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ORIGINAL ARTICLE

Bond strength to radicular dentin and sealing ability of AH Plus in combination with a bonding agent

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Abstract

Objective. To evaluate the sealing ability and bond strength of AH Plus sealer associated with the hybridization protocol of radicular dentin with Scotchbond Multi Purpose (SB). ***Materials and methods.*** Ninety palatal roots of maxillary molars were selected and divided into three groups ($n = 30$) according to filling protocol (G1, AH Plus/Resilon; G2, SB/AH Plus/Resilon; and G3, AH Plus/Gutta-Percha). In groups in which AH Plus + resin cones were used, dentin was hybridized before applying sealing material. For the bond strength test, 60 roots of bovine teeth were selected. Six holes were made in each root, two in the cervical, middle and apical third of the root. The roles were filled with AH Plus sealer with or without an adhesive system and submitted to push out test and the fracture mode was examined using a stereomicroscope (×32). ***Results.*** Statistical analysis showed that ScotchBond Multi Purpose (SB) + Resilon cone + AH Plus group promoted higher sealing ability than the gutta-percha + AH Plus group ($p < 0.05$). Bond strength was lower with SB application than without it ($p < 0.05$). A reverse correlation was found between bond strength and sealing ability. The fracture mode methodology revealed 22.77% of adhesive, 11.67% of cohesive and 65.55% of mixture fractures for SBMP/AH Plus protocol, whereas the AH Plus protocol indicated 86.11% of cohesive and 13.89% of mixed fractures. ***Conclusion.*** It was concluded that the use of the adhesive system Scotchbond Multi Purpose improved coronal sealing ability of AH Plus, but bond strength of sealer was reduced when adhesive was applied.

Key Words: *adhesive system, bond strength, obturation, sealing*

Introduction

Bond strength of endodontic sealers to dentin is an important property of filling materials because it minimizes the risk of the detachment of the filling materials from dentin during restorative procedures [1–3] or the masticatory function [4], ensuring that sealing is maintained and, consequently, clinical success of endodontic treatment.

The association of AH plus to resin cones (Epiphany points, Pentron Clinical, Wallingford, CT) showed higher bond strength than the association of these cones with epiphany sealer [5]. However, the inability of epoxy resin to form a hybrid layer with dentin [6] would be a limiting factor to obtain a so-called 'monoblock obturation' [7]. This limitation may be overcome by applying an adhesive system before inserting the endodontic sealer [8–10].

One of the main objectives of obturation is sealing the root canal space and preventing the micro-organism contamination into the periapical region [11], thus collaborating for endodontic treatment success [12]. A tendency in Endodontics is to apply the principles of adhesion of restorative materials used in Operative Dentistry when obturating the root canal because it is believed that the adhesive systems promote better coronal and apical sealing [13]. There are few studies in the literature that correlate bond strength and quality of sealing of endodontic filling materials [14,15]. Therefore, the aim of this study was to assess the sealing ability and bond strength of AH plus sealer after hybridization of radicular dentin using the Scotchbond Multi-Purpose® (SB) adhesive system to verify the existence of a correlation among these properties.

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Materials and methods

Assessment of sealing ability

Ninety maxillary molars with completely formed roots were examined clinically using a stereoscopic lupe Leica MZ 75 (Leica Microsystems, Essen, NW, Germany) and radiography to verify if there were any cracks or fractures. Afterwards, the palatal root was sectioned with the aid of a double-faced diamond disk (KG Sorensen, Barueri, SP, Brazil) below the amelocemental limit to obtain a root remainder of 12 mm. This study was approved by the Human Research Ethics Committee of the School of Dentistry of Piracicaba (UNICAMP, Brazil).

