	13.33
G4 (control)	Coronal	73.33	6.67	20.00	_	_
	Middle	66.67	_	33.33	_	-
	Apical	66.67	6.67	26.66	-	_

**Table 3.** Failure modes observed on the debonded specimens of the three experimental groups (n=15) and control group (n=15) after the push-out test.

## DISCUSSION

The use of composite resin for intracanal restoration of weakened roots prior to cementation of prefabricated, fiber or metallic posts has shown good results in the increase of root resistance to fracture.<sup>2,3</sup> However, there is little published data referring to the bond strength along the root canal in these cases.<sup>3</sup> The present study evaluated the regional bond strength of quartz fiber posts cemented to root canal dentin walls reinforced with a composite resin photoactivated with different light-exposure times.

In the present study, the push out test was performed with 1-mm thick serial root slices, which allowed applying the shearing force more uniformly towards the adhesive interface, with less interference of tensile forces.<sup>10,13</sup> The use of this methodology was particularly important because it permitted evaluating the efficacy of the composite resin reinforcement in a regional fashion, indicating precisely the sites where failures occurred. In addition, since the push out test is based on the application of extrusion shear forces, the confined root canal spaces may be more reliably simulated than in the conventional shear and microtensile strength tests.<sup>10,11</sup>

The findings of the present experiment showed that, regardless of the light-exposure time of the composite resin used for root reinforcement, bond strength means were either higher or similar to those obtained in the non-experimentally weakened control specimens. It was also observed that the post/canal coronal and middle regions had similar bond strength means to those of the apical region. Even in the control group, although there was a decrease in bond strength in the middle and apical regions compared to the coronal region, these differences were not significant statistically. These results do not agree with those of previous studies that evaluate the bond strength in different regions of cemented post (coronal, middle and apical) and observed lower bond strength in the middle and apical thirds.<sup>10,12-14</sup> These results might be due to the following events: easier application of the adhesive system to the artificially flared root dentin walls; adhesive system used; total light-curing time of resin reinforcement and post cementation; and materials used for root reinforcement (light transmitting post and translucent composite resin).

In the present study, experimental root weakening (root dentin flaring) might have facilitated the access to deeper areas of the post space, thus enhancing the adhesive procedures. It is known that non-uniformly adapted or incompletely polymerized resinbased material due to the difficult in accessing the root canal may interfere negatively with dentin bonding and compromise the longevity of the adhesive interface.<sup>26-28</sup>

Additionally, All Bond 2 three-step total-etch adhesive system, used prior to the application of Duo-Link resin cement, might have successfully hybridized the more apical root dentin, even in the control group. In deeper root canal regions, drying of the acid-etched dentin is more critical than in more coronal regions. Due to its acetone- and alcohol-based composition, this system can act well on moist or slightly wet dentin, with increased bond strength. Akgungor & Akkayan<sup>12</sup> investigated the influence of dentin bonding agents and polymerization modes on the bond strength between translucent fiber posts and three dentin regions within a post space and found similar results in all regions when a self-etching adhesive system was used. These authors suggest that it is likely that the shortcomings inherent to bond strength in deeper regions may be attributed not only to the

access difficulties, but also to the chemical composition and technique of application of the tested adhesives systems.

The light-exposure time did not influence the bond strength in the post/resinreinforced groups. However, it should be mentioned that, when the DT Light Post was cemented to the canal, the dual resin cement was photoactivated for additional 40 s in order to control its setting time and thus facilitate specimen handling. As a result, in these groups, the total light-exposure time ranged from 80 s to 160 s, which is a considerably longer time than that recommended by the manufacturer. It may explain, in part, the similar bond strength recorded within the post space (depths of 2, 6 and 10 mm), regardless of the time of light exposure.

The light-activated composite resin used for root reinforcement (Light Core, Bisco, Inc.) might also have influenced the results because, according to the manufacturer, its translucency provides a curing depth of up to 5 mm, which is considerably greater than that recommended for photoactivation of non-translucent resins (2-mm-thick increments).

The cementation of light-transmitting fiber posts simultaneously with composite resin reinforcement (single-step technique) seems to be easier and faster that the two-step root reinforcement technique used in the present study, whereby the adhesive system applied to dentin was light activated before composite resin placement and fiber post cementation. A previous scanning electron microscopic study<sup>26</sup> found that simultaneous light curing of the adhesive system to dentin during fiber post cementation may produce a thinner hybrid layer and less resin tag formation, especially in the apical post space region. Other authors have stated that resin cement applied to root canal dentin.<sup>12</sup>

The cavity configuration factor (C-factor) is the ratio of the bonded surface area in a cavity to the unbonded surface area.<sup>8</sup> It is know that when a cavity to be restored has more free/unbonded surfaces, there is a higher material flowing and lesser internal stress incidence during the curing process.<sup>8-13</sup> These forces, resulting from polymerization shrinkage, pull the bonded restoration away from the dentin walls and cause material dislodgment. During polymerization, unbonded surfaces can move and flow, thereby relieving shrinkage stresses. However, as the unbonded surface area decreases, like in a long narrow root canal, there is insufficient stress relief and a high likelihood of debonding of one or more bonded areas<sup>8,13,29</sup> Therefore, when the C factor is associated with a resin cement thickness of up to 150 µm around the post, for example, it may result in a considerably higher bond strength than the C factor observed in occlusal class I composite resin restorations.<sup>13,30</sup> In addition, Moreira et al.<sup>29</sup> reported that cavities with a greater C factor restored with composite resin presented a larger number of interfacial failures (gaps). It may be assumed that, in the present study, post cementation subsequently to composite resin reinforcement of root structure decreased the bonded-to-debonded surface ratio during resin photoactivation, which might have provided a higher material flow and, consequently, lower composite polymerization shrinkage. It might have resulted in lesser gap formation and consequently higher bond strength to dentin. This rationale may also explain the higher bond strength observed in the weakened/reinforced groups compared to the control group as well as the fact that there was a higher percentage of adhesive failures between the resin material and the post than between the resin material and the dentin walls.

The analysis of the failure modes after the push out test (Table 3) showed a higher percentage of adhesive and mixed failures between the post and the cement mainly in the control group. In the weakened/resin-reinforced groups, there were adhesive failures between the resin and the dentin (30.37%), but still in a lower percentage than that of the adhesive failures in the post (54.81%). Furthermore, some specimens presented dentin fracture, mainly in the apical third. This seems to suggest that, although composite resin root reinforcement provides similar or superior bond strength to that recorded for non-experimentally weakened teeth, it does not guarantee that the reinforced dentin would have the same resistance as that of the original dentin with greater thickness.

It may be stated that the dissipation of tensions produced by root reinforcement with composite resin, which is elastically compatible with the dentinal structure<sup>3</sup>, did not avoid that the dentin in the experimental groups fractured more than the dentin of the control group. Likewise, it has been advocated that, although the restoration of weakened roots with light-activated composite resin increases their fracture strength, it cannot be categorically stated that the original condition of the removed dentin is reestablished.<sup>2,3</sup>

The findings of the present study are not definitive and cannot be directly correlated with those of other materials due to the structural differences existing among the diverse types of composites and posts. Moreover, the study outcomes were somewhat limited because aging cyclic loads were not incorporated to the methodology, which could determine whether the adhesive resistance effect is lasting or transitory. Further research should improve the adhesive process in order to provide a more reliable and long-lasting adhesive interface, especially in severely weakened teeth requiring root reinforcement.

# CONCLUSIONS

Within the limitations of an *ex vivo* study, the following conclusion may be drawn:

1. In the flared/reinforced groups, the use of longer light-exposure times (80 and 120 s) than that used as control (40s) did not provide increased bond strength between the fiber post and the resin-reinforced dentin, regardless of the analyzed region (coronal, middle and apical).

2. Overall, the reinforcement of weakened roots with light transmitting post and translucent composite resin restoration produced bond strength between the post and root canal dentin similar or superior to that of the control group (non-flared/non-reinforced roots), mainly at the coronal and middle regions.

3. Adhesive failures in the post occurred more frequently than mixed failures or adhesive failures in the dentin. Cohesive failures in the dentin or resin occurred only in the weakened/resin-reinforced specimens.

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# **CAPÍTULO 3**

# Interfacial evaluation of experimentally weakened roots restored with

adhesive materials and fibre posts. An SEM analysis \*

# Artigo submetido à revista:

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# Interfacial evaluation of experimentally weakened roots restored with adhesive materials and fibre posts. An SEM analysis.

Short Title: Interfacial evaluation after root reinforcement

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# Interfacial evaluation of experimentally weakened roots restored with adhesive materials and fibre posts. An SEM analysis.

# SUMMARY

*Objectives:* To evaluate the bonding interface in experimentally weakened roots reinforced with adhesive restorative materials and quartz fibre posts, varying the light-exposure time of the composite resin used for root reinforcement.

*Methods:* Twelve extracted human maxillary incisors teeth were used. The crowns were removed and the roots were endodontically treated. After post space preparation, the roots were assigned to four groups. The thickness of the root dentine was reduced and adhesively restored with composite resin light-activated through a translucent fibre post for either: 40 s (group 1), 80 s (group 2) or 120 s (group 3). In the case of control (group 4), the roots were not weakened. One day after post cementation, the specimens were sectioned transversally in three slices and processed for scanning electron microscopic analysis to observe bonding interface formation, quality of the hybrid layer and density of resin tags using a four-step scale method.

*Results:* Formation of a hybrid layer and resin tags were evident in all groups. There was no statistically (p > 0.05) significant difference between the regions analysed in each group (Friedman test) and between groups in each section depth (Kruskal-Wallis test). Furthermore, comparison of the flared/reinforced groups showed that the different times used for composite resin cure did not affect significantly the results (Kruskal-Wallis test, p = 0.2139).

*Conclusions:* Different light-exposure times used for composite resin polymerisation during root canal reinforcement did not affect significantly the formation and quality of the dentine/adhesive/composite resin bonding interface.

