Universidade de Uberaba

Vera Beatriz Pacheco

Avaliação da adaptação e resistência de união de um cimento resinoso em canais radiculares

Uberaba – MG

2013

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Trabalho apresentado ao programa de Mestrado em Odontologia da Universidade de Uberaba - UNIUBE, para a obtenção do Título de Mestre em Odontologia - Área de concentração em Biomateriais.

Orientador: Prof. Dr. Luciano de Souza Gonçalves

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Ata da Sessão Pública de defesa de dissertação para obtenção do título de Mestre em Odontologia, área de concentração em Biomateriais, a que se submeteu a aluna Vera Beatriz Miranda Pacheco – matrícula 6102384/1, orientada pelo Prof. Dr. Luciano de Souza Gonçalves

Ao primeiro dia do mês de março do ano de dois mil e treze, às 14 horas, no anfiteatro da biblioteca na Universidade de Uberaba, reuniu-se a Comissão Julgadora da defesa em epígrafe indicada pelo o Colegiado do Programa de Mestrado em Odontologia da Universidade de Uberaba, composta pelos Professores Doutores: Luciano de Souza Gonçalves - **Presidente**, Adriano Fonseca de Lima e Thiago Assunção Valentino, para julgar o trabalho da candidata Vera Beatriz Pacheco, apresentado sob o título: **"Avaliação da Adaptação e Resistência de União de um Cimento Resinoso em Canais Radiculares"**. O Presidente declarou abertos os trabalhos e agradeceu a presença de todos os Membros da Comissão Julgadora. A seguir a candidata dissertou sobre o seu trabalho e foi argüida pela Comissão Julgadora, tendo a todos respondido às respectivas argüições. Terminada a exposição, a Comissão reuniu-se e deliberou pelo seguinte resultado:

(X) **APROVADO**
( ) **REPROVADO** (anexar parecer circunstanciado elaborado pela Comissão Julgadora)

Para fazer jus ao título de MESTRE EM ODONTOLOGIA ÁREA DE CONCENTRAÇÃO BIOMATERIAIS, a versão final da tese, considerada aprovada devidamente conferida pela Secretaria do Mestrado em Odontologia, deverá ser entregue à Secretaria dentro do prazo de 30 dias, *a partir da data da defesa. O aluno Aprovado que não atender a esse prazo será considerado Reprovado. Após a entrega do exemplar definitivo, o resultado será homologado pela* Universidade de Uberaba, conferindo título de validade nacional aos aprovados. Nada mais havendo a tratar, O Senhor Presidente declara a sessão encerrada, cujos trabalhos são objeto desta ata, lavrada por mim, que segue assinada pelos Senhores Membros da Comissão Julgadora, pelo Coordenador do Programa de Mestrado em Odontologia da UNIUBE, com ciência da aluna. Uberaba, ao primeiro dia do mês de março de dois mil e treze.

*Prof. Dr. Luciano de Souza Gonçalves*_______________

Prof. Dr. Adriano Fonseca de Lima _______________

Prof. Dr. Thiago Assunção Valentino _______________

Prof. Dr. José Bento Alves_______________
Coordenador do Programa de Mestrado em Odontologia da UNIUBE

Poliana Gomes da Silva Alves _______________
Secretária do Programa de Mestrado em Odontologia da UNIUBE

Ciência da Aluna:_______________

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Dedicatória

Dedico esse trabalho aos meus pais, **Waldomiro Pacheco** (*in memoriam*) e **Cleide Miranda Pacheco**, que sempre me incentivaram para que não desistisse de meus ideais, ajudando-me a superar os obstáculos por meio da palavra sincera, amiga e segura.

