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ORIGINAL ARTICLE

# Influence of adhesive system in bond strength of fiber glass posts to radicular dentin using dual cure resin cement

Influência do sistema adesivo na resistência de união de pinos de fibra à dentina radicular utilizando cimento resinoso dual

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## ABSTRACT

The purpose of this study was to evaluate the bond strength between root dentin and glass fiber posts cemented with dual cure resin cement associated with the total-etch and self-etch adhesive systems. Twenty-four single-rooted human teeth were selected, and the crowns were removed at the cement enamel junction. The roots received biomechanical preparation and obturation followed by the intraradicular preparation compatible with a drill diameter of the glass fiber posts. The roots were divided into two groups, according to cementation protocol (n-12): Group 1 - etched with 37% phosphoric acid for 30 seconds, total-etch adhesive system Single Bond (3M ESPE) and Enforce resinous cement (Dentsply); Group 2 – self-etch adhesive system One Up Bond F (Tokuyama) and Enforce resin cement (Dentsply). After the posts cementation, the roots were kept at 37º C for one week, and submitted to pull out test on the universal testing machine Emic to a traction speed of 1 mm/min. Data in MPa were submitted to the statistical t-test (5%). The t-test showed significant differences between the two groups ($p = 0.003$). The average values in MPa (±standard-deviation) were: Group 1: 5.28(± 3.25), Group 2: 10.05 (± 3.78). Enforce associated with the self-etch adhesive system One Up Bond F showed significantly higher bond strength values than Group 1 - Enforce associated with the total-etch adhesive system Single Bond.

## RESUMO

O objetivo deste estudo foi avaliar in vitro a resistência de união à dentina radicular de pinos estéticos pré-fabricados cimentados com cimento resinoso dual associado aos sistemas adesivos convencionais e autocondicionantes. Foram utilizados 24 dentes unirradiculares, nos quais as raízes foram removidas na junção esmalte-cemento. Realizou-se a instrumentação e a obturação dos canais radiculares seguido do preparo intra-radicular com uma broca compatível com o diâmetro do retentor intra-radicular de fibra de vidro. Os pinos estéticos pré-fabricados foram divididos em 2 grupos, de acordo com o protocolo de cimentação (n - 12): Grupo 1 - condicionamento com ácido fosfórico 37%, aplicação do sistema adesivo convencional Single Bond (3M ESPE) e cimento resinoso Enforce; (Dentsply) Grupo 2 – aplicação do sistema adesivo autocondicionante One Up Bond F(Tokuyama) e cimento resinoso Enforce (Dentsply). Após a cimentação os espécimes foram armazenados a 37º C por 7 dias e submetidos ao ensaio de pull-out na máquina de Ensaio Universal EMIC a uma velocidade de 1 mm/min. Os valores de MPa foram analisados de acordo com teste estatístico de t (5%). O teste t mostrou que houve diferença significante entre os dois grupos ($p = 0{,}003$). Os valores de média MPa (±desvio-padrão) são: Grupo 1: 5,28(± 3,25), Grupo 2: 10,05(± 3,78). Enforce associado ao sistema adesivo autocondicionanate One Up Bond F apresentou valores de resistência de união à dentina intrarradicular superiores quando comparado ao Grupo 1 - Enforce associado ao sistema adesivo convencional Single Bond.

## KEYWORDS

Dental Cements; Adhesives; Dentin; Traction.

## PALAVRAS-CHAVE

Cimentos dentários; Adesivos; Dentina; Tração.

## INTRODUCTION

In dental practice, intraradicular dentin posts and cores are often used to restore endodontically treated teeth that are weakened by the loss of tooth structure [1,2]. The adequate intraradicular support and retention with a material that has modulus of elasticity similar to dentin offers greater security compared to cast metallic posts, which have the potential to transfer stress to the adjacent tooth structure [3,4].