The chemical-mechanical preparation was performed by a single operator following the technique proposed by the manufacturer of Protaper Universal® (Dentsply/Maillefer, Tulsa, OK). The working length of each root canal was established using a size 15 K-file (Dentsply Maillefer, Ballaigues, Switzerland) into the canal until visible at the apical foramen. The roots were instrumented with a standard crown-down rotary technique using Protaper Universal® to a master apical size #50. For root canal disinfection, all groups received 2% chlorhexidine gel (Endogel®, Itapetininga, SP, Brazil) before the use of each instrument, followed by 5 mL saline solution (HalexIstasr, Goiás, Brazil). Three milliliters of 17% ethylenediaminetetraacetic acid (Drogal, Piracicaba, SP, Brazil) was then used to irrigate the canal for 5 min. The root canals were dried with absorbent paper tips (Microtipped, Endo Points, Paraíba do Sul, RJ, Brazil) and the roots were divided into three groups according to the filling protocol used (G1, AH Plus/Resilon; G2, SB/AH Plus/Resilon; and G3, AH Plus/gutta-percha). The Adper Scotchbond™ multi-purpose plus (3M, St Paul, MN) adhesive system was applied on the roots in G2 before performing the endodontic obturation. The application sequence of the adhesive system for the procedures inside the root canal was performed with the aid of a super fine applicator (SDI, Melbourne, Victoria, Australia) following the manufacturer's instructions. A single resilon (Pentron Clinical, Wallingford, CT) or gutta-percha (Odous De Deus, Belo Horizonte, MG, Brazil) #50/0.06 cone was coated with AH Plus and slowly inserted into the canal to the working length. Complementing the obturation stage for all groups, a cut of 4-mm was made in the coronal portion of the cone with the aid of the 'Touch'n heat' system (SybronEndo, Orange, CA) followed by vertical condensation of the obturation with Schilder's type condenser (Odous DE Deus, Belo Horizonte, MG, Brazil). After obturation, the specimens were kept at 100% humidity for 1 week. The materials used are listed in Table I.

After 7 days the samples were fixated with cyanoacrylate gel (SuperBonder, Loctite, Diadma, SP, Brazil) to a connection platform prepared by inserting a stainless steel tube, obtained from cutting a 18G disposable needle (INJEX, Ourinhos, SP, Brazil), into a hole created in the center of an acrylic plate (20 × 20 × 3 mm), placing the metal tube in the center of the cervical portion of the root. Two coats of red nail varnish (Revlon, New York, NY) were applied to all external root surfaces to guarantee external sealing of the roots. The positive control group consisted of two specimens that were prepared and obturated with gutta-percha cones without using sealer and two teeth were obturated and received two coats of varnish to serve as a negative control.

The platform containing the sample to be tested was connected to a fluid filtration device (FLODEC, De Marco Engineering, Geneva, Switzerland), kept under constant pressure of 18 mmHg, and fluid filtration was read for 10 min for each sample. The first 2 min of reading were discarded for being the time required to stabilize the device [16]. The FLODEC system provides a rate of fluid filtration of obturation expressed in μL/min.

Bond strength test

To analyze bond strength (BS), 30 bovine teeth were cleaned with water after extraction and kept in 0.2% thymol (Dinâmica, Ribeirão Preto-SP, Brazil) under refrigeration for a maximum of 6 months.

The crowns of the teeth were separated from the roots with the aid of a double-faced diamond disk (KG Sorensen, São Paulo, SP, Brazil). Each root fragment was included in polystyrene resin and taken to the cutting machine IsoMet (Buehler, Lake County, IL) where 2-mm cuts were made to obtain 2-mm-thick dentin slices. The cuts were made parallel to the long axis of the tooth and two dentin slices were obtained per root.

The dentin slices were taken to a preparation standardizing machine and with the aid of cylindrical diamond tips (3100, KG Sorensen, São Paulo, SP, Brazil) with water refrigeration, 1.6-mm diameter holes were made in the cervical, middle and apical thirds to simulate the root canal space [17]. One hole on each side of the root canal was made, totalling two holes per third (Figure 1).

The sample were immersed in culture plate wells (TPP, Trasadigem, Schaffhausen, Switzerland) containing 5 ml of 2% chlorhexidine gel for 12 min. To remove the smear layer, each dentin slice remained in contact with 17% EDTA for 3 min and then kept in distilled water for 10 min to remove the EDTA.

The specimens were divided into two groups, as follows: G1, AH Plus; G2, SB/AH Plus). To insert the sealer into the spaces created in the radicular dentin, 3 ml disposable syringes (Injex, Ourinho, SP, Brazil)

Table I. Materials, manufacturers and compositions.