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Resumo

O objetivo neste estudo foi avaliar a resistência de união e a formação de fenda durante o preenchimento de canais radiculares e cimentação de pino pré-fabricado de fibra de vidro utilizando um cimento resinoso auto adesivo associado ou não à um sistema adesivo de condicionamento ácido total. Quarenta incisivos centrais bovinos foram selecionados, tratados endodonticamente e divididos aleatoriamente em quatro grupos: Controle – Cimento RelyX U 100 (U100) + pino de fibra de vidro; Grupo 1 – ScotchBond Multi uso Plus (SB) + U100 + pino de fibra de vidro; Grupo 2 – preenchimento com UC e Grupo 3 – SB + UC. Vinte e quatro horas após a fotoativação os corpos-de-prova foram seccionados transversalmente ao longo-eixo do dente em uma cortadeira metalográfica. Réplicas em resina epóxi foram confeccionadas para visualização da formação de fenda na interface cimento resinoso/dentina em microscópio eletrônico de varredura (MEV). O teste de resistência de união push-out foi realizado em uma máquina de ensaio universal com velocidade constante de 0,5 mm/min. Os valores de resistência de união em Mega Pascal foram submetidos ao teste de Kruskall-Wallis e o post-hoc teste de Dunn. O padrão de falha dos corpos de prova foram observados em MEV. Os resultados obtidos demonstram que a presença do pino não influenciou a resistência de união dos grupos testados. Não foi encontrada também diferença de resistência de união entre as diferentes profundidades do canal radicular. Entretanto, o uso do sistema adesivo influenciou positivamente a resistência de união do cimento resinoso à dentina nos terços cervical e médio, o que pode ser confirmada pela diminuição da frequência de fraturas adesivas nos grupos que utilizaram o adesivo. Não foram observadas fendas na interface entre o cimento resinoso e a dentina independente da utilização do sistema adesivo. No entanto, um número expressivo de bolhas foi encontrado nos grupos onde os pinos de fibra de vidro não foram utilizados, o que pode ter contribuído para o aumento das fraturas mistas e coesivas. Dentro das limitações do estudo foi possível concluir que o sistema adesivo potencializou a união entre o cimento resinoso e a dentina radicular. O uso do cimento resinoso U100 como monobloco primário é menos indicado em relação a utilização dos pinos de fibra de vidro devido a presença de bolhas nas três profundidades avaliadas.

Palavras chaves: resistência de união, cimentos de resina, pinos de fibra de vidro, materiais dentários.

Abstract

The aim of this study was to evaluate the bond strength and gap formation on the root canals filled with Glass fiber posts (GFP) cemented with a self-adhesive resin cement or completely filled with this agent, associated or not to a total-etch adhesive system. Forty central bovine incisors were selected, endodontically treated and randomly divided into four groups: Control – RelyX U 100 resin cement (U100) + GFP; Group 1 – Scotch Bond Multi-purpose Plus (SB) + (U100) + GFP; Group 2 canal root filled with U100 and Group 3 – SB + U100. Twenty-four hours after light-curing, the specimens were sectioned in low speed diamond saw. Epoxy resin replicas were made to evaluate the gap formation on the resin cement/dentin interfaces with scanning electron microscope (SEM). Push out Bond strength test was performed in a universal testing machine with a crosshead speed of 0.5 mm/min. Bond strength values in Mega Pascal were submitted to Kruskall-Wallis and Dunn's post-hoc test. The failure mode was observed under SEM. The results showed that the GFP had no influence on the bond strength values of the tested groups. Differences on bond strength among the three depth of cure were not found. However, the adhesive system had a positive influence on the bond strength of the resin cement to dentin, results corroborated by the lower adhesive failures observed in the groups with adhesive application. Gaps in the resin cement/dentin interface were not observed regardless the use of the adhesive system. However, a large number of air bubbles were found in the groups where the GFP were not used. This fact may be contributed to increasing of cohesive and mixed failures. Within the limitations of the study it was possible to conclude that the adhesive system improved the bond strength between the self-adhesive resin cement and root dentin, Use of U100 as primary monoblock is less indicated when compared to use of GFP due presence of air bubbled in all depths of the canal root.

Keywords: Bond strength, resin cements, fiber posts, dental materials.