**Keywords:** bonding, fibre post, hybrid layer, light-exposure time, root reinforcement, scanning electron microscopy.

# 1. Introduction

Endodontically treated teeth may require extensive coronal reconstruction and, depending on the severity of coronal tissue loss, intracanal post placement may be necessary to achieve retention to the core and restoration.<sup>1</sup> Furthermore, severely damaged teeth may require previous reinforcement of their weakened root structure, which should be performed preferably with materials that have a modulus of elasticity similar to that of dentine.<sup>2</sup>

Because of their stiffness, metallic and ceramic materials transmit greater stresses during the masticatory efforts, which may predispose the teeth to irreversible root fractures, mainly when a great amount of intracanal dentine has been lost due to caries, excessively hard tissue removal or restorations.<sup>2-4</sup> In these cases, the root dentine may be reinforced with adhesive materials, such as composite resins or glass ionomer cements, prior to the post luting procedure.<sup>5-7</sup>

The success of the root dentine adhesive restorative technique is directly associated with the quality and uniformity of the resin-dentine interdiffusion zone (hybrid layer), resin tags and adhesive lateral branches produced upon infiltration of the adhesive system within the demineralised dentine substrate as well as the formation of a gap-free interface between the resin material and the canal walls.<sup>8-12</sup> In recent years, optical, electron and transmission microscopic studies have provided a comprehensive analysis of the bonding interface not only of coronal dentine, but also of root dentine, when interacting with adhesive systems, resin-based materials and intracanal posts.<sup>10-17</sup>

Despite the advances in intra-radicular reinforcement materials and techniques, the apical areas of the post preparation into the root canal continue to represent a challenge in terms of the bonding protocol and pose additional difficulties with regard to the insertion and photoactivation of adhesive restorative systems.<sup>18,19</sup> Furthermore, failure to obtain a gap-free and adequately cured composite resin layer within the canal space also contributes to producing a less durable bonding interface and lower bond strengths.<sup>20,21</sup>

Translucent fibre posts should allow light to be transmitted into the root canal, optimizing the photoactivation of the resin-based materials used for root reinforcement and post cementation.<sup>22,23</sup> In addition to their high levels of fatigue and tensile strength, these fibre posts have an elasticity modulus closer to that of dentine than metallic posts.<sup>24</sup> Their chemical nature is compatible with the Bis-GMA resin matrix, present in the composition of most adhesive systems and other resin-based materials. Ideally, adhesively luted intracanal fibre posts should form a single unit with the adhesive-infiltrated root dentine, the composite resin and the luting resin cement, yielding a higher flexural strength of the root after reinforcement. Therefore, the forces generated during mastication can be transmitted through the post with less stress accumulation, reducing the risk of root dentine fractures.<sup>7,8,24</sup>

However, light transmitting posts alone may not be sufficient to allow adequate composite resin polymerisation within the canal space.<sup>25</sup> It has been reported that the decrease in energy density in regions more distant from the light source may be compensated for by increasing the exposure time in order to generate the amount of energy necessary for an adequate polymerisation.<sup>26,27</sup> On the other hand, longer light-exposure

times may produce higher polymerisation shrinkage and compromise the quality of the resin-dentine interface.<sup>28</sup>

Therefore, the aim of this study was to evaluate, using scanning electron microscopy (SEM) the bonding interface produced at the flared root canal dentine after adhesive system application, composite resin reinforcement and intracanal post cementation. The null hypothesis is that the use of different light-exposure times for composite resin polymerisation during root canal reinforcement does not affect significantly the quality of the dentine/adhesive/composite resin bonding interface.

# 2. Material and methods

### 2.1 *Tooth Selection and Preparation*

Twelve extracted non-carious human maxillary central incisors were stored in 0.1% thymol diluted with saline at 4°C, pH=7 and used within 1 to 3 months following extraction. The crowns were sectioned transversally at the cementoenamel junction with a double-faced diamond disc No. 6911H (Brasseler Dental Products, Savannah, GA, USA) at low speed under air/water spray coolant and discarded. Root length was standardised at approximately 17 mm. After improving access to the root canal system with a diamond-coated bur, a step-down preparation was performed using Gates-Glidden drills No. 4, 3 and 2 (Dentsply Maillefer, Ballaigues, Switzerland). Subsequently, a size 10 K-file was introduced into each canal until it could be seen through the apical foramen and the length was measured. Working length was established by subtracting 1.0 mm from this length. Root canal preparation was performed according to the crown-down technique using a .04 taper

ProFile instruments (Dentsply Maillefer) up to master apical rotary (MAR) size 50. Sodium hypochlorite (1% NaOCl, 2 mL) was used to irrigate the canals after each filing using a 27-gauge irrigating needle. After completion of the canal preparation, the canals were rinsed with 10 mL of deionised water to remove any remnants of the irrigating solution.

The root canals were then dried with paper points (Dentsply/Maillefer) and filled with gutta-percha main cones and accessory points (Dentsply/Maillefer) and a resin-based endodontic sealer (AH Plus; Dentsply De Trey, Konstanz, USA) (Table 1). Root canal obturation was carried out according to Tagger's hybrid technique.<sup>29</sup> After vertical compaction and placement of provisional restorations (Citodur; Septodont, Switzerland), the roots were stored under conditions of 100% relative humidity and 37°C for 24 h.

# 2.2 Post Space Preparation

Twenty-four hours after root canal filling, the gutta-percha was removed from the root canal up to a depth of 12 mm using Gates-Glidden drills No. 4 and 3 (Dentsply/Maillefer) and heated digital compactors (Dentsply/Maillefer), maintaining 4 mm of filling material in the apical third.

The specimens were randomly assigned to 4 groups. In the 3 experimental groups (groups 1, 2 and 3), the thickness of the root canal dentine walls was reduced using high-speed diamond burs Vortex No. 4137 (Vortex Ind. e Comércio, São Paulo, SP, Brazil) and KG No. 717 (KG Sorensen, São Paulo, SP, Brazil) under air/water spray coolant, in such a way as to produce a circumferential space of approximately 1.0 mm between the fibre post DT Light Post size 2 (Bisco Inc., Schaumburg, IL, USA) and the circumjacent dentine

walls. Each post was sectioned at the coronal third perpendicularly to its long axis, 4 mm above the coronal border of the root using a doubled-faced diamond disc (Brasseler No. 911H) at low speed under air/water spray coolant. In the group 4 (control), the roots were not experimentally weakened.

# 2.3 Reinforcement with Adhesive Restorative Materials

Prior to composite resin reinforcement, the canals of the flared roots were irrigated with 10 mL of deionised water and dried with absorbent paper points. The intracanal dentine was etched with 32% phosphoric acid (Uni-Etch; Bisco Inc.) for 15 s, rinsed with deionised water for 30 s and gently dried with absorbent paper points (Dentsply/Maillefer). A 3-step "etch-and-rinse" adhesive system (All Bond 2; Bisco Inc.) was applied to the slightly moist dentine with disposable microbrush tips (3M/ESPE, St. Paul, MN, USA), according to the manufacturer's instructions. The material was photoactivated by positioning the tip of the light-curing unit (Curing Light 2500; 3M/ESPE; 500 mW/cm<sup>2</sup> light intensity) at the canal entrance for 20 s.

For root reinforcement, each canal was bulk filled with a translucent composite resin (Light Core; Bisco Inc.) using digital compactors (Dentsply/Maillefer). In each canal, a fibre post (DT Light Post; Bisco Inc.) coated with a thin coat of petroleum jelly (Vimak, São Paulo, SP, Brazil) was centrally inserted into the resin mass along the whole post space extension. After removal of the excess resin, the tip of the light-curing unit was placed over the post and the device was activated according to the light-exposure times established for each group: 40 s (group 1); 80 s (group 2) and 120 s (group 3). After composite resin polymerisation, the post was clamped with needle-nose pliers and removed from the canal.

The restored (weakened/reinforced groups) and non-restored (non-flared/nonreinforced group) root canal walls of each specimen were enlarged with low-speed drills supplied by the fibre post manufacturer (DT Light Post System; Bisco Inc.) to a depth of 12 mm from the coronal border of the root. After removal of the petroleum jelly coating, the posts were rinsed with water and dried in a mild air stream. Primer B (AB, Bisco Inc.) was applied to the post surface, gently air thinned and light cured for 10 s. The post spaces were etched with 32% phosphoric acid (Bisco) for 15 s, washed with water for 30 s and gently dried with paper points. Two consecutive drops of primer B were applied and the excess was removed with absorbent paper points. Equal amounts of Duo Link<sup>™</sup> Composite Luting Cement (Bisco Inc.) were mixed and the material was applied into the root canal space by means of a lentulo and onto the post surface. The fibre posts were then seated, excess cement was removed with a microbrush tip and the material was light activated for 40 s. After 4 min, the specimens were stored under conditions of 100% humidity in black lightproof containers for 24 h at 37°C.

# 2.4 Specimen Preparation for Scanning Electron Microscopy Analysis

The root specimens were individually placed on a precision cutting machine (Isomet 1000; Buehler, Lake Forest, IL, USA) with a water-cooled diamond saw (South Bay Technology, San Clement, CA, USA) and sectioned perpendicular to the long axis of the root. Three slices of each specimen were obtained, representing the coronal, middle and apical regions of the post space preparation (one slice per region), at depths of 2, 6 and 10 mm, respectively, from the coronal border of the root. The root sections (nine per group) were fixed in 2.5% glutaraldehyde (Merck KGaA, Darmstadt, Germany) buffered with 0.1 M sodium cacodylate buffer at pH 7.4 for 12 hours at 4°C. After fixation, the sections were rinsed with 20 mL of 0.1 M sodium cacodylate buffer at pH 7.4 for 1 hour with three changes, followed by distilled water for 1 minute. They were then sequentially dehydrated in ascending grades of ethanol (25°, 50°, 75° and 95° for 20 min/each, and 100° for 60 min), and transferred to hexamethyldisilizane (HMDS, Ted Pella, Redding, CA, USA) for 10 min. The root sections were embedded in epoxy resin (Epo-Thin, Buehler, Lake Bluff, Ill, USA) and wet ground in a polishing machine until complete exposure of the resin/cement/post interfaces, and then polished with wet silicon carbide paper of decreasing abrasiveness (up to 1200 grit) and 1.0 and 0.3  $\mu$ m alumina polishing pastes. After rinsing in running water and 10-min ultrasonication with deionised water, the samples were demineralised in 6N HCl for 30 s and deproteinised in 2% NaOCl for 10 min to observe the hybrid layer.