Materials	Manufacturers	Compositions
AH Plus	DeTrey-Dentsply Konstanz, Germany	Epoxide paste: Diepoxide,Calcium tungstate, Zirconium oxide, Aerosil, Pigment
		Amine paste: 1-adamantane amine, N,N′-dibenzyl-5-oxa-nonandiamine-1, 9,TCD-Diamine, Calcium tungstate, Zirconium oxide Aerosil, Silicone oil
Resilon	Pentron Clinical, Wallingford, CT	Organic part: thermoplastic synthetic polymer–polycaprolactone, Inorganic part: bioactive glass, bismuth oxychloride, barium sulfate
Guta-percha	Odous De Deus, Belo Horizonte, MG, Brazil	Organic part: gutta-percha, resins and waxes Inorganic part: zinc oxide and metal sulfates
Adper Scotchbond™ multi-purpose plus	3M, St Paul, MN	Activator: ethanol based solution of a sulfinic acid salt Primer: HEMA and a polyalkenoic acid Catalyst: BIS-GMA, HEMA and 2-hydroxyethyl ester benzoyl peroxide

HEMA, 2-hydroxyethyl methacrylate; BIS-GMA, bisphenol glycidyl methacrylate.

and 20 × 0.55 needles (Nipro medical, Sorocaba, SP, Brazil) were used. In G2, the Scotchbond Multi-Purpose® (SB) adhesive was applied using fine disposable applicators (SDI, Melbourne, Victoria, Australia). Before inserting AH Plus sealer into the dentin slice, clear plastic adhesive Con-Tact (Vulcan, Rio de Janeiro, RJ, Brazil) was used to prevent sealer flow. The specimens were kept in an oven at 37°C for 24 h for the sealer to set.

The samples were taken to a universal test machine (EMIC, São José do Pinhais, PR, Brazil) in which the push-out tests were performed at a speed of 1 mm/min until fracture occurred. Rupture strength was expressed in Newtons (N) in the EMIC machine and converted into Megapascals (MPa) using the formula MPa = N/10.05. Since each specimen had two holes per third, the test was performed in duplicate in relation to the root thirds.

After fracture of the test specimens, the roots were assessed under a stereoscopic lupe Leica MZ 75 (Leica Microsystems, Essen, NW, Germany) at 32 ×magnification and the fracture pattern was assessed using the IM 50 program. The failures were classified as adhesive (dentin interface, filling material), cohesive in sealer or mixed (involving two or more substrates).

Statistical analysis

The level of significance adopted in this study was = 0.05. The Kolmogorov-Smirnov normality test, Cochran test for homogeneity of variances and Tukey's HSD multiple comparison test (Honestly Significant Difference) were applied.

Results

Assessment of sealing ability

The sealing of group obturated with gutta-percha/AH Plus was less effective than those obturated with resin cone protocol/SBMP/AH Plus and equal or less effective than resin cone/AH Plus, as shown in Table II.

Bond strength test

The results of the analysis of variance for the bond strength test showed that the factor 'thirds' was not significant ($p = 0.5926$). On the other hand, the factor 'cement' was highly significant ($p < 0.0001$).

In Table III, the superiority in bond strength of AH Plus sealer in comparison with the association