Post-based fiber-reinforced composites (carbon fibers, polyethylene, glass or quartz) are materials of high resistance [5,6], aesthetically advantageous and that provide strength to the tooth structure [2]. The quality of cements used for bonding root canal posts has a primordial importance for success, because its correct employment will provide adequate adhesion and marginal sealing and will guarantee the characteristics required for good clinical performance of the restorations. To enable this to occur, there must be compatibility among the post/cement/dental structure, but also improved mechanical properties and aesthetic qualities [7,8].

Several different types of materials may be used for luting these posts, and the resin cements have been presenting better adhesiveness, compatibility and esthetics, in addition to better mechanical properties [7,8]. Dual cure resin cement was introduced to overcome a disadvantage of light cure cements, that presented restricted conversion depth of the luting agent, in which light transmission, and therefore, cement cure is diminished between the tooth and the prosthetic part [7,8]. Dual cure resin cement can be associated with the total-etch adhesive systems and with the self-etch adhesive systems.

The total-etch adhesive systems presents several particularities: (1) excessive etching enables o formation of a deep zone of demineralized dentin; the adhesive may not infiltrate throughout the entire decalcified area, originating a hybrid layer with regions of exposed collagen fibers that are weakened and more susceptible to hydrolysis (weak zone) [9]; (2) excessive drying on conditioned dentin may lead to the collapse of collagen fibers, whereas excess residual water on it may contribute to separation of the adhesive system phases promote longitudinal degradation of the restoration [10].

Taking into account the possibility of failures in hybrid layer with total-etch adhesives and following the modern trend of simplifying the clinical steps and saving operating time, self-etch adhesives were developed. They are composed of acidic resin monomers that simultaneously promote superficial dentin demineralization and resin adhesive infiltration into the dentin tissue[11]. These adhesive reduce the possibility of weak zone formation, reduce the clinical steps eliminating the acid etching and washing stages, and the technique sensitivity as regards maintenance of dentin humidity [12].

However, the interaction of the resin cements with different adhesives (total-etch and self-etch) can alter the mechanical properties of the restoring material. Thus, the aim of this study was to evaluate the in vitro bond strength of aesthetic glass-fiber posts cemented to intraradicular dentin with dual cure resin cement associated with total-etch and self-etch adhesive systems. The null hypothesis tested was there are not differences in bond strength between the dentin and glass-fiber posts cemented with dual cure resin cement associated with total-etch or self-etch adhesive systems.

## MATERIAL & METHODS

This study was approved by the Ethics Committee of the São José dos Campos School of Dentistry (protocol number 049/2007-PA/CEP). In this study, 24 incisors uniradicular human teeth were extracted for periodontal or orthodontic reasons. After extraction, the teeth were immersed in distilled water and kept in a freezer at -18 °C until the time they were used, not exceeding 6 weeks. The crowns were sectioned with carborundum disks (Dentorium, Labordental, São Paulo, SP, Brazil) in a high speed lathe, and the root size standardized at 15 mm. The roots received biomechanical preparation determining the working length at 1 mm to the nearside of the apex, and using the serial instrumentation technique (classical), with Kerr type (Dentsply Maillefer, Ballaigues, Switzerland) endodontic files, until instrument 40 K- file, alternately irrigating the canals with 1% sodium hypochlorite. To finalize biomechanical preparation, scaled instrumentation was performed, with progressive anatomical withdrawal until instrument 80 K - file.

The root canals were obturated through lateral condensation of gutta-percha (Dentsply Maillefer, Ballaigues, Switzerland) and with a resin sealer (Sealer 26, Dentsply DeTrey GmbH, Konstanz, Germany). The root canals were placed in a humidor (100% relative humidity) for 1 week at 37 °C. Gutta-percha was removed from the cervical region of the root canal to a depth of 5 mm.

The roots were included in a 25 mm diameter/hight PVC tube, with self-polymerizing acrylic resin (Classic, São Paulo, SP, Brazil). A parallel-meter (Bio Art Ltda., São Carlos, SP, Brazil) was used to keep the roots in the direction to the long axis of the PVC tube.