All samples were dried, mounted on aluminium stubs (Ted Pella), placed in a vacuum chamber and sputter-coated with a gold layer of approximately 300 Å (Bal-Tec SCD 005, Bal-Tec Co., USA). They were observed under a field-emission scanning electron microscope (Phillips XL30 FEG, Philips, Eindhoven, Netherlands) operating at 10.0 to 20.0 kV.

# 2.5 SEM Analysis

The qualitative analysis of the bonding interfaces addressed the following characteristics: 1) formation and uniformity of the hybrid layer; 2) adhesive layer thickness; and 3) dentine/adhesive/resin and post/resin cement interfaces. In addition, resin tag formation and morphology and its relation with the intratubular dentine were analysed quantitatively using a four-step scale method. The interface of the adhesive system with the demineralised intracanal dentine and hybrid layer formation were analysed using secondary electron (SE), back scattering electron (BSE) or simultaneous SE and BSE (MIX) imaging modes. SEM micrographs with higher standardized magnifications of representative areas of these interactions were taken to illustrate the results obtained at the three root regions (coronal, middle and apical) in each group.

For quantitative evaluation of the formation, morphology and interaction of the resin tags, SEM micrographs ( $\times 100$ ,  $\times 250$  and  $\times 500$  magnifications) were taken from four standardised areas of each root section (Figure 1).



**Fig. 1** Areas of the resin-dentine interface analysed. Four distinct standardised areas (point 1-4 of the sample) were examined using SEM. R = composite resin, De = dentine, P = post.

A four-step (0 to 3) scale method was established for each evaluated condition, according to a modified Ferrari *et al.*<sup>9</sup> criteria (Figure 2): a score of 0 was assigned when no resin tag formation was detected; a score of 1 was assigned when few and short resin tags were formed; a score of 2 was assigned when long resin tags were visible, with a few lateral branches; a score of 3 was assigned when long, dense resin tags with numerous lateral branches were evident.



**Fig. 2** Description of the four-step scale method. (a) A score of 0 was assigned when no resin tag formation was detected ( $\times 250$ ). (b) A score of 1 was assigned when few and short resin tags were formed ( $\times 500$ ). (c) A score of 2 was assigned when long resin tags were visible, with a few lateral branches ( $\times 500$ ). (d) A score of 3 was assigned when long, dense resin tags with numerous lateral branches were evident ( $\times 500$ ).

Four points were examined at each root section (at 2, 6 and 10 mm levels, corresponding to the coronal, middle and apical regions), totalling 12 per root and 36 evaluations per group. SEM evaluation was performed double blind by two different operators. In case of discrepancy between the two readers, the lower score was recorded.

The scores assigned to the resin tags observed at the three depths were analysed with the Kruskal-Wallis test to detect significant intergroup differences. The intragroup analysis was performed with the Friedman test. The level of statistical significance was set at p = 0.05.

#### 3. Results

The results of the qualitative and quantitative data analyses are given in Figures 3-7 and Tables 1-2. The centralisation and uniform circular aspect of the canal space after artificial intracanal dentine flaring to simulate root weakening could be observed (Figure 3).

The number of samples showing gaps/voids at the interface between resin materials and root walls or fibre post is summarized in Table 1. Although bubbles/voids were found within the composite resin reinforcement (Figure 3), formation of a hybrid layer, resin tags and adhesive lateral branches was observed in all regions analysed, both in the experimentally weakened groups (Figures 4-5) and in the non-flared/non-reinforced control group (Figure 7). In general, the adhesive layer was thicker in the apical region (Figure 5) compared to the coronal and middle regions (Figure 4). The thickness of the hybrid layer was not affected by location in the root canal independently of the analysed group.

Groups	Interface				
	Adhesive/composite resin	Post/resin cement	Total		
1 (40s)	2	-	2		
2 (80s)	2	-	2		
3 (120s)	2	1	3		
4 (control)	-	3	3		
Total	6	4	10		

Table 1: Number of the samples within the presence of gaps/voids was noted:

Table 2. Mean resin tag formation scores recorded at depths of 2, 6 and 10 mm in each group\*

Group	2 mm (coronal region)	6 mm (middle region)	10 mm (apical region)
Group 1 40 s	3.00	3.00	2.75
Group 2 80 s	2.83	2.75	2.58
Group 3 120 s	2.83	2.91	2.67
Group 4 Control	2.91	2.75	2.75

\* The application of the non parametric Kruskal-Wallis (unpaired intergroup analysis) and Friedman (paired intragroup analysis) tests showed statistical similarity of the results at the 5% level.

Although the 3-step "etch-and-rinse" adhesive system used in the present study yielded hybrid layer formation, some areas with interfacial gaps/voids were evident

(Figures 6-7). In the artificially flared/reinforced groups, interfacial gaps were more frequent (71,4%) at the top of the hybrid layer (Figure 6b). Additionally, the presence of a thinner adhesive layer over the hybrid layer was frequently associated with interfacial gap/void (66,6%).



**Fig. 3** Representative SEM micrographs of the regions in flared-reinforced groups. (a) Coronal region (×17). (b) Middle region (×19). (c) Apical region (×20). The centralisation and uniform circular aspect of the canal space after artificial flaring of the intracanal dentine to simulate root weakening can be observed. Bubbles were evident within the composite resin in all regions analysed (arrows). P = post; R = composite resin; De = dentine.



**Fig. 4** SEM micrographs of the coronal (a, c and e) and middle (b, d and f) regions of specimens in group 1 - composite resin light-activated for 40 s. The specimens light-activated for 80 s (group 2) or 120 s (group 3) had similar results. (a) and (b) Note the formation of long and numerous resin tags throughout the extension of the resin-dentine interface (×100). (c) Higher magnification showing the formation of a thick hybrid layer bonded to the composite resin (×2000) and some points of interfacial failures (arrows). (e): Close-up view (×8000) of the area within the dark square in Figure 4(c) showing in detail

the presence of adhesive lateral branches. Note that the resin tags show a very rough surface morphology (peritubular hybridisation). (d) In the middle region, in addition to the features described for the coronal region, the presence of a porous hybrid layer was observed, indicating hybridisation with the subjacent dentine (×2000). Note that the resindentine interface has no gaps. (f) Detail of another specimen of the middle region (×152) showing interfacial adaptation and absence of gaps between the post, cement and composite resin. Cross-section of the post showing the circular aspect of the quartz fibres (clear areas) wrapped in the resin matrix (dark areas). De = dentine; HI = hybrid layer; Lb = lateral branch; P = post; R = composite resin; RC = resin cement; T = resin tag(s).

In the control group, the gaps/voids were observed only at the interface between the post and the resin cement (Figure 7). A thinner resin cement layer was observed in a few areas (Figure 7b).

The data referring to resin tag morphology and density are summarised in Table 2. The statistical analysis ( $\alpha = 0.05$ , Kruskal-Wallis Test) showed that the light-exposure time did not affect significantly the resin tag morphology and density at the bonding interface in the experimental groups (p = 0.2190). Furthermore, there was no statistically (p > 0.05) significant difference between the regions analysed in each group (Friedman Test) and between groups in each section depth (Kruskal-Wallis Test). Although the apical part of the post restoration had a lower density of resin tags than the other regions, there was no statistically difference and all specimens exhibited long tags apparently well hybridised with the intratubular dentine (Figure 5).



**Fig. 5** SEM micrographs of the apical region of specimens in group 1 – composite resin light-activated for 40 s. The specimens light-activated for 80 s (group 2) or 120 s (group 3) had similar results. (a) Note the long and numerous tags formed throughout the extension of the resin-dentine interface and a thick layer of adhesive resin (white arrows). The asterisks point to regions with a thin adhesive layer, and presence of interfacial gaps (×100). (b) General view of specimen, at ×50 magnification, showing a resin cement layer between the post and the composite resin. Note the presence of bubbles within the composite resin (black arrows). (c) Close-up view (×1000) of the area within the dark square in Figure 5(a) showing the resin-dentine interface with no gaps, formation of a uniform hybrid layer and long and numerous tags adhered to the hybrid layer. (d) Higher magnification (× 2000) of the interface of another specimen, with presence of thick adhesive layer, resin tags and adhesive lateral branches. Ad = adhesive resin; De = dentine; HI = hybrid layer; Lb = lateral branch; P = post; R = composite resin; RC = resin cement; T= resin tags.



**Fig. 6** Representative SEM micrographs of an interfacial failure. Middle region of a specimen in group 3 – composite resin light-activated for 120 s. (a) Presence of a wide area of separation (arrows) between the composite resin and the resin-dentine interdiffusion zone (×100). (b) Close-up view (×2000) of area within the dark square in Figure 6(a) showing the occurrence of interfacial failure between the top of the hybrid layer and the composite resin. Note the absence of an adhesive layer covering the hybrid layer. The debonded area shows fractured resin tags. Ad = adhesive resin; De = dentine; HI = hybrid layer; R = composite resin; T= resin tags.

# 4. Discussion

Scanning electron microscopy studies have demonstrated that the bonding mechanism of the adhesive systems in root canals is essentially of a micromechanical nature, based on the demineralisation and resin infiltration of the dentine substrate, forming a hybrid layer, resin tags and adhesive lateral branches.<sup>8,14,21</sup> Moreover, a non-uniform hybridisation and formation of none or a few and short resin tags in deeper regions of the root canal have been observed.<sup>8,10,30</sup> In the present study, the adhesive system was able to infiltrate the etched dentine, forming a uniform hybrid layer and several long, funnel-shaped resin tags
along the entire post space. These results corroborated, in part, those of previous studies that used the same adhesive system (All-Bond 2) and also reported the formation of long and numerous tags, but only in the coronal and middle third of the root dentine.<sup>9,13,14</sup>



**Fig. 7** SEM micrographs of specimens in control group. (a) General view of specimen of apical region, at  $\times$ 57 magnification. Note the resin-dentine interface and formation of long and numerous resin tags. (b) Close-up view ( $\times$ 500) of area within the dark square in Figure 7(a). At some points of the post/cement/dentine interface, the resin cement layer was thinner and at others it was almost absent (arrows). Note the presence of interfacial failure between the post and the resin cement (asterisk). (c) In the coronal region ( $\times$ 1000), although the resin cement layer was thicker, the occurrence of interfacial failure between the post and the resin cement was common (arrows). D: Close-up view ( $\times$ 4000) of area within the dark square in Figure 7(c). The interface between the resin cement and the adhesive (hybrid layer) was intact and gap-free. Ad = adhesive resin; De = dentine; HI = hybrid layer; P = post; RC = resin cement; T= resin tags.