Sumário

Original Research

Evaluation of adaptation and bond strength of a self-adhesive resin cement to root canals

Vera Beatriz Pacheco[1], Túlio Henrique Leandro Duarte[2], Ailla Carla Lancellotti[3], Janisse Martinelli[4], Fernando Carlos Hueb de Menezes[5], Luciano de Souza Gonçalves[5]

1. Post-graduate student, Biomaterials Division, Uberaba University, Uberaba – MG
2. Undergraduate student
3. Post-graduate student Departament f Restorative Dentistry, Dental Materials Division, University of Campinas, Piracicaba – SP.
4. Professor, Prothesis Division, Uberaba University, Uberaba – MG
5. Professor, Biomaterials Division, Uberaba University, Uberaba – MG

Corresponding author:

Prof. Luciano de Souza Gonçalves

Avenida Nenê Sabino, 1801. Sala 2R02 - Bairro Universitário - CEP: 38.055-500. Uberaba – MG.

Phone: 55 (34) 3319 8958

Fax: 55 (34) 3314 8910

Email: goncalves1976@yahoo.com.br

Abstract

This study evaluated the bond strength (BS) and gap formation on the root canals filled with a glass fiber post (GFP) and/or a self-adhesive resin cement. Forty bovine incisors were selected, endodontically treated and divided into four groups: Control – RelyX U 100 resin cement (U100) + GFP; Group 1 – ScotchBond Multi-Purpose Plus (SBM) + U100 + GFP; Group 2: root canal filled with U100 and Group 3 – SBM + U100. The specimens were sectioned using a low-speed diamond saw and replicas were made to evaluate the gap formation at the adhesive interface by scanning electron microscopy (SEM). Push-out test was performed in a testing machine with a crosshead speed of 0.5 mm/min. BS values (MPa) were submitted to Kruskall-Wallis and Dunn's *post-hoc* test and failure mode observed by SEM. BS values was not influenced by GFP presence and depth of root. SBM had positive influence on the BS between U100 and dentin, and lower adhesive failures were found in groups with SBM application. No gaps in the cement/dentin interface were observed in all groups. However, a large number of air bubbles were found in the groups where the GFP were not used. In conclusion, SBM was crucial to establish suitable BS between the resin cement and root. U100 as primary monoblock is less indicated when compared to U100+GFP due presence of air bubbles in all depths of the canal root.

Keywords: Bond strength, resin cements, fiber posts, dental materials.

Introduction

The use of additional retention, as intraradicular posts, is commonly necessary for restoration of teeth with extensive loss of coronal structure (1, 2), and the use of glass-fiber posts (GFP) has increased since its introduction in dentistry in 1990s.

The good acceptance of the GFP is due to its properties, as elastic modulus similar to the dentin, decreasing the occurrence of root fracture (3-5), or most favorable prognosis repair (6, 7). High resistance corrosion and good aesthetic appearance (8-11) are important advantages of GFP when compared to custom cast cores. In addiction, the adhesive cementation provides a better stress distribution between the restorative materials and root dentin (12) behaving like a single body or a monoblock, as called in the endodontic literature (13).

Monoblocks are classified according to number the interfaces among the substrate (root dentin) and the materials used in the restorative materials: primary monoblocks – present a unique circumferential interface between the root filling material and the wall of the root canal; secondary monoblocks – present two circumferential interfaces; Third monoblock – a third circumferential interface is introduced between the bonding substrate and the abutment material (i.e. GFP). In a previous study (14), the combination of the adhesive system and the dual resin cement was considered a primary interface, since both materials form a structure with a single elastic modulus. When these materials are associated with GFP, a secondary monoblock is formed.

Self-adhesive resin cements were introduced in dentistry in order to reduce the steps of adhesive procedures, eliminating the use of the adhesive system (14-18). Compared to the conventional technique, lower bond strength values have been reported (19, 20). However, based on its chemical bond to dentin (21), the filling of the root canal represents the possibility of creating a genuine monoblock, without the interface with the adhesive system. The main objective of this monoblock would strengthen the tooth structure, and provide retention for the prosthetic crow without the use of GFP. Nevertheless, many factors must be considered. The high C-factor into the canal root, volumetric shrinkage and the shrinkage stress of composite bulk when the GFPs are not used may lead to a gap formation and consequently, failure of adhesive interface (15, 22).

Therefore, the purpose of this *in vitro* study was to evaluate the influence of the adhesive system and GFP on the filling of root canal using self-adhesive dual resin cement. It was hypothesized that the use of adhesive systems and GFP would not influence the bond strength and gap formation of resin interface.