The root areas for the posts were prepared to a depth of 10 mm with special drills supplied by the manufacturer (White Post Drill DC-E, FGM, Joinvile, SC, Brazil), with a handpiece attached to the parallel-meter, allowing the intraradicular preparation to be in the direction to the long axis of the roots.

The glass fiber posts (White Post DC-E, Number 2, FGM, Joinvile, SC, Brazil) were treated with 10% hydrofluoric acid (Dentsply DeTrey GmbH, Konstanz, Germany) for 5 min, followed by water rinsing for 2 min and air-drying. A silane coupling agent (Dentsply DeTrey GmbH, Konstanz, Germany) was applied according to the manufacturer's instructions. Next, the glass fiber posts were adapted to the vertically movable rod of the parallel-meter, allowing the intra-radicular cimentation in the direction to the long axis of the roots.

Twenty-four roots were randomly divided according to the adhesive system used (n-12):

• Group 1 - The root areas for cementing the posts were etched with 37% phosphoric acid (Scotchbond Etchant, 3M ESPE, St. Paul, MN, USA) for 15 seconds and washed with air/water jet for 20 seconds and dried with paper points (Dentsply Maillefer, Ballaigues, Switzerland). The Single Bond total-etch adhesive system (3M ESPE, St. Paul, MN, USA) was applied to the inner walls of the root dentin in accordance with the manufacturer's instructions. Excess adhesive was removed using a microbrush and an air jet was applied for 5 s. The adhesive layer was cured using a QTH Light Curing unit XL 3000 (3M ESPE, St. Paul, MN, USA) with power density of 600 mW/cm$^2$ for 10 seconds.

• Group 2 - The One Up Bond F Plus self-etch adhesive system (Tokuyama, Shibuya-ku, Tokyo, Japan) was applied to the root dentin inner walls in accordance with manufacturer's instructions. Excess adhesive was removed using a microbrush and an air jet was applied for 5 s. The adhesive layer was cured (QTH Light Curing unit XL, 3M ESPE, St. Paul, MN, USA) for 10 seconds.

A dual cure resin cement Enforce (Dentsply DeTrey GmbH, Konstanz, Germany) was used for cementation. This cement is composed of a catalyst paste and a matized base paste in shade A3. The pastes were mixed in accordance to the manufacturer's instructions, and placed in the root canal using a lentulo spiral instrument (Dentsply Maillefer, Ballaigues, Switzerland). Posts were coated with cement and slowly seated in place using a parallel-meter. Excess cement was removed with an explorer. Cement was cured for 40 s.

The trade name, chemical composition and manufacturer of the materials used are presented in Table 1.

**Table 1 -** Trade name, lot, manufacturer and chemical composition and application mode of the materials

| Trade name | Lot | Manufacturer | Composition | Application mode |
|---|---|---|---|---|
| Enforce resin cement | 154345 | Densply De Trey GmbH D, Konstanz, Germany | Bis-GMA, BHT, EDAB, BDMA, HEMA, TEGDMA, fumed silica, silanized barium, aluminum bore-silicate glass (66 wt%) | One part of catalyzer paste was mixed in one part of matized paste, placed in the root canal together with posts and cured for 40 s. |
| Single Bond total-etch adhesive | 7LH | 3M ESPE, St.Paul, MN, USA | Bis-GMA, HEMA, dimethacrylate, methacrylate functional copolymer of polyacrylic and polytaconic acid, water, alcohol, photoinitiator | Two coats applied for 15 s each, actively; Air dry (5 s at 20 cm); Light cure (10 s at 600 mW/cm²) |
| Scotchbond Etchant | 5EX | 3M ESPE, St. Paul, MN, USA | 37% phosphoric acid | Acid etch (15 s), Rinse (15 s); moisture excesses removed with absorbent paper |
| One Up Bond F self-etch adhesive | 000231E | Tokuyama, Shibuya-ku, Tokyo, Japan | Agent A: MAC-10; methacryloxyalkyl acid phosphate; Bis-GMA; TEG-DMA; Photoinitiators. Agent B: HEMA, water, aluminum glass poder; Silica; Photoinitiators | One drop of Agent A is mixed in one drop of Agent B. One coat of mixture applied for 20 s, passively. Air dry (5 s at 20 cm); Light cure (10 s at 600 mW/cm²) |