Other studies have also reported the absence of, or a significant decrease in, the number of resin tags formed, and a lower-quality hybridisation at the resin-dentine interfaces from the coronal to the apical regions of the prepared root canal space.<sup>8,11,12,30</sup> These observations were found to be correlated to the smaller number of dentinal tubules in the apical areas of the root canals.<sup>31,32</sup> Nevertheless, in the present study, there was resin tag formation in all groups, even in the apical region of the root restoration. Therefore, the lack of hybridisation quality and resin tag formation at resin-dentine interfaces might be better explained by technical difficulties related to the acid etching and adhesive system application in deeper regions of the root canal.<sup>9</sup> Additionally, it could be assumed that artificial root dentine flaring, performed in the experimental groups, would not only produce smoother and more regular canal walls, but also facilitate adhesive system application to deeper areas. However, the control specimens also presented similar adhesive features at the three regions/depths. This may be due to the fact that the greater enlargement of the root canal after endodontic and post space preparations facilitated the use of thin microbrush tips, which are able to reach all root dentine walls, and the application of the adhesive agent with a certain pressure, resulting in a deep diffusion of adhesive resin into the dentinal tubules.<sup>8,14,20</sup>

Furthermore, in this study, the uniform resin tag formation may be attributed to the penetration capacity of the three-step etch-and-rinse adhesive system, All Bond 2, which contains biphenyl-dimethacrylate (BPDM) in the primer and HEMA in the bonding agent. BPDM features a double benzene ring structure that provides higher monomer conversion rates due to the greater availability of free radicals (two directional cross-linking). The result is a more consistent and complete curing reaction than those obtained with single

benzene ring primers commonly found in most other systems, as well as better molecular contact with the bonding surface. Most adhesive resins contain HEMA due to its wettability and affinity with dentine, producing a more acid-resistant dentine substrate after resin impregnation.<sup>33,34</sup> HEMA-containing adhesive systems offer increased wettability and hydrophilicity, which in turn can increase the bond strength of the adhesive resin through the infiltration of monomers into the tooth substrates.<sup>35</sup>

In the root canal reinforcement procedures, the use of adhesive restorative materials together with a resin cement luting the post, could allow less microleakage than conventional luting cements (e.g., zinc phosphate) due to the formation, ideally, of a bonded single unit<sup>36</sup> with a uniform hybrid layer at the dentine substrate.<sup>8</sup> Nevertheless, during the process of composite resin curing, polymerisation shrinkage creates stresses that may be of sufficient magnitude to result in detachment of the adhesive resin from the underlying hybrid layer, thus producing an accessory, and an even larger entrance route for microleakage.<sup>37</sup>

In the present study, the three root canal regions exhibited statistically similar results with respect to the quality and formation of the bonding interfaces, independently of the light-exposure time used to cure the composite resin. These results are to be expected given the technical procedures used for the root canal reinforcement, and were mostly not affected by the additional step of luting the fibre post.

The cementation of light-transmitting fibre posts simultaneously with composite resin reinforcement (single-step technique) seems to be easier and faster than the two-step root reinforcement technique used in the present study, whereby the adhesive system applied to dentine was light activated before composite resin placement and fibre post cementation. A previous scanning electron microscopic study<sup>8</sup> found that simultaneous light curing of the adhesive system to dentine during fibre post cementation may produce a thinner hybrid layer and less resin tag formation, especially in the apical post space region. Other authors have stated that resin cement application and post seating may damage the unpolymerised adhesive system layer applied to root canal dentine.<sup>38</sup>

The cavity configuration factor (C-factor) is the ratio of the bonded surface area in a cavity to the unbonded surface area.<sup>6</sup> It is known that when a cavity to be restored has more free/unbonded surfaces, there is a greater material flow and lower incidence of internal stress during the curing process.<sup>37</sup> These forces, resulting from polymerization shrinkage, pull the bonded restoration away from the dentine walls and cause material dislodgment. During polymerization, unbonded surfaces can move and flow, thereby relieving shrinkage stresses. However, as the unbonded surface area decreases, as in a long narrow root canal, there is insufficient stress relief and a high likelihood of debonding of one or more bonded areas.<sup>39</sup> In a recent study, Moreira da Silva et al.<sup>39</sup> reported that cavities with a greater C factor restored with composite resin presented a larger number of interfacial failures (gaps). It may be assumed that, in the present study, post cementation after composite resin reinforcement of the root structure decreased the bonded-to-debonded surface ratio during the resin photoactivation, which might have led to a higher material flow and, consequently, lower composite polymerization shrinkage. This may have resulted in less interfacial gap formation and may explain, in part, the absence of statistical differences between the flared and reinforced groups.

It is always possible that the interfacial gaps observed through SEM were created during the sample preparation. In this study, the SEM analysis was carried out on a direct view of the samples and since the specimens were placed in the vacuum chamber of the microscope the gaps/voids noted could be considered as artifacts. However, in the flared/reinforced groups of this study, no resin gap formation occurred when there was adhesive penetration into the dentinal tubules and formation of a thick adhesive layer above the hybrid layer. On the other hand, in areas where a hybrid layer was formed with long and numerous resin tags but with a thinner adhesive layer, the occurrence of failures between the adhesive resin and the top of hybrid layer were consistently observed (Figure 6).

According to Van Meerbeek *et al.*<sup>40</sup> these observations provide evidence for the concept of elastic bonding, in which a sufficiently thick and relatively elastic unfilled or semi-filled adhesive resin absorbs, in part, the polymerisation shrinkage stresses of the composite material by elastic elongation, preventing the interface from detaching. The hybrid layer may also be considered as part of this elastic complex, mainly due to its elasticity modulus, which is significantly lower than that of the surrounding tissues.<sup>41</sup> However, the weaker portion of the hybrid layer is its top surface, which is usually porous and may have a "ghost" hybrid layer or a "hybridoid" region, which is a misinfiltrated hybrid layer in which the adhesive did not envelop the collagen fibres exposed by acid etching.<sup>10</sup> Thus, it seems reasonable to assume that a more effective bond strength of the resin-dentine interdiffusion zone is expected when there is a relatively thick adhesive layer

protecting the hybrid layer before the placement of the resin cement or composite resin, as occurs with intracanal reinforcement.

The manufacturer of the adhesive system used in the present study (All-Bond 2) recommends the application of 4 to 5 drops of primer/adhesive on the acid-etched dentine. As this type of adhesive has a high penetration capacity, forming resin tags that reach more than 200 µm in length (Figure 4), if a small amount of material is applied no adhesive layer is expected over the hybrid layer. In this experiment, several areas exhibited a very thin or even absent adhesive layer, mainly within the coronal third. As the laboratory procedures were carried out always maintaining the root apex turned downwards, an adhesive flow towards the apical region was often observed. This may also explain why gap formation was observed in the coronal region of the flared/reinforced groups, regardless of the density and length of the tags formed. Moreover, in these groups, although small bubbles and voids were observed within the composite resin, the resin/cement/post interface was frequently uniform and gap-free. On the other hand, in the non-flared/non-reinforced control group failures occurred exclusively between the post and the resin cement.

In some non-experimentally weakened specimens, the resin cement layer was very thin or even absent. Although the teeth used in the present study (maxillary central incisors) have a more uniform intracanal configuration than other dental groups, anatomic variations of the root determine differences in the amount and three-dimensional distribution of the resin cement used for post cementation. Differences between root canal anatomy and post dimensions may explain the fact that, clinically, the weakest point in adhesively cemented post-core restorations is the interface between the fibre post and the resin-based cement.<sup>8,14,18,21,42</sup>

In this study, regardless of the density and length of the resin tags, the occurrence of interfacial gaps along the hybrid layer surface or the post-cement interface reflects the difficulty in obtaining a high quality bonding in the root canal space.<sup>21,42</sup> Difficulties in the insertion and photoactivation of the adhesive restorative materials in the root canal, as well as the type of cavity itself, which provides a high C-factor, may negatively affect the bond strength in deeper regions of the post space and remain a challenge to dental rehabilitation.<sup>21</sup>

The test method used in the present study, i.e. SEM analysis, has a number of limitations, mainly related to the quantification of gaps/voids. The evaluation of the formation of resin tags, and of their length, is another limited parameter. Future studies using Transmission Electron Microscopy (TEM) analysis would provide new information and more complete data.

## 5. Conclusions

Based on the qualitative and quantitative data obtained in this *ex vivo* study, it may be concluded that there was formation of a hybrid layer, resin tags and lateral branches in all regions analysed. The null-hypothesis tested in this study was confirmed. Different light-exposure times used for composite resin polymerisation during root canal reinforcement did not affect significantly the formation and quality of the dentine/adhesive/composite resin bonding interface.

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A restauração de dentes tratados endodonticamente deve priorizar, além da estética e função, a longevidade do elemento dental. Com este objetivo, quando a dentina radicular tiver pouca espessura remanescente, a restauração interna das paredes do canal com materiais resinosos pode proporcionar maior resistência estrutural e, dessa forma, diminuir o risco de fraturas após a cimentação de pinos e núcleos metálicos (GONÇALVES et al., 2006; WU et al., 2007) ou pinos de fibra (MACCARI et al., 2007). Porém, cuidados adicionais devem ser tomados na seleção dos materiais e das técnicas que serão utilizadas na execução dos procedimentos de reforço, de forma a proporcionar interface de união com melhores propriedades mecânicas e adequada resistência adesiva.