Materials and Methods

Specimen's preparation

For the bond strength test, 40 freshly extracted central bovine incisors with similar length (18 mm) and apexogenesis completed were selected, cleaned and storage in distilled water for a maximum period of 14 d. The roots were sectioned perpendicular to the long axis 17 mm from the apex with a water-cooled low-speed diamond saw (Isomet 1000; Buehler, Lake Bluff, Ill, USA). The pulp was removed and the root canals were instrumented up to 1 mm short of the apex using #40 to #80 K-files (Dentsply-Maillefer, Tulsa, OK, USA), copiously irrigated with 1% Sodium hypochlorite (NaOCl) solution at each change of instrument. Then, the root canals were irrigated with distilled water, dried using absorbents paper cones and filled with gutta-percha and Sealer26 (Dentsply-Maillefer, Tulsa, OK, USA), using the lateral condensation technique with a finger spreader (Dentsply-Maillefer, Tulsa, OK, USA). After the filling, the cervical opening of root canals were sealed with a eugenol-free temporary restorative material (Coltosol – Colténe AG, Altstäten, Switzerland) and storage in distilled water at 37°C by 7d.

After this period, the gutta-percha was removed using Gates-Glidden drills (Dentsply-Maillefer, Tulsa, OK, USA) up to 1 mm to the apical sealing, The root canals were standardized to a depth of 15 mm using size post-hole drills supplied by the #3 glass-fiber post system kit used (Exacto, Angelus, Londrina, PR, Brazil) and the roots were randomly divided into four groups (n=10) according to the following procedures: Control (CO) – GFP cemented with a self-adhesive resin cement (RelyX U100, 3M ESPE, St. Paul, MN, USA) shade A3, according to the manufacture's instructions. Group 1 (G1) – GFP cemented with RelyX U100 associated with the total etching adhesive system ScotchBond Multi-Purpose Plus (3M ESPE St. Paul, MN, USA), following the manufacture's instructions for endodontic posts. Group 2 (G2) – Canal roots were entirely filled with the resin cement Rely X U100 mixed according manufacture's instructions without GFP. Group 3 (G3) – Total etching adhesive system ScotchBond Multi-Purpose Plus applied on the internal walls and the root canal was filled with the resin cement Rely X U100 mixed, according manufacture's instructions without GFP. All specimens were light-cured for 60 s from the top using a LED source (Radii-cal, SDI Ltd. Bayswater, Victoria, Australia) with 645 mW/cm^2 irradiance, verified with a calibrated power meter (Ophir Optronics Ltda, Jerusalem, Israel). The specimens were stored at 37°C in 100% of relative humidity, protected from the light for 24h.

Push out Bond strength test

After the storage, the specimens were perpendicularly sectioned to the long axis with a low-speed water-cooled diamond saw (Buehler, Lake Bluff, Ill, USA). The first 1-mm-segment was discarded, and three segments (2.5 mm thick), corresponding to the cervical, middle, and apical regions of the root were prepared for the push-out test. The root slabs were positioned in the push-out device, with the apical surface facing the plunger tip, avoiding the contact with the dentin. The push-out test was performed in universal testing machine (Instron 4411, Instron®, Canton, MA, USA) at a crosshead speed of 0.5 mm/min. The results in MPa were submitted to Kruskal-Wallis test and Dunn's post-hoc test with $\alpha=0.05$. After testing, fractured specimens were mounted on metallic stubs, sputter coated with gold and observed under scanning electron microscopy (SEM) (JEOL, JSM-5600LV; Tokyo, Japan) for failure mode classification based on the following criteria: Adhesive – at least 75% of the bond area was in dentin, and it was possible to observe the dentin tubules, resin tags and/or resin cement on the surface of the plug; Cem/dent – involving the resin cement and dentin; (Cem/post) – involving the resin cement and the GFP; Mixed – involving three substrates and Cohesive – more than 75% of the fracture occurred into the resin cement bulk.

