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Influence of root canal moisture conditions on the bond strength of endodontic sealers to dentin

Influência das condições de umidade do canal radicular na resistência de união de cimentos endodônticos à dentina

Influencia de las condiciones de humedad del conducto radicular en la fuerza de unión de los selladores endodónticos a la dentina

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Abstract
Objective: To investigate the influence of intraradicular moisture conditions on the bond strength of 3 different endodontic cements in the coronal, middle, and apical thirds of the root canal. *Materials and Methods*: 90 single-rooted teeth were instrumented with a reciprocal R#50 file and randomly divided into 3 experimental groups: Dry group: dry canals with absorbent paper tips; Moist group: suction syringe and capillary tip coupled to a vacuum pump; Wet group: aspiration with endodontic suction. The groups were divided according to the endodontic sealer used: AH plus (Denstply), Hiflow (Brasseler) and Bio-c Sealer (Angelus). Obturation was performed using Tagger's hybrid technique with a single cone. The teeth were subjected to the "push-out" test to evaluate the bond strength of the cements. *Results*: Data were analyzed using the Kolgomorov-Smirnoff normal distribution test and the Mann Whitney U test to compare means within and between groups. The standard error established was 5% at a significance level of 95%. AH plus showed better results regardless of canal moisture conditions. Hiflow showed better bond strength in the dry group compared to the moist group ($p=0.023$) in the cervical area. Bio-C Sealer showed no statistically significant differences ($p > 0.05$). *Conclusion*: Moisture conditions affected the bond strength values of endodontic cements. The prevalence of failure moduli was mixed in all root thirds, regardless of the cement used.
Keywords: Root canal filling materials; Bond strength; Dental stress analysis.

Resumo
Objetivo: Investigar a influência das condições de umidade intrarradicular na resistência de união de 3 diferentes cimentos endodônticos nos terços coronal, médio e apical do canal radicular. *Materiais e Métodos*: 90 dentes uniradiculares foram instrumentados com lima recíproca R#50 e divididos aleatoriamente em 3 grupos experimentais:

Grupo Seco: canais secos com pontas de papel absorvente; Grupo úmido: seringa de sucção e ponta capilar acoplada a uma bomba de vácuo; Grupo úmido: aspiração com aspiração endodôntica. Os grupos foram divididos de acordo com o cimento endodôntico utilizado: AH plus (Denstply), Hiflow (Brasseler) e Bio-c Sealer (Angelus). A obturação foi realizada pela técnica híbrida de Tagger com cone único. Os dentes foram submetidos ao teste de "push-out" para avaliar a resistência de união dos cimentos. *Resultados*: Os dados foram analisados pelo teste de distribuição normal de Kolgomorov-Smirnoff e pelo teste U de Mann Whitney para comparação de médias intra e intergrupos. O erro padrão estabelecido foi de 5% ao nível de significância de 95%. O AH plus apresentou melhores resultados independentemente das condições de umidade do canal. Hiflow apresentou melhor resistência de união no grupo seco comparado ao grupo úmido (p=0,023) na região cervical. Bio-C Sealer não apresentou diferenças estatisticamente significativas (p > 0,05). *Conclusão*: As condições de umidade afetaram os valores de resistência de união dos cimentos endodônticos. A prevalência de módulos de falha foi mista em todos os terços radiculares, independente do cimento utilizado.
Palavras-chave: Materiais obturadores do canal radicular; Força de ligação; Análise do estresse dentário.

Resumen
Objetivo: Investigar la influencia de las condiciones de humedad intrarradicular en la fuerza de unión de 3 cementos endodónticos diferentes en los tercios coronal, medio y apical del conducto radicular. *Materiales y Métodos*: Se instrumentaron 90 dientes uniradiculares con lima recíproca R#50 y se dividieron aleatoriamente en 3 grupos experimentales: Grupo seco: conductos secos con puntas de papel absorbente; Grupo húmedo: jeringa de succión y punta capilar acoplada a una bomba de vacío; Grupo húmedo: aspiración con succión endodóntica. Los grupos se dividieron según el sellador endodóntico utilizado: AH plus (Denstply), Hiflow (Brasseler) y Bio-c Sealer (Angelus). La obturación se realizó mediante la técnica híbrida de Tagger con un solo cono. Los dientes se sometieron a la prueba de "empuje hacia afuera" para evaluar la fuerza de unión de los cementos. *Resultados*: Los datos se analizaron utilizando la prueba de distribución normal de Kolgomorov-Smirnoff y la prueba U de Mann Whitney para comparar las medias dentro y entre los grupos. El error estándar establecido fue del 5% a un nivel de significación del 95%. AH plus mostró mejores resultados independientemente de las condiciones de humedad del canal. Hiflow mostró una mejor fuerza de unión en el grupo seco en comparación con el grupo húmedo (p = 0,023) en el área cervical. Bio-C Sealer no mostró diferencias estadísticamente significativas (p > 0,05). *Conclusión*: Las condiciones de humedad afectaron los valores de resistencia adhesiva de los cementos endodónticos. La prevalencia de los módulos de falla fue mixta en todos los tercios radiculares, independientemente del cemento utilizado.
Palabras clave: Materiales de obturación del conducto radicular; Fuerza de unión; Análisis del estrés dental.

1. Introduction

Complete filling and sealing of the root canal system after the instrumentation and shaping steps is critical to the success and longevity of endodontic treatment (Craveiro et al. 2015). Due to the complex anatomy of the canal system, filling cements must have properties that allow them to fill irregularities and penetrate the dentinal tubules. The ideal cement should promote a three-dimensional seal, be biocompatible, have low cytotoxicity, adhere well to dentin walls, be insoluble in tissue fluids, not shrink after curing, be antibacterial, and have good flowability (Grossman & Meiman, 1992).