O grau de conversão da resina composta, a resistência de união e a qualidade de infiltração dos monômeros do sistema adesivo na dentina são fatores primordiais para que a interface formada após o reforço radicular possa ser confiável a curto e em longo prazo (NAKABAYASHI et al., 1982; ERICKSON, 1992; DE MUNCk et al., 2003). No presente estudo, buscou-se avaliar esses fatores mediante a realização de três experimentos: dureza da resina composta; resistência de união e interface adesiva entre pino, cimento, resina e dentina do canal após o reforço radicular.

Em relação à metodologia utilizada nesses experimentos, alguns aspectos merecem destaque para seu melhor entendimento, principalmente quanto à seleção dos materiais e aproveitamento dos espécimes dentais. A escolha dos materiais utilizados neste estudo buscou reduzir o número de variáveis que pudessem interferir ou mascarar os resultados. Como os objetivos principais foram avaliar a influência do tempo de fotoativação e a distância da unidade fotoativadora no reforço radicular com resina composta e pinos de fibra de quartzo, optou-se por padronizar todos os materiais utilizados nos diferentes grupos.

A seleção da resina e pino translúcidos teve como intuito maximizar a transmissão de luz ao longo do canal, diminuindo ao máximo a interferência do quesito cor nos resultados. Muitos estudos têm verificado que a cor, bem como o tamanho da partícula de carga, pode afetar a transmissão da luz no compósito, e que cores mais claras possibilitam melhores resultados do que cores mais escuras ou de maior opacidade (AGUIAR et al., 2005; LAZARCHIK et al., 2007). Além disso, mesmo a cor A1 (considerada como uma das mais claras da escala de cores) apresenta agentes opacificadores que interferem na passagem de luz. Em recente estudo, LAZARCHIK et al. (2007) verificaram que a resina de tonalidade translúcida não foi afetada pelo tipo de técnica de fotoativação utilizada, obtendo valores de dureza Knoop (HK) semelhantes aos observados na superfície de topo até a profundidade de 3 mm, independentemente de ter sido fotoativada em incrementos ou em bloco único. Por analogia, no presente estudo, supôs-se que a resina Light Core, por sua translucidez, pudesse ser utilizada para o reforço intracanal na técnica de incremento único.

A escolha do pino DT Light Post deveu-se à sua translucidez e capacidade de possibilitar a fotoativação de materiais resinosos (YOLDAS; ALAÇAN, 2005; FARIA E SILVA et al., 2007), bem como sua resistência flexural e módulo de elasticidade compatível com a dentina (SADEK et al., 2006; PERDIGÃO et al., 2006). É importante ressaltar que estudos comparativos com outros materiais devem ser realizados, a fim de comprovar a suposta superioridade destes materiais, resina translúcida e pino de quartzo fototransmissor, antes de indicá-los como melhor escolha para os procedimentos de reforço radicular.

Quanto ao aproveitamento dos espécimes, um dos aspectos positivos da metodologia empregada neste estudo foi à possibilidade de uso do mesmo elemento dental para a confecção de vários corpos-de-prova, por meio de secções transversais da raiz, para os diferentes experimentos. Estes corpos-de-prova foram divididos uniformemente entre os experimentos de dureza e resistência de união ao cisalhamento por extrusão, *push-out*, o que proporcionou a avaliação local de acordo com a profundidade de fotoativação e região do reforço intracanal. Além disso, permitiu que os mesmos corpos-de-prova selecionados para o teste de dureza fossem também utilizados na análise da interface adesiva. Como vantagem adicional, foi possível realizar comparações de dados e correlacionar os resultados obtidos nos três experimentos. Deve-se salientar que, em função da dificuldade de obtenção de dentes de humanos, o máximo aproveitamento de cada espécime,

propiciado por esta metodologia, deve servir de exemplo para a realização de outros experimentos.

O primeiro experimento deste estudo teve por objetivo avaliar a influência do tempo de fotoativação na dureza da resina composta utilizada no reforço radicular, nas diferentes regiões do preparo do canal e com relação à distância lateral do pino de quartzo fototransmissor (DT Light Post). Considerando que o aumento da distância seccional com relação à fonte de luz causa diminuição da quantidade de energia recebida, e que esta diminuição pode ser compensada pelo aumento do tempo de irradiação (CORRER-SOBRINHO et al., 2000; YAP et al., 2000), tempos adicionais de fotoativação foram propostos, observando a dureza da resina composta como marcador indireto do grau de conversão monomérica obtido pelo material, em todas as regiões e distâncias analisadas.

Os resultados obtidos com o aumento do tempo de fotoativação, promovendo aumento da dureza da resina composta nas áreas mais distantes da fonte de irradiação, sugerem que seja preconizada a utilização de tempos adicionais para ativação do compósito utilizado na técnica do reforço radicular. Além disso, verificou-se que a dureza do material em regiões mais distantes da fonte fotoativadora foi, via de regra, maior do que 80% da dureza obtida próxima da fonte de luz em cada grupo. Esses resultados puderam ser explicados pelos materiais utilizados e pela fotoativação adicional de 40 s, quando da cimentação do pino no canal. Independentemente disso, a sugestão de tempos maiores de fotoativação pode ser embasada pelo fato de que, na região apical do reforço, o tempo de fotoativação da resina por 120 s mostrou-se superior aos demais. Como a dureza pode servir de parâmetro indireto na avaliação da polimerização da resina composta, pode-se dizer que quanto maior a dureza, maior o grau de conversão do compósito e melhor serão suas propriedades físico-químicas, resultando em maior estabilidade e longevidade do conjunto (VISVANATHAN et al. 2007).

Por outro lado, diferentemente do que ocorreu com a dureza da resina composta ao longo do canal, o tempo de fotoativação pareceu não influenciar os resultados de resistência de união alcançados nos grupos fragilizados. Na seleção dos corpos-de-prova que seriam utilizados no teste de dureza ou no teste de resistência de união, buscou-se, a princípio, avaliar a existência de possível correlação entre a dureza alcançada pela resina composta e a resistência de união observada na respectiva região. Com essa finalidade, teve-se o cuidado de testar a dureza do material na face adjacente ao espécime que seria utilizado para a avaliação da resistência adesiva na mesma região. De modo geral, observou-se que os valores médios de dureza dos grupos experimentais tiveram correlação positiva com os valores médios de resistência de união. Porém, ao analisarmos as regiões em grupo, não foi observada correlação significativa entre as variáveis. Novos experimentos devem ser realizados a fim de observar a correlação desses dados. Ainda em relação aos resultados de resistência de união regional após reforço de raízes fragilizadas, observou-se que, de modo geral, a resistência adesiva foi semelhante ou superior ao grupo controle, não fragilizado. Levando-se em conta apenas esses resultados, poder-se-ia imaginar que o elemento dental, após a fragilização e reforço, pudesse ficar mais resistente do que era antes da fragilização. Porém, na análise do tipo de falha observada após desunião, verificou-se que, diferentemente do grupo controle, em muitos espécimes dos grupos fragilizados ocorreu falha coesiva da dentina, principalmente na região apical do reforço. Isto pode significar que, mesmo após o reforço, as áreas finas de dentina podem absorver tensões e fraturar, com maior facilidade do que quando não fragilizadas.

No mesmo experimento, outro resultado interessante foi a não constatação de diferenças na resistência de união à dentina alcançada nas diferentes regiões do reforço intracanal, dentro de cada grupo. Mesmo na região apical do reforço, a resistência adesiva mostrou-se similar às observadas nas demais regiões analisadas, independentemente do grupo em questão. Esses resultados podem ser explicados pela análise interfacial realizada em MEV. No estudo da interface adesiva, as regiões observadas ao longo do canal radicular mostraram resultados semelhantes quanto aos aspectos qualitativos de hibridização da dentina, em todos os tempos de fotoativação empregados. Apesar da quantidade de *tags* de resina ter sido ligeiramente menor na região apical do preparo, não houve diferenças

significativas tanto na interface observada quanto na resistência de união com relação às demais regiões. Aparentemente, mais importante do que a quantidade de *tags* formados, a qualidade da hibridização dentinária foi determinante para a maior resistência adesiva da interface de união (PERDIGÃO et al., 2006). Vale ressaltar que o critério técnico e a seleção dos materiais utilizados no reforço intracanal podem ter sido igualmente importantes para o alcance desses resultados.

No entanto, deve-se ressaltar que, como em outros estudos laboratoriais, há um grande número de aspectos que devem ser considerados e que limitam maior abrangência dos resultados alcançados. Como por exemplo, pode-se citar o uso de apenas um tipo de resina composta e de pino. Seria interessante comparar os resultados obtidos com a utilização de outros materiais adesivos para o reforço, bem como a cimentação de outros tipos de pinos. Por outro lado, como não foram realizados tratamentos que simulassem o envelhecimento desses materiais, outros estudos devem ser realizados a fim de observar estes efeitos, *in vitro* e *in vivo*, após a técnica de reforço radicular sugerida.

Diante do exposto, é importante que novas pesquisas laboratoriais sejam realizadas e posteriormente complementadas por estudos clínicos, a fim de observar a qualidade da adesão e resistência de união entre os materiais utilizados na técnica do reforço radicular e, finalmente, possibilitar maior longevidade à reabilitação dental após tratamento endodôntico. Como conclusão final do presente estudo, observou-se que diferentes tempos de fotoativação, empregados no reforço com resina composta e pinos de fibra, influenciaram nos resultados de dureza da resina alcançados ao longo do canal, embora não tenham afetado significativamente a resistência de união e a interface adesiva observada. Finalmente, a região do reforço (profundidade e distância lateral do pino) afetou a dureza da resina composta, porém não influenciou significativamente na resistência adesiva interfacial.



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### **DETALHAMENTO DA METODOLOGIA**

#### 1- Seleção e preparo dos corpos-de-prova

Para a realização da fase experimental dessa tese, todos os procedimentos foram aprovados pelo Comitê de Ética em Pesquisa da Universidade de Ribeirão Preto (Anexo 2).