Interface evaluation

For interface evaluation, five additional specimens for each group were prepared and sectioned as reported above. The specimens were embedded in epoxy resin (Buehler, USA) and were wet-polished with 600-, 1200- and 2000-grit silicon carbide papers, and then polished with 1 mm, 0.3 mm and 0.05 mm Al_2O_3 suspensions. After polishing the surfaces were etched with phosphoric acid 50% for 3 s, immersed in sodium hypochlorite 1% for 20 min. Impressions of the specimens were made with polyvinyl siloxane material (Express, 3M/ESPE, USA) and epoxy resin replicas were prepared to evaluate the presence of gaps, avoiding the influence of the vacuum of the SEM technique. In order to observe the formation of adhesive tags, the polishing with Al_2O_3 suspensions, phosphoric acid etching and, sodium hypochlorite immersion were repeated as previously preformed. Both specimens (original and replicas) were sputter coated with gold and observed under SEM (JEOL, Tokyo, Japan).

Results

No gaps were observed in all regions of the root after replicas analysis. However, bubbles can be seen in areas of greater thickness of the resin cement, especially in the groups without GFP use. Hybrid layer and tags formation were observed only in the groups where the adhesive system was used (G1 and G3), different from the control and G2 where an interface between dentin and resin cement in intimate contact was observed (Fig 1).

Failure mode analysis showed a predominance of adhesive failures at the Control, as well as a large number of cem/dent failures, different from G1 were many cem/post and mixed failures were observed. G2 and G3, which have not used posts, presented a large number of adhesive and cem/dent, however, cohesive failures could be seen, especially at G3 (Fig. 2).

G1 and G3 showed higher bond strength values compared to Control and G2 on the cervical and middle thirds (Table 1). The presence of GFP and root regions had no influence on the values of the bond strength, however, a large amount of air bubbles were found at cervical, middle and apical thirds when the canal root was filled with only resin cement (Fig. 3).

Discussion

According a previous study (13) teeth endodontical-treated could be successfully restored when a homogenous unit is formed by restored material with the same elasticity modulus to the root dentin. The authors designed that structure by “monoblock” term. However, dentistry does not have a single material that fulfills this role, since some materials as adhesive systems, resin cements and GFPs are combined in order to achieve this effect. Nevertheless, the stress in the root canal can be increased by adding new interfaces, when different materials are present (13). Under oblique coronal tooth charge, to finite element study analysis (FEA) (14), when the tooth structure is too weak to resist overloads, the use of only resin cement to restore canal root creating a primary monoblock, can limit the amount of stress concentrated on these weak parts of the tooth, reducing the possibility to fracture. On the other hand, at the clinical situation, the effect of shrinkage stress of resin materials could be unfavorable for the adhesion. Based on these information and the results of the present study the hypothesis was partially rejected.

The results of the present study showed an increase in the bond strength values when total-etch adhesive was used prior the self-adhesive dual-cured resin cement to cervical and middle third analyzed, corroborated by other studies (23-25), what did not occur to the apical root region. The phosphoric acid etching used at the conventional total etch adhesive system removes the smear layer, opening the dentin tubules, exposes the collagen fibrils allowing the adhesive infiltration (25), the polymerization of the monomers amonng the colagen structure resutlts in the hybrid layer resultin in high bond strength values. On the other hand, the self-adhesive cement promoted limited hybridization with weak coating bonding and disorganized collagen fibrils that could be degraded over time (26). Due to the methacrylate phosphoric esters present in this cement does not present effectiveness as phosphoric acid to dissolve the smear layer obtained during the preparation of the walls of the canal root, and suitable hybrid layer is not formed, reducing the interlocking between the two substrates (resin cement-dentin), and consequently promoting lower values of bond strength (27, 28). In addition, the high viscosity of the resin cement compared to the bonding agent limits the infiltration of the material in the demineralized dentin, reducing the effectiveness of the bonding, explaining the obtained results. This situation is showed in the Figure 1.

At the apical root region the dentin hybridization could be critical and influenced by the dentin morphology, adhesive system, luting agent and material cured type (29). In the present study, the use of the total-etch adhesive did not improve the apical bond strength when compared with the others groups, what can be explained by the restrict access of the light at this root region (27, 30), although translucent posts minimize this problems (31). Moreover, the anatomic particularity of the apical portion of the root canal system like sclerosis or less distribution and density of dentinal tubules could make difficult the dentin hybridization (25). In addition, the solvent present at the primer component of the adhesive system could not been evaporated or removed due the depth and the narrow diameter of the apical root region (32). The bond strength of U100 probably was not reduced because, according the manufacture, the chemical adhesion promoted between the phosphoric groups and calcium. This reaction is improved by the wetness present at dentin (33).