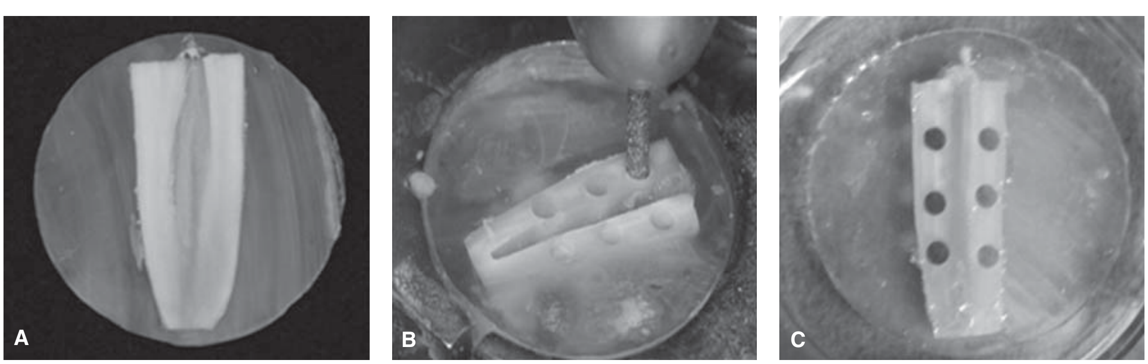

Figure 1. Sample preparation for push out test. (A) *Dentin lamina*. (B) Preparation of spaces to receive endodontic sealer. (C) Final aspect of specimen after perforations.

Table II. Comparison of means (± standard deviation) and fluid filtration (µL/min) for the different filling protocols (n = 60).

Filling protocol	Mean (± SD)
Resin cone/AH Plus	0.0559 (± 0.0179)^{ab}
Resin cone/SBMP/AH Plus	0.0508 (± 0.0174)^{b}
Gutta-percha cone/AH Plus	0.0661 (± 0.0296)^{a}

Different superscript letters indicate a significant difference among the means at a level of significance of 5% by Tukey's HSD test.

Table IV. Distribution of fracture pattern according to the cementation technique.

	Sealer cement	
Fracture pattern	SBMP/AH Plus	AH Plus
Cohesive in cement	11.67%	86.11%
Mixed/predominantly cohesive	29.44%	13.89%
Mixed/predominantly adhesive	36.11%	0%
Adhesive	22.78%	0%

between AH Plus and ScotchBond Multi-Purpose adhesive system (SBMP) is shown.

The distributions of fracture patterns assessed using the stereoscopic loupe (32×) are shown in Table IV. In the group in which only AH Plus sealer was used, a predominance of cohesive fracture in the sealer was found (86.11%), but no adhesive fractures or predominantly adhesive fractures were found in these groups. On the other hand, when the dental adhesive (SB) was applied before the endodontic sealer, most fractures were adhesive or predominantly adhesive (58.89%).

Correlation between sealing and bond strength

To verify the presence of a correlation between the sealing ability and bond strength, Person's linear correlation test was applied. The interval estimate at the level of confidence of 95% showed: 0.3587 ≤ ≤ 0.9898, showing that there is a correlation among the properties assessed.

The search for an association in the different dental treatment protocols between the mean values obtained for resistance and sealing showed a directly proportional relationship (r = 90.67%) between these two properties (Figure 2). However, one must consider that, in the case of sealing, the higher the values found, the worse is the sealing ability of the material. Therefore, the result of the correlation should be interpreted as an inverse proportional relationship in which improvement in sealing is associated with a worsening of bond strength.

Discussion

In this study two experimental models were applied to analyze coronal sealing and bond strength of different filling protocols by assessing the influence on dentin and seeking to verify the existence of a correlation among the results. In both tests, an endeavor was made to verify if applying the The Adper Scotchbond™ multi-purpose plus system before the epoxy resin-based sealer would improve the results. This assumption was based on the assertion that dentin adhesives are designed to achieve micro-mechanical and chemical bonding to dentin resulting in the formation of a hybrid layer, producing high bond strength and reducing microleakage [1].

The results of the sealing test were superior in the groups in which hybridization of radicular dentin was followed by obturation with resin cone and AH Plus sealer than in those in which the gutta-percha cones were used without applying the adhesive cement. This result suggests an advantage in applying the adhesive system before obturation of the root canal which may be explained by the possible adhesion between the epoxy resin and adhesive systems [18]. Some studies [8,10,19] confirm the results of the present study, showing the good performance of the adhesive to promote sealing of the root canal, probably due to the formation of the hybrid layer [20].

The difficulty in carrying out all steps of an adhesive system inside the root canal is an important aspect to take into consideration [21]. In the present study, the Adper Scotchbond™ multi-purpose plus was used as it is possible to apply it inside the root canal by using microbrushes, which favor a uniform distribution of the adhesive [22], and because this system has components (activator, primer and catalyzer) capable of generating chemical polymerization, making its clinical use in Endodontics feasible. The groups obturated with resin cones and hybridized with SB shown

Table III. Comparison of the means (± standard deviation) of bond strength (MPa) for the different cements (n = 180).