Incisivos centrais superiores de humanos foram radiografados no sentido vestíbulo-lingual e examinados cuidadosamente com lupa estereoscópica (aumento de 4X, Illuminated magnifying glass, Tokyo, Japan) com o objetivo de verificar a existência de canais únicos, retos, forame apical totalmente formado e ausência de trincas e imperfeições. Os dentes selecionados (n = 60) foram raspados com curetas periodontais SM 17/18 (Hu-Friedy, Rio de Janeiro, RJ, Brasil), limpos com jatos de bicarbonato ar/água (Jet-Sonic, Gnatus, Ribeirão Preto, SP, Brasil) - (Figura 1A) e armazenados na temperatura de 9°C, imersos em solução de timol 0,1% diluído em soro fisiológico 0,9%, pH=7, pelo período máximo de três meses.

A coroa de cada dente foi seccionada 1 mm acima da junção cementoesmalte, com o auxílio de um disco diamantado de dupla face (Brasseler Dental Products, Savannah, Ga, EUA) em baixa rotação (Figuras 1B e 1C) e sob spray ar/água, padronizando o comprimento das raízes em 17 mm (Figura 1D).



**Figura 1: Preparo e corte dos dentes para padronização das raízes. A)** Amostra de um incisivo central superior selecionado após raspagem e limpeza. **B)** e **C)** Corte do dente acima da junção cemento/esmalte. **D)** Padronização das raízes em aproximadamente 17 mm.

O comprimento do dente foi determinado pelo método direto introduzindose uma lima endodôntica flexofile #15 (Dentsply Maillefer, Tulsa, OK, EUA) no canal radicular até que sua ponta atingisse o forame apical. O comprimento de trabalho foi determinado como sendo de 1 mm aquém do comprimento do dente. O preparo endodôntico foi realizado pela técnica coroa-ápice, com o uso de brocas de Gates-Glidden (Union Broch, York, PA) #4 a #2 e de instrumentos rotatórios (Profile .04/06 taper, Dentisply Maillefer, Tulsa, OK, EUA) utilizados incrementalmente até o instrumento #50 .04/taper. Durante todo o preparo, os canais foram irrigados com 2 mL de solução de hipoclorito de sódio 1% (Dermus, Florianópolis, SC, Brazil) entre cada instrumento e, ao final, com 2mL de água deionizada.

Na seqüência, os canais foram secos com cones de papel (Dentsply Maillefer, Tulsa, OK, EUA) e obturados pela técnica híbrida de Tagger (Figura 2A) com a utilização de compactadores de guta percha #70 (Moyco Union Broach, York, Pennsylvania, EUA). Foram utilizados cones de guta-percha principais e acessórios (Dentsply Maillefer) e cimento AH PLus (Dentsply De Trey, Konstanz, EUA). Após a realização da compactação vertical e colocação de material restaurador provisório (Citodur, Septodont, Suíça), foi realizada radiografia para observação da qualidade do tratamento endodôntico (Figura 2B). As raízes foram mantidas em umidade relativa de 100% e em estufa a 37°C por 24h.

#### 1.1- Preparo dos espécimes para receber os pinos

Após 24 horas do término da obturação, os canais tiveram a guta-percha removida até uma profundidade de 12 mm do total da raiz (Figura 2D), com brocas de Gates-Glidden (# 3 e # 4), brocas específicas do sistema de pinos DT LIght Post (Figura 2,c, #2, Bisco Inc.) e compactadores digitais (Dentsply Maillefer, Tulsa, EUA) aquecidos ao rubro. Os 4 mm de material obturador no terço apical foram mantidos e comprovados por tomada radiográfica (Figura 2J). Cada pino foi provado no interior do canal (Figura 2F) e seccionado em sua porção coronal perpendicularmente ao seu longo eixo, 4 mm acima do bordo cervical da raiz. Para esta finalidade foi utilizado um disco de diamante de dupla-face (Brasseler Dental Products, Savannah, Ga, EUA) em baixa rotação e sob *spray* ar/água.



Figura 2: Obturação endodôntica, preparo para o pino e fragilização dos espécimes. A) Obturação pela técnica híbrida de Tagger. B) Radiografia para observação da qualidade da obturação. C) Broca de preparo do sistema DT Light Post. D) Broca calibrada em 12 mm. E) Preparo do canal após remoção da guta-percha. F) Verificação da adaptação do pino ao canal. G) Pontas diamantadas utilizadas na fragilização dos espécimes. H) e I) Comparação do diâmetro da entrada do canal, antes e após a fragilização. J) Amostras de imagens radiográficas obtidas após a remoção da guta-percha e preparo do canal (Grupo Controle, GC) e após o sobrepreparo/fragilização (Grupos Fragilizados, GF).

As raízes foram divididas aleatoriamente em 4 grupos (n = 15), um controle (não fragilizado) e 3 grupos experimentais (fragilizados). Nos grupos fragilizados, a espessura das paredes de dentina foi reduzida com o uso seqüencial de pontas diamantadas em alta rotação nº 4137 (Vortex Ind. e Comércio, São Paulo, SP, Brasil) e KG 717 (KG Sorensen, São Paulo, SP, Brasil) em peça reta, ambos sob *spray* ar/água, fazendo com que os pinos de fibra (DT Light Post #2), com diâmetro coronal de 1,8 mm e apical de 1,0 mm, ficassem com folga circunferencial de aproximadamente 1,0 mm com relação à estrutura dentinária circundante, conforme ilustra a FIGURA 2H-J.

#### 1.2- Reforço com resina composta

Os materiais utilizados nos procedimentos experimentais estão listados na TABELA 1 (fabricante, composição, modo de uso e lote).

Previamente ao reforço com resina composta, os canais foram irrigados com 10 mL de água deionizada e secos com pontas de papel absorvente. O espaço do pino foi condicionado com ácido fosfórico (UNI-ETCH, Bisco Inc., Schaumburg, IL, EUA) por 15 s, lavado com água deionizada e gentilmente seco com pontas de papel absorvente. O sistema adesivo All Bond 2 (Bisco Inc., Schaumburg, IL, EUA), um sistema convencional de três passos, foi aplicado com pincéis microbrushes finos (3M ESPE, Dental Products, St. Paul, Minn., EUA) à dentina ligeiramente úmida (Figura 3A-J) (Tabela 1). A fotoativação foi realizada posicionando a ponta do aparelho Curing Light 2500 (3M ESPE, St. Paul, MN) pelo tempo de 20 s na entrada do canal com intensidade de luz de pelo menos 550 mW/cm2. As raízes fragilizadas (n = 45) foram aleatoriamente divididas em três grupos experimentais (n = 15) de acordo com o tempo de polimerização da resina composta utilizada no reforço da raiz: Grupo 1, 40 s; Grupo 2, 80 s e Grupo 3, 120 s. **Tabela 1**: Materiais utilizados nos procedimentos experimentais, com os respectivos fabricantes, composição, modo de uso e lote de fabricação:

Produto	Composição	Modo de Uso	Lote
(Fabricante)			
AH Plus <sup>™</sup> (Dentsply DeTrey, Konstanz, Germany)	<i>AH Plus Paste A:</i> Bisphenol-A epoxy resin; Bisphenol-F epoxy resin; Calcium tungstate; Zirconium oxide; silica; iron oxide pigments. <i>AH Plus Paste B:</i> Dibenzyldiamine; aminoadamantane; Tricyclodecane-diamine; Zirconium oxide; silica; silicone oil.	Misture quantidades iguais da pasta A e pasta B por 5-10 s. Leve ao canal com o auxílio da espiral lentulo ou do cone principal de guta- percha. Realize a obturação do canal pela técnica escolhida.	0603002042
<b>All-Bond 2</b> (Bisco Inc, Schaumburg, Ill)	<i>UNI-ETCH</i> : 32% Phosphoric acid, Benzalkonium Chloride and xanthum gum thickener	Aplique o ácido por 15 s; lave com água por 20s. Seque levemente com ponta de papel.	0600001033
	Primer A: NTG-GMA, acetone, ethanol and water Primer B: BPDM, acetone, ethanol and photoinitiator	Misture o primer A e o primer B* e aplique 5 gotas (p/rest c/ resina) ou 2 gotas (para cimentação do pino). Seque por 5 a 6 s com jato de ar e com cones de papel removendo bem os excessos.	0600001076 0600001077
	<i>D/E Resin</i> : Bis-GMA, UDMA, HEMA, photoinitiator (CQ) and amine activator	Aplique fina camada de D/E Resin. Fotoative por 20 s.	060000717
Ligtht-Core <sup>™</sup> (Bisco Inc, Schaumburg, III)	Bis-GMA, Ethoxylated Bisphenol A Dimethacrylate, glass frit (>60%)	Aplique dentro do canal calcando a resina em camadas até preencher o conduto. Posicione o pino (previamente isolado com vaselina) centralmente no canal. Remova os excessos. Fotoative pelo tempo pré-determinado. Remova o pino com o auxílio de porta-agulha.	0600004829
<b>Duolink</b> (Bisco Inc, Schaumburg, Ill)	<i>Base:</i> Bis-GMA, TEGDMA, UDMA, glass filler	Misture quantidades iguais de base e catalisador (10 a 15 s). Leve o cimento dentro do canal (c/ seringa ou lentulo) e na superfície do pino. Posicione o pino no canal por 5-10 s. Remova os excessos e fotoative por 40s a partir do pino.	0600004680

*NTG-GMA,* Na-N-tolylglycine glycidylmethacrylate; BPDM, Biphenyl dimethacrylate; *Bis-GMA*, bisphenol A diglycidyl methacrylate; *UDMA*, urethane dimethacrylate; *HEMA*, 2-Hydroxyethyl methacrylate; *TEGDMA*, Triethylene glycol dimethacrylate; *CQ*, canforquinona.

\* Para prevenir a presa prematura do cimento quando estiver utilizando o ALL-Bond 2, aplique apenas o Primer B, tanto no canal, quanto no pino. Não fotoative antes da cimentação do pino.



**Figura 3**: **Metodologia de uso do sistema adesivo All Bond 2**. **A)** Ácido fosfórico 31% UNI-ECTH (Bisco). **B)** Aplicação do ácido fosfórico. **C)** Irrigação com água. **D)** Secagem do canal com cone de papel. **E)** *Primers* A e B (Bisco). **F)** Mistura do *Primer* A + *Primer* B. **G)** Aplicação das camadas de *primer* com pincel microbrushe. **H)** Adesivo D/E Resin (Bisco). **I)** Aplicação do adesivo com cone de papel. J) Remoção do excesso de adesivo com cone de papel.