The results showed that the use of the glass-fiber post did not affect the bond strength, possible explained by the dual-cured cement luting and the adhesive resin. When posts are used in root restoration, the amount of resin cement is reduced along canal length, what can promote less volume shrinkage at the adhesive interfaces (13). Controversy results concerning post use are present at the literature. Some authors found negative influence of the posts on the bond strength, because the increased C-factor promoted by resin cement bulk reduction. This reduction at the resin cement thick difficult the stress release, increasing the contraction at the adhesive interfaces (32).

SEM evaluation of the groups with posts reveled different failure modes. When only U100 was used, adhesive failures were more present, however, when SBM was applied prior the resin cement, more mixed failures occurred (Fig. 2), showing direct influence of the hybrid layer formed along dentin interface instead the post presence (27). To the groups that used SBM prior the resin cement application, SEM analysis showed different failure modes. When posts were used, high number of mixed and Cem/post failures occur, revealing the weak link out of the Cem/post interface (25) compared to the adhesion of SBM to dentin. This behavior revels the importance of the adhesive system in endodontic restorations claiming about limitation still present at the self-adhesive materials. To the group with post absence, adhesive and cohesive failures were the majority failures. Problems with cohesive strength of U100 are already showed in a previous study (33), and this issues is related to the presence of water, necessary for acid ionization; however, the same water can induce a weak bulk if the amount released was not entirely consumed in the reaction, especially when the thickness of the cement is significant. The presence of numerous bubbles along the bulk of the resin cement can be partially responsible for the different failures observed in the groups using U100 without the fiber post (Fig. 3). These bubbles probably were created during the material insertion, due to the large volume of cement, promoting a weak link at this region, and been responsible to initiate the stress, transmitting to the entire material (Figure 3, B, C and D).

Conclusion

Even with self-adhesive characteristics of U100, previously use of the adhesive system is crucial to obtaining satisfactory bond strength values. GFPs had no effect on the bond strength.

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Table 1. Means of Push-out test followed by the standard deviation.

	CONTROL	G1	G2	G3
Cervical	4.38 (2.61 – 7.49)Ba	15.08 (12.08– 18.24)Aa	6.69 (3.95 – 8.40)Ba	8.18 (2.41 – 13.04)Aa
Middle	5.88 (4.27 – 7.28)BCa	12.10 (10.22 – 16.22)Aa	3.17 (2.70 – 6.56)Ca	10.67 (4.40 – 13.58)ABa
Apical	4.56 (3.45 – 6.67)Aa	11.89 (8,93 – 14.29)Aa	5.24 (2.97 – 8.15)Aa	9.55 (4.71 – 15.68)Aa

The same capital letters in row and small letters in column indicate statistical similarity to the Kruskal-Wallis ans Dunn's *post-hoc* test ($P<0.05$).