Sealer cement	Mean (± SD)
AH Plus	6.77 (± 2.76)^{a}
SBMP/AH Plus	3.76 (± 0.245)^{b}

Different superscript letters indicate a significant difference among the means at a level of significance of 5% by Tukey's HSD test.

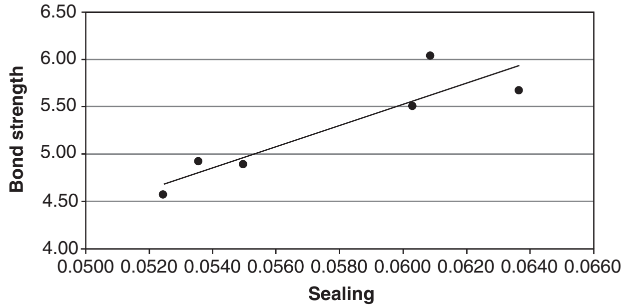

Figure 2. Correlation between sealing and bond strength.

to be more capable in promoting coronal sealing than the groups in which gutta-percha was used. The explanation may lie in the nature of the resin cone [11] which would favor the chemical bond of the resin-based sealer [5], since the bond between the gutta-percha and the sealer does not occur [23].

The modified push out model, specifically developed to assess the retentive potential of sealer to radicular dentin, was used to assess bond strength. The advantage of this model is that the compressive load is applied on the sealer and not on the resilient material, such as gutta-percha, as the latter procedure would lead to erroneous interpretations [17,24]. Previous studies revealed many micromorphology similarities between human and bovine dentin, justifying the use of bovine teeth as a substitute for human teeth in bond strength tests [25,26]. Furthermore, bovine teeth do not involve ethical issues, facilitating to obtain a larger number of specimens [27].

The bond strength test showed significantly higher values in Mpa for the groups in which the AH Plus sealer was applied without the previous application of the adhesive. The reduction in the bond strength values when AH Plus was associated with Scotchbond Multi Purpose can be explained by the lower conversion of monomers into polymers promoted by self-curing adhesives [28], which could lead to a reduction in bond strength values.

In the present study, besides the difference in bond strength, also observed was the difference in the fracture distribution for different cement protocols application. Adhesive and mixed failures were observed in cases where the dentin was hybridized; cohesive and mixed predominantly cohesive in cases where the adhesive system was not applied. Cohesive failures are often due to high strength bond between adhesive material and dentin [29]; on the other hand, adhesive failures can be explained by chemical bond failures between the adhesive and the sealer [9]. The best results for AH Plus, without the use of the adhesive system, can be justified by the greater bond strength of the epoxy resin-based sealer with the root dentin [29]. Another possible explanation for the superiority of AH Plus is the fact that this sealer is able to penetrate more deeply in the dentinal tubules than the adhesive system [30]. However, studies showed that the penetration of epoxy resin-based sealers did not increase bond strength [31] and that there are a lack of correlations between the sealer penetration into the dentinal tubules and sealability [32].

Despite considerations about the structural difference in dentin found in different portions of the root canal [33], the present study found no statistically significant differences among the three root thirds. A correlation between the sealing ability and bond strength was determined in this study and an inverse correlation was found between the two properties assessed, that is, the better the coronal sealing, the worse the bond strength. This result is contrary to the affirmation of Ørstavik et al. [34] that there is no correlation between infiltration and bond strength of the endodontic sealer to the radicular dentin. According to Schwartz [35], sealing capability should be more important in endodontics than bond strength, as a material that has a relatively lower bond strength can be considered a good endodontic material if it is capable to prevent infiltration.

It can be concluded that the use of the adhesive system associated with resin cones and epoxy resin-based endodontic sealer proved to be a good option to improve coronal sealing of the root canal. However, the use of Scotchbond Multi Purpose adhesive harmed the bond strength of AH Plus sealer.

Declaration of interest: The authors report no conflicts of interest. The authors alone are responsible for the content and writing of the paper.

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