*Bis-GMA = bisphenol A glycidyl methacrylate; BHT = 2,6 di-tert-butyl-p hydroxyl toluene; EDAB = ethyl-4-dimethylaminobenzoate; BDMA = Benzyl Dimethyl Amine; HEMA = 2-hydroxyethyl methacrylate; MAC-10 = malonic acid 10-methacryl oxide camethylene; TEGDMA = triethyleneglycol dimethacrylate.

Specimens were stored in distilled water at 37 °C for 7 days and then submitted to pull-out test.

The pull-out test was carried out according to Guimarães et al. [13]. The roots were individually attached to a custom device in a vertical position and a ring fixed to the post was grasped by the clamping apparatus in an Instron machine (DL-1000, EMIC, São José dos Pinhais, PR, Brasil). The pull-out test was performed along the long axis of the post and the tooth at a 0.5 mm/min cross speed and 50 Kgf load cell, until dislodgement of the post from the root. The force required to dislodge each post was then recorded in MPa.

Fracture failure mode was assessed by stereomicroscopy. Adhesive, mixed and cohesive failures were used for the statistical analysis.

Data were submitted to the statistical t-test at a 5% level of significance.

## RESULTS

The application of the t-test (Table 2) showed a significant difference between the adhesive systems tested.

**Table 2 -** Mean and standard deviation values of bond strength (MPa) and results of t-test

| Group | Adhesive + Cement | Mean ± SD | t-value | p-value |
|---|---|---|---|---|
| 1 | Single Bond + Enforce | 5.28 ± 3.25 | 3.31 | 0.003* |
| 2 | One Up Bond F + Enforce | 10.05 ± 3.78 | | |

*significant difference ($p < 0.05$)

For the fracture type, in Group 1 it was observed that adhesive failures were prevalente (100%); in Group 2, 90% cohesive failure (40% in dentin and 50% in material) and 10% adhesive failure.

## DISCUSSION

The total acid etching technique with phosphoric acid at a 35 to 37% concentration for 15 s produces complete removal of the smear layer and enables the collagen fibers to be exposed, providing support for the formation of the hybrid layer, or dentin hybridization, described by Nakabayashi [14] in 1982, thus ensuring a high bond strength between dentin and restorative material [14,15].

However, the possibility of failures in hybrid layer formation can be observed [14,15]. Excessive etching enables formation of a deep zone of demineralized dentin; the adhesive may not infiltrate throughout the entire decalcified area, originating a hybrid layer with regions of exposed collagen fibers that are weakened and more susceptible to hydrolysis (weak zone) [16,17]. In addition, maintenance of ideal dentin humidity after etching is critical, because excessive drying may lead to the collapse of collagen fibers, whereas excess residual water on dentin may contribute to separation of the adhesive system phases at the time of application, or to longitudinal degradation of the restoration [18,19].

Self-etching adhesives are composed of acidic resin monomers that simultaneously promote superficial dentin demineralization and resin adhesive infiltration into the dentin tissue [19,20]. These adhesives reduce the possibility of weak zone formation, reduce the clinical steps, eliminating acid etching and washing stages, and technique sensitivity regarding dentin humidity maintenance [19,20]. The acidity of these adhesive solutions comes from the ionization of radicals present in the molecules of the hydrophilic monomer itself, also responsible for impregnating the substrate [15].

Commercial presentation of Enforce is resin cement, to be applied after the etching technique and adhesive system Prime & Bond NT application. However, this study used the total-etch Single Bond (3M ESPE) adhesive system associated with Enforce resin cement because it is a total-etch adhesive used widely in daily clinical procedures and in most researches regarding bond strength to dentin. Studies have shown that Single Bond showed higher bond strength values when compared to Prime & Bond NT adhesive [21-23].