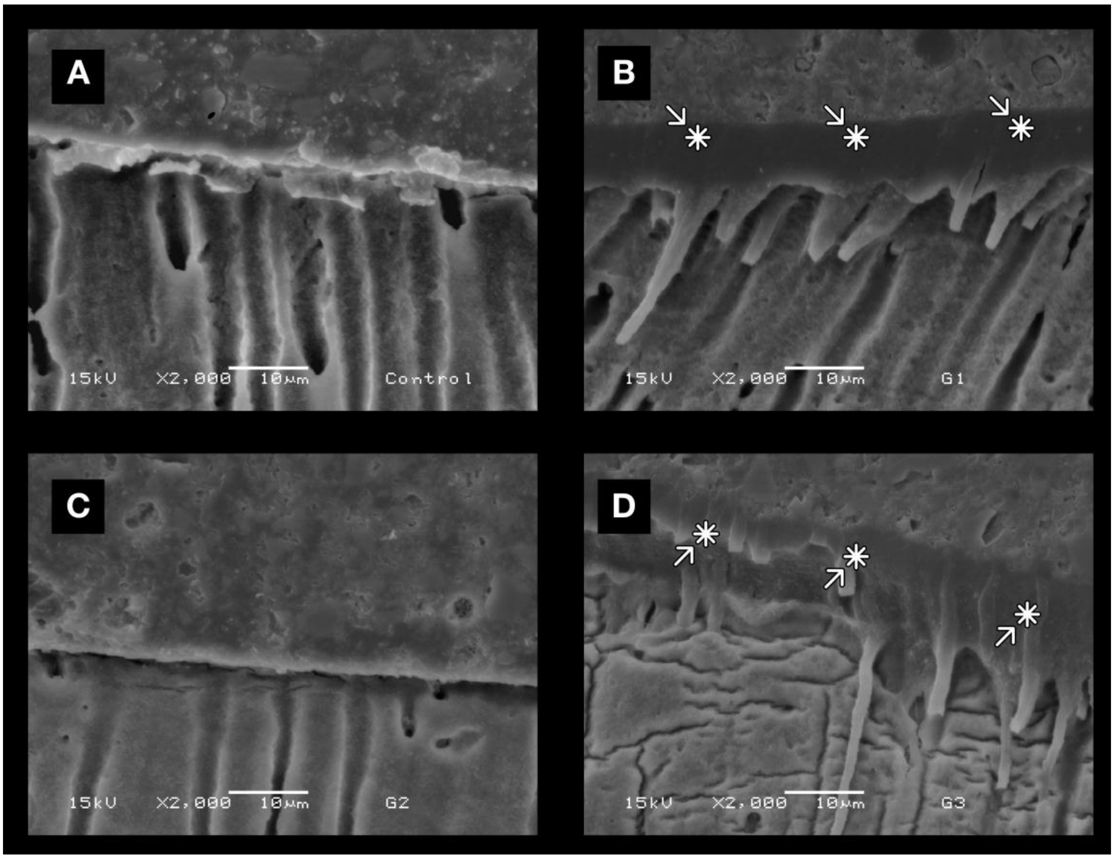

Figure 1. Adhesive interface of the tested groups: A) Control, B) G1 , C) G2 and D) G3. The arrows with (*) highlight the hibrid layer in G1 (B) and G2 (D)

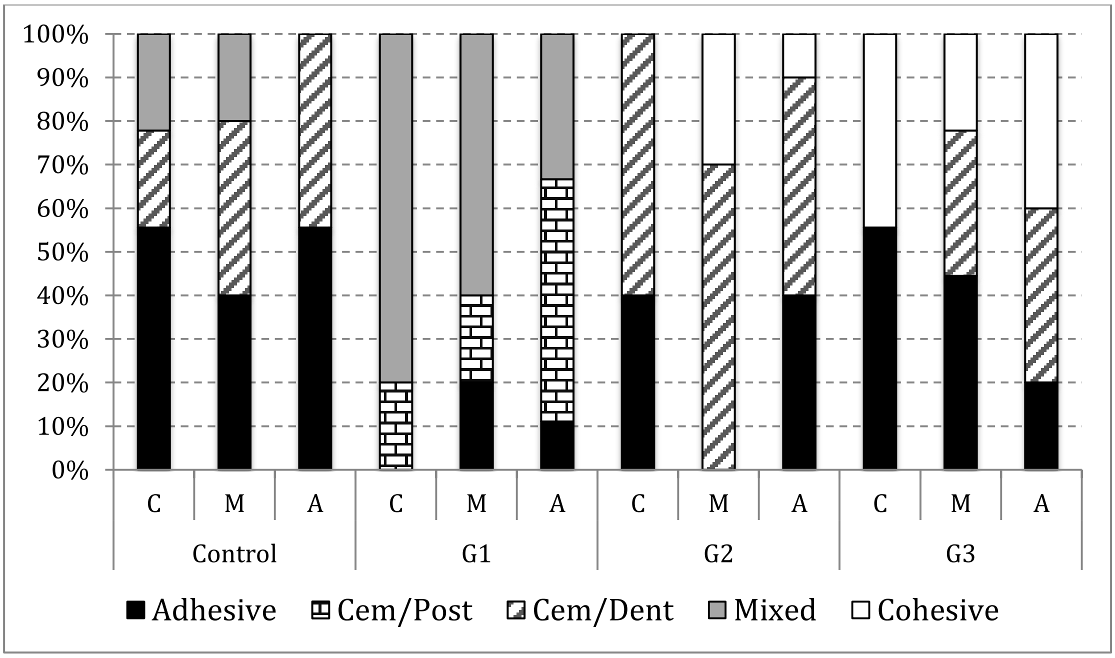

Figure 2. Distribution of the failure mode.