AH Plus (Dentsply De Trey GmbH, Constance, Germany) is an epoxy resin-based cement that is often used as a control group in scientific studies (Saleh et al. 2003, Dias et al. 2014). It is considered the gold standard due to its low solubility and long-term dimensional stability (Resende et al. 2009, Borges et al. 2012).

Calcium silicate- based cements are characterized by good biocompatibility properties and are the only ones with bioactive properties, as they induce the deposition of hard tissue through the formation of hydroxyapatite. They are also radiopaque, expand after the material hardens, are easy to handle, have antibacterial properties, and their smaller particles promote good flow. Unlike other cements, calcium silicate- based cements are hydrophilic, i.e., they are moisture tolerant, which is a significant advantage because moist tissue can affect the properties of the materials and, consequently, the sealing and adequate filling of the canals. (Almeida et al. 2017).

The EndoSequence BC Sealer Hiflow (Brasseler, USA) is a CSC and was developed with special attention to thermal sealing. According to the manufacturer, BC Sealer HiFlow has reduced particle size, a higher concentration of zirconia to increase radiopacity, and lower viscosity when heated. The setting time is about four hours at room temperature, but it can take up to 10 hours in dry canals (Al-Haddad & Aziz, 2016).

Bio-C Sealer (Angelus Ind. Prod. Odontológicas, Londrina, Brazil) is composed of tricalcium silicate and dicalcium silicate, which promote greater resistance of the material. It also consists of tricalcium aluminate and calcium oxide, which are responsible for the release of calcium ions, and zirconium oxide, which is free of eugenol (Zordan-Bronzel et al. 2019). According to the manufacturer, the curing time is approximately 240 minutes, depending on the local humidity, and it is not necessary to leave intraradicular moisture, as the curing of the material is triggered by moisture from the apical tissues and dentinal tubules.

The adhesion of endodontic cements can be affected by the moisture state of the intraradicular dentin, which can directly affect the process of microleakage. Therefore, the protocol for canal drying must be established to allow better adhesion between the dentin and the filling cements. Although absorbent paper tips are the first option for drying the root canal, there are also suction tips (capillary tips) with vacuum adapters that help to aspirate root canal fluids (Engel et al. 2005; Nagas et al. 2012; Prado et al. 2013).

In the present study, the bond strength of the cements was evaluated and compared: Hiflow, BioCSealer and AH Plus by the push-out test in the three thirds of the root canal subjected to different intraradicular drying protocols, using the tagger hybrid technique with a single cone of gutta-percha after instrumentation.

2. Methodology

Selection and preparation of specimens:

This work was approved by the Human Subjects Ethics and Research Committee of the local Dental Research Center under CAAE 35321820.4.0000.5374. For the study, 90 recently extracted, single-rooted and straight teeth with fully developed roots, no previous endodontic treatment, and no fractures or calcifications were collected. The teeth were stored in a 2% thymol solution (Lenza Farma, Farmacia de Manipulação, Belo Horizonte - MG, Brazil). Then, they were standardized at a root length of 15 mm by cutting with a double-sided diamond disk coupled with a straight mandrel and a micromotor and cooled with air/water spray. These measurements were confirmed with a digital caliper. Patency was confirmed with a #10 K file (Dentsply Maillefer, Ballaigues, Switzerland) and periapical radiographs with a digital sensor (Carestream, New York, USA). Instrumentation was performed with a hand file to the #25 K-file and with the Reciproc R#50 reciprocal instrument (VDW-Silver, Munich, Germany) to the working length (1 mm short of the apical foramen). The canals were irrigated with NavTips (Ultradent, South Jordan, USA) at each instrument change, totaling 20 mL of 2.5% sodium hypochlorite NaOCl- (Lenza Farma, Farmácia de Manipulação, Belo Horizonte - MG, Brazil). The final rinse was with 3mL of 17% liquid EDTA (Lenza Farma, Fármacia de Manipulação, Belo Horizonte- MG, Brazil) in three cycles, each cycle stirring 1mL of EDTA with Easy Clean (Bassi/Easy Equipamentos Odontológicas, Belo Horizonte- MG /Brazil) for 20s and renewing after 40s; and later a rinse with 6 mL of sodium hypochlorite, shaken in the same way as the previous solution.

Experimental groups:

After final irrigation, the canals were randomly divided into 3 experimental groups to test the effect of different intracanal humidity conditions previously defined by Zmener et al (Zmener et al. 2008) with some modifications: DRY Group (S): canals were dried with absorbent paper tips until the last absorbent paper tip was completely dry (observed by visual inspection); WET Group (U): aspiration with a syringe and a 0.014 capillary needle tip coupled to a vacuum pump for 5 seconds at a distance of 4 mm from CT + 1 absorbent paper tip; WET Group (M): aspiration with endodontic suction at the mouth of the canal for 5 seconds;

For each moisture condition, samples were further subdivided by endodontic sealer: AH Plus Jet, Hiflow, and BiocSealer. Gutta-Percha ProTaper F3 (Dentsply, Maillefer, Bailagues, Switzerland) was used in all groups. Thus, we had 9 experimental groups (n=10):

AHS group- Completely dry canals with absorbent paper tips and filled with AH Plus;

AHU group- canals are partially dried with a capillary tip coupled to a vacuum pump for 5 seconds + 1 absorbent paper tip and filled with AH Plus;

AHM group- the canals are suctioned with an endodontic suction device at the mouth of the canal for 5 seconds and filled with AH Plus;

HFS group- canals are completely dried with absorbent paper tips and filled with HiFlow;