Figura 4: Reforço intracanal com resina composta. A) Resina composta Light-Core (Bisco).
B) Inserção da resina dentro do canal radicular. C) Compactação da resina composta com o uso de calcadores digitais. D) Aplicação da camada de vaselina sólida sobre o pino DT Light Post. E) Adaptação do pino e remoção do excesso de resina com espátula. F) Fotoativação. G) Remoção do pino com auxílio de pinça hemostática. H) Espécime após o reforço. I) Cimento Duo Link usado na cimentação do pino. J) Imagem do pino com o cimento. K) Pino inserido centralmente no canal e remoção dos excessos para posterior fotoativação.

Para o reforço, cada canal foi preenchido com resina composta (Light Core, Bisco) com auxílio de compactadores (SSWhite, Rio de Janeiro, Brazil) e o pino, previamente isolado com fina camada de vaselina, sólida foi inserido centralmente em toda a extensão do preparo (Figura 4A-E).

Após a remoção dos excessos da resina, a ponta do fotopolimerizador foi posicionada sobre o pino e ativada (Figura 4F) de acordo com os tempos determinados para cada grupo, com intensidade de luz de pelo menos 550 mW/cm2. Após o término da fotoativação da resina, o pino foi removido do interior do canal com o auxílio de pinça hemostática (Figura 4G-H).

Em todos os grupos o espaço para a colocação do pino foi repreparado com a broca específica do sistema de pinos utilizado (#2, DT Light Post), lavado com 10mL de água deionizada e seco com ponta de papel absorvente. Após a remoção da vaselina, limpeza e secagem da superfície dos pinos, uma camada do *primer* B do sistema All-Bond 2 foi aplicada, secando o excesso com jato de ar e fotoativando por 10 s. O espaço protético foi condicionado com ácido fosfórico 32% (UNI-ETCH, Bisco Inc., Schaumburg, IL, EUA) por 15 s, com o auxílio de pincéis Microbrush (3M ESPE, Dental Products, St. Paul, Minn., EUA), lavado com água deionizada por 30 s e seco com pontas de papel. Na seqüência, foram aplicadas duas gotas do *primer* B, sendo os excessos removidos com pontas de papel absorvente. Quantidades iguais de base e catalisador do cimento resinoso (Duo Link<sup>™</sup>, Bisco Inc., Schaumburg, IL, EUA) (Figura 4I) foram manipuladas e levadas ao redor do pino (Figura 4J) e ao interior do canal com o auxílio de uma espiral lentulo. Cada pino foi posicionado dentro do respectivo canal com leve pressão e os excessos de cimento foram removidos com o auxílio de microbrushes e espátulas (Figura 4K). A fotoativação foi realizada por 40 s (Figura 5B-C). Após 4 min, os espécimes foram colocados em recipientes pretos e mantidos em umidade relativa de 100%, em estufa a 37 °C pelo período de 24 h.

#### 1.3- Corte dos espécimes

Os espécimes foram incluídos em dispositivos retangulares de acrílico, preenchidos com silicona de condensação densa (Coltex/Coltoflax, Coltene, Altstatten, Suíça), ficando com seu longo eixo centralizado e paralelo à superfície externa dos mesmos (Figura 5A). Após a remoção dos excessos e presa do material, os espécimes foram acoplados individualmente à máquina de cortes Isomet 1000 (Buehler, Lake Forest, IL, EUA) (Figura 5D), na qual um disco diamantado (South Bay Technology, San Clement, CA, EUA), sob refrigeração constante, realizou cortes no sentido mésio-distal (perpendiculares ao longo eixos dos pinos) com peso de 75 g e à velocidade de 325 rpm. Foram obtidas fatias correspondentes às regiões cervical, média e apical do pino/reforço com aproximadamente 1,0mm (±0,1 mm) de espessura (6 fatias no total).



**Figura 5**: **Fotoativação do cimento resinoso e corte dos espécimes. A)** Espécime inserido no dispositivo de acrílico preenchido com silicona de condensação. **B)** Ponta do fotoativador. **C)** Verificação da intensidade da luz de fotoativação através do radiômetro de cura Gnatus. **D)** Espécime acoplado a máquina de cortes Isomet (Buehler).



**Figura 6**: **Seleção, inclusão e preparo dos corpos-de-prova para o teste de dureza.** A) Seleção de uma fatia de cada região do reforço (C= cervical; M = médio e A = apical) para o teste de dureza. B) Inclusão das fatias selecionadas em tubos de polietileno. C) Amostra após o polimento. D) Corpo-de-prova sendo submetido ao teste de dureza.

#### 2- Teste de dureza da resina composta

Para o teste de dureza, foram utilizadas três fatias de cada um dos espécimes pertencentes aos grupos fragilizados (n = 15). De acordo com o tempo de fotoativação utilizado no reforço com resina composta formaram-se três grupos: o G1 (controle)- 40 s; G2- 80 s e G3- 120 s.

As três fatias selecionadas de cada espécime (1º, 3º e 5º, Figura 6A), correspondentes as profundidades de teste de 2, 6 e 10 mm respectivamente, foram inseridas em cilindro plástico (Figura 6B) e incluídas em resina epóxica de forma a manter a face a ser testada voltada para cima (Figura 6C). Após a polimerização da resina epóxica, o polimento foi realizado sob irrigação constante com lixas d'água de granulação decrescente de 600 até 1500, seguido do polimento com pastas abrasivas de diamante (3 µm, 1 µm, 0,5 µm e 0,03 µm) – (Buehler Ltda, Lake Bluff, IL, EUA) por 1 min cada. Entre cada lixa ou pasta, os espécimes foram cuidadosamente lavados com água corrente e colocados em banho de ultra-som por cinco minutos.

Vinte e quatro horas após o polimento, os espécimes foram submetidos ao teste de dureza com indentador de pirâmide de diamante de base quadrada (dureza Vickers, HVN- *Hardness Vickers Number*) no microdurômetro Shimadzu HMV2 (Newage Testing Instruments, Inc., Southampom, PA, EUA). Nas regiões cervical (C), média (M) e apical (A) do reforço com resina, a dureza foi calculada

em três diferentes áreas laterais, determinadas nas distâncias de 50  $\mu$ m (p), 200  $\mu$ m (m) e 350  $\mu$ m (d) em relação à interface cimento/pino (Figura 7A).



**Figura 7**: **Amostra de imagens de microscopia óptica durante o teste de dureza.** A) Determinação das distâncias de teste das áreas de indentações (p = próximo ao pino; m = meio; d = próximo à dentina) (100×). B) Amostra das indentações realizadas em cada região (nove no total e três em cada uma das áreas determinadas). Observa-se a manutenção da distância aproximada de 150 μm entre cada uma delas (400×).

Cada indentação utilizou carga estática de 100 g pelo tempo de 15 s. A dureza da resina composta foi calculada com base na média das diagonais estabelecidas com o auxílio de um microscópio óptico acoplado ao microdurômetro Shimadzu HMV2 (Newage Testing Instruments, Inc., Southampom, PA, EUA) e aumento de 400 X. Em cada distância lateral (p, m e d) foi calculada a média de três mensurações, totalizando nove indentações em cada região (Figura 7B). Todas as médias foram calculadas e registradas como dureza Vickers (Hardness Vickers Number, HVN) utilizando o programa Newage C.A.M.S (Computer Assisted

Measurement System, Newage Testing Instrumensts, Inc., Southampom, PA, EUA). Imagens representativas de cada região do reforço foram fotografadas para análise e ilustração dos resultados nos aumentos de 100 e 400 X. Os resultados foram avaliados estatisticamente por meio do teste análise de variância de três vias (3-Way, ANOVA) para comparação das variáveis: tempo de fotoativação, região do reforço e distância lateral da resina composta. De acordo com os resultados encontrados, foram realizadas comparações múltiplas pelo teste estatístico de Tukey *post hoc* com um intervalo de confiança de 95% ( $\alpha = 0,05$ ).

# 3- Teste de cisalhamento por extrusão (*push-out*) e análise da falha ocorrida.

Para o teste de *push-out*, quatro grupos foram formados (n = 15): três grupos fragilizados (G1 = 40 s, G2 = 80 s e G3 = 120 s) e um grupo não fragilizado (Grupo controle).

Foram selecionados 3 cortes para o ensaio de cisalhamento por extrusão dos mesmos espécimes utilizados para o teste de dureza, sendo que os cortes  $2^{\circ}$ ,  $4^{\circ}$  e  $6^{\circ}$  foram aproveitados no teste de resistência de união. Durante o corte, a face apical de cada fatia foi marcada com um ponto preto com caneta à prova d'água. Cada corpo-de-prova foi fixado a uma base metálica de aço inoxidável contendo um orifício de 2,5 mm de diâmetro na região central, acoplado na porção inferior da máquina de ensaio universal Instron, Modelo 4444 (Instron, Canton, MA, EUA). A secção radicular contendo o pino DT Light Post foi posicionado na mesma direção do orifício da base metálica com sua face cervical voltada para baixo. Uma haste metálica, com ponta ativa de 0,6 mm, fixada na porção superior da máquina de ensaio universal (Figura 8) foi acionada com velocidade de cruzeta de 0,5 mm/min, até o deslocamento do pino.



**Figura 8:** Esquema ilustrando espécime adaptado ao dispositivo da máquina Instron, no momento do teste de cisalhamento por extrusão. A: Suporte da Instron; B: Base metálica; C: pino metálico com 0,6 mm de diâmetro; D: Espécime.

A força necessária para o deslocamento do pino foi aferida em quiloNewtons (KN), transformada em Newtons (N) e convertida em Mpa pela divisão da área lateral do pino, conforme indicado na seguinte fórmula: RU=F,

 $S_L$ 

Onde RU é a resistência de união em Mpa; F é a força em N e S<sub>L</sub> é a área lateral do pino em mm<sup>2</sup>.