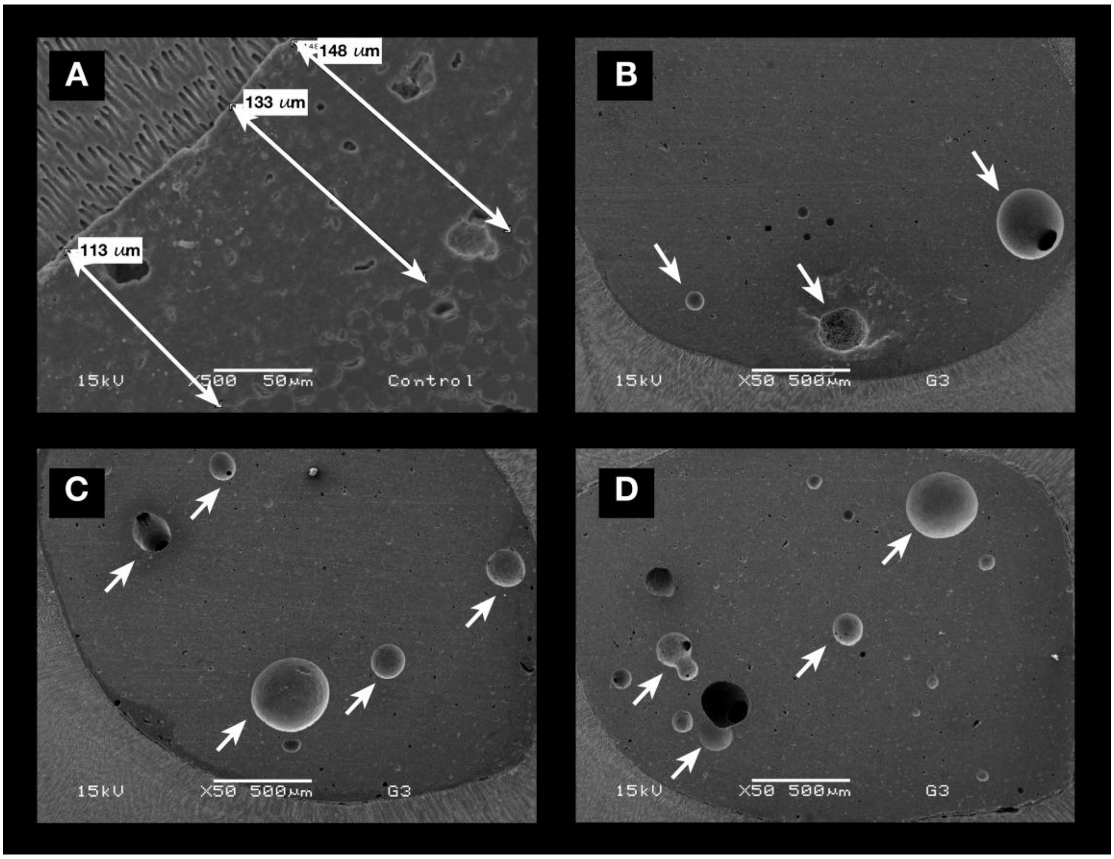

Figure 3. Images of the interfce. In A, the arrows pointe the thickness of cement layer. In B, C and D the arrows highlight the presence of bubbles for G3, in cervical, middle and apical third respectivelly.

ANEXO 1

Guideliness for authors
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<u>Mailing address:</u>
Quintessenz Verlags-GmbH, Juliane Richter
The Journal of Adhesive Dentistry,
Ifenpfad 2-4, D–12107 Berlin, Germany

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