The results of this study showed that self-etch One Up Bond F adhesive system associated with Enforce resin cement system showed higher bond strength values than the acid-etch Single Bond adhesive system associated with Enforce resin cement. Therefore, the null hypothesis was rejected.

The carboxylic acids (MAC-10) present in One Up Bond F are meant for superficial dentin demineralization, according to manufacturer's Tokuyama. Also, this self-etch adhesive has in its composition, resin monomers that promote simultaneous impregnation of the demineralized substrate, thus preventing the formation of unprotected collagen areas [15,24]. The MAC-10 molecule is composed of 10 carbon atoms, turning it in a hydrophobic monomer hydrolytically unstable [24]. Such characteristics of the One Up Bond F may explain the higher bond strength values for One Up Bond F compared to Single Bond, observed in this study.

In addition, the self-etch adhesives form a transition layer between adhesive and resin and a more uniform hybrid layer when compared to total-etch adhesives, promoting a better hybrid layer infiltrated by adhesive resin [25]. Moreover, the preservation of unaltered residual hydroxyapatite within the submicron adhesive layer may serve as receptor of additional chemical bonds with resin monomers [15,26,27].

One Up Bond F Plus is considered a self-etching adhesive of moderate aggressiveness (pH ≈ 1.2). The total-etch adhesive systems promote the formation of a thicker hybrid layer (around 4.2 $\mu$m), when compared with moderate self-etch adhesives, such as One Up Bond F Plus (with a thickness of 1.2 – 2.2 $\mu$m) [28]. However, the quality of the hybrid layer formed is the most important factor in obtaining higher bond strength values between dentin and restorative material. Various studies have shown that the thickness of the hybrid layer is not related to bond strength [29,30]. Thus, the stability of the adhesive interface and bond strength are related to a compact and homogeneous hybrid layer formation, and not to thickness itself [29,30].

The quality and longevity of the hybrid layer are directly related to a proper resin infiltration, a complete impregnation of the exposed collagen and to an adequate degree of conversion [31,32]. The self-etch adhesives promote the formation of a hybrid layer with upper and lower portion. The upper portion is a result of resin infiltration into the hybridized smear layer and the lower portion is considered the true hybrid layer formed through dentin demineralization [31]. Therefore, the demineralization is sufficient to allow penetration of the adhesive, without formation of a deep zone of demineralized dentin, avoiding regions of exposed collagen fibers.

Borges *et al.* [32] observed that One Up Bond F had a higher monomer conversion means when compared to Single Bond. A low degree of conversion of adhesives is associated with low bond strength values and mechanical properties, whilst a high degree of conversion of adhesives is an important factor in the successful adhesion to dental tissues. The results of this study are in agreement with the authors Yokota *et al.* [33] and Sensi *et al.* [34] who observed that bond strength values of Single Bond to dentin were significantly lower than One Up Bond F.

Regarding fracture type analysis, total-etch Single Bond adhesive system associated with Enforce Single Bond showed 100% adhesive failure, whilst self-etch One Up Bond F adhesive system associated with Enforce showed 90% cohesive failure. Leloup *et al.* [34] demonstrated that adhesive systems with high bond strength values present higher rates of cohesive failures. Whereas, adhesive systems that showing low bond strength values, present higher rates of adhesive failures [35].

The main factor affecting the durability of adhesive systems in vivo is hydrolysis at the components interface such as collagen and resin, and subsequent degradation products, and this contributes to the limited durability and failure of adhesive restorations, loss of retention and marginal adaptation, due to degradation over the course of time [36, 37]. Thus, longitudinal and in vivo studies are needed to evaluate the effects of bond between self-etch adhesives associated with cements and dentinal substrates.

## CONCLUSION

Considering the limits of this study it was concluded that the association self-etch adhesive system and resin cement showed significantly higher mean bond strength than the association total-etch adhesive system and resin cement for fiber glass post cementation to radicular dentin.

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