A resistência máxima ao deslocamento foi calculada dividindo o valor do pico de carga registrado em N dividido pela área lateral do espécime (1 MPa = 1 N/mm<sup>2</sup>. Para o cálculo exato da área lateral aderida, o design do pino foi considerado de acordo com o respectivo nível do corte realizado. A altura de cada espécime foi mensurada com o auxílio de um paquímetro digital (Mitutoyo Messgerate GmbH, Neuss, Germany; acurácia de: 0,001 mm) e a área de adesão foi calculada pela fórmula da área lateral do tronco de cone:

$$S_{L} = \pi (R+r) \sqrt{h^{2} + (R-r)^{2}}$$

onde;

SL é a área lateral do pino;

R= medida do raio do pino em sua porção coronal;

r= medida do raio do pino em sua porção apical;

h= altura/espessura do pino.

Para a análise fractográfica, os espécimes foram submetidos a criterioso exame visual com lupas de 4 X de aumento (Illuminated magnifying glass, Tokyo, Japan). Cada falha foi classificada em um dos cinco subtipos (Figura 9) descritos a seguir: **a) adesiva ao pino** – se o conjunto resina/cimento foi deslocado do pino; **b) adesiva à dentina** – se o material resinoso deslocou-se da dentina; **c) mista** – quando o material resinoso deslocou-se tanto do pino quanto da dentina; **d)**  **coesiva da resina** quando ocorreu fratura somente na resina e **e) coesiva da dentina** quando ocorreu fratura da dentina. As falhas de união observadas foram determinadas em percentuais.



**Figura 9:** Imagens dos tipos de falhas observadas após a realização do teste de resistência de união. R= Resina composta; D= Dentina; P= Pino.

Os dados foram analisados pelos testes estatísticos de *two-way* ANOVA e Tukey *post hoc test* e pelo teste de proporções com um nível de significância de 5%.

#### 4- Análise em Microscopia Eletrônica de Varredura

Para esta pesquisa foram selecionados 12 incisivos superiores de humanos, hígidos, extraídos e armazenados em solução de timol 0,1% diluído em soro fisiológico 0,9%, pH = 7, pelo período máximo de 1 a 3 meses. Os dentes foram limpos, preparados e tiveram suas coroas seccionadas como anteriormente descrito. Após 24 h do tratamento endodôntico, a guta-percha foi removida em 12 mm e as raízes foram aleatoriamente divididas em 4 grupos (n = 3) de acordo com a condição da raiz: 3 experimentais (raízes fragilizadas) e 1 controle (raízes não fragilizadas).

Nos grupos experimentais, os dentes foram aleatoriamente divididos em três grupos de acordo com o tempo de polimerização da resina composta utilizada para o reforço da raiz: Grupo 1 (G1) t = 40 s; Grupo 2 (G2) t = 80 s e Grupo 3 (G3) t = 120 s.

Todos os procedimentos de reforço, cimentação do pino e corte das raízes seguiram os mesmos passos descritos anteriormente, de acordo com o grupo em questão (fragilizado e não fragilizado) e o tempo de fotoativação da resina composta.

Após o corte, três fatias de cada espécime, correspondentes às profundidades de 2, 6 e 10 mm respectivamente, foram selecionadas para a

análise em MEV. O preparo para microscopia foi realizado seguindo o protocolo sugerido por Perdigão *et al.*, (1999).

Os espécimes foram deixados em solução de glutaraldeído 2,5%, tamponado com solução de cacodilato de sódio 0,1 Mol/litro (pH = 7,4) (Dermus, Florianópolis, SC, Brasil) por 12 h a 4 °C. Na seqüência, as fatias foram submetidas a três lavagens com solução de cacodilato de sódio 0,1 Mol/litro em pH = 7,4 (por 20 min cada) e desidratadas em soluções com crescente graduação de álcool (Dermus, Florianópolis, SC, Brasil) 25°, 50°, 75°, 95° (por 20min de imersão em cada solução) e 100° por 1h. Após este período, os espécimes ficaram imersos em solução de Hexametildisilizano (HMDS, Ted Pella, Redding, CA, EUA) por 10 min completando o processo de desidratação e fixação. Estando secos, foram montados em blocos de resina epóxica e, após a polimerização, desgastados até a completa exposição da interface resina/cimento/pino. Em seguida, foram polidos com lixas d'água de granulação decrescente (400, 600, 800 até 1500) e pastas de polimento de óxido de alumínio com granulação de 1,0 e 0,3 µm. Após serem lavados em água corrente e colocados em ultra-som com água deionizada durante 10 min, os espécimes foram submetidos à desmineralização com o uso de ácido clorídrico 6mol/litro por 30 s (Dermus, Florianópolis, SC, Brasil) e desproteinização com solução de hipoclorito de sódio 2% por 10 min (Dermus, Florianópolis, SC, Brasil).

Todas as interfaces foram secas em estufa a 37º C por 24 h, colocadas em uma câmara de vácuo e recobertas com uma camada de ouro de

aproximadamente 300 A° (Bal-Tec SCD 005, Bal-tec Co., EUA). A análise foi realizada em microscópio eletrônico de varredura (Phillips XL30 FEG, Philips, Eindhoven, Netherlands) operando entre 10 Kv e 20 Kv.

A análise qualitativa das interfaces adesivas foi realizada em MEV e incluíram as seguintes características: 1) formação e uniformidade da camada híbrida, 2) camada de adesivo e espessura da camada híbrida; 3) interface dentina/adesivo/resina e pino/cimento resinoso. Além disso, a formação e morfologia dos *tags* de resina e sua relação com a dentina intratubular foram quantitativamente analisados através de escores.

Através das observações feitas em microscópio eletrônico de varredura, no modo de escaneamento de elétrons (SE), no modo de elétrons retro-difundidos (BSE) ou no modo MIX (SE + BSE), foram analisadas qualitativamente as interações dos sistemas adesivos com a dentina condicionada e a formação de camada híbrida na interface dentina-resina. Fotomicrografias de áreas representativas desta interação foram realizadas, a fim de ilustrar os resultados obtidos nos diferentes terços, em cada grupo.

Para a avaliação qualitativa ordinal da formação, morfologia e interação dos *tags* de resina, foram feitas fotomicrografias (X 500) em quatro pontos distintos (superior, inferior, direito e esquerdo), correspondendo aos quadrantes da interface dentina/adesivo/resina (Figura 10), em cada região do reforço analisada (cervical, média e apical). De acordo com os critérios de Ferrari *et al.* (2002), foram determinados escores de 0 a 3 para cada condição observada (Figura 11A-

D): o escore 0,0 foi determinado quando a formação de *tags* de resina não foi detectada (Figura 11A). O escore 1,0 foi determinado quando se observou formação de *tags* de resina curtos e em pequeno número (plugs de resina) (Figura 11B). O escore 2,0 foi registrado quando ocorreu a formação de *tags* de resina numerosos e longos, mas sem a presença de ramificações laterais (Figura 11C). O escore 3,0 foi registrado quando se observou a formação de grande número de *tags* de resina, longos e com ramificações laterais uniformemente evidentes (Figura 11D).



**Figura 10**: Áreas observadas em MEV. As observações foram feitas em quatro pontos distintos (superior, inferior, direito e esquerdo), correspondendo aos quadrantes da interface dentina/adesivo/resina em cada região do reforço analisada (cervical, média e apical). CR= resina composta, D= dentina, P= pino.

Foram feitas 4 avaliações em cada região do reforço totalizando 12 avaliações nas três regiões de cada raiz e 36 em cada grupo (144 no total). Os escores médios registrados nas profundidades de 2, 6 e 10 mm foram estatisticamente avaliados pelos testes de Kruskall-Wallis e Friedman, a fim de detectar diferenças estatísticas significativas entre e dentro dos grupos, com um nível de significância de 5% (P < 0,05). Os resultados (dados) medianos foram utilizados para análise.



**Figura 11:** Determinação dos escores na análise quantitativa da interface adesiva nos diversos grupos experimentais e controle. A) O escore 0 foi determinado quando não se observou formação de *tags* de resina. (X 250, BSE). B) O escore 1,0 foi registrado quando poucos ou esparsos *tags* resinosos foram visíveis (X 500, MIX). C) O escore 2,0 foi determinado na presença de poucos, mas uniformes *tags* de resina, com formação de ramificações laterais (X 500, MIX). D) O escore 3,0 foi registrado quando foi observado grande número de *tags*, longos e numerosos, com ramificações laterais uniformemente distribuídos (D, X 500, BSE).





Ribeirão Preto, 16 de março de 2007.

Prezado Senhor,

Vimos por meio desta informar que Comitê de Ética em Pesquisa da Universidade de Ribeirão Preto CEP/UNAERP analisou e aprovou sem restrições, o Projeto intitulado "EFEITOS DA REGIÃO DA RAIZ DO TEMPO DE POLIMERIZAÇÃO NO REFORÇO RADICULAR COM RESINA COMPOSTA E PINOS DE FIBRA" tendo como pesquisador Prof. Dr. Manoel Damião de Sousa Neto, em reunião ocorrida na data de 20 de novembro de 2006, registrado sobre o ComÉt: 089/06.

Temos ciência de que os estudos estão sendo conduzidos na Universidade de Ribeirão Preto – UNAERP.

Solicitamos que o senhor encaminhe os relatórios parciais e finais, bem como envie-nos possíveis emendas e novos termos de consentimento livre e esclarecido, notifique qualquer evento adverso sério ocorrido no centro e novas informações sobre a segurança do estudo para que possamos fazer o devido acompanhamento.

Atenciosamente,

Prof<sup>®</sup> Dr<sup>\*</sup> Luciana Rezende Alves de Oliveira Coordenadora do Comitê de Ética em Pesquisa da UNAERP Universidade de Ribeirão Preto Artigo 1: "Effects of Light-Exposure Time on Composite Resin Hardness after

Root Reinforcement Using Translucent Fibre Post"

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Manuscript title: Bond Strength of Fiber Posts to Experimentally Flared Root Canal Dentin Reinforced With Composite Resin: Effect of Light-Exposure Time Submission type: O Manuscript number:

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Running Title: Adhesion of fiber posts to weakened/resin-reinforced root dentin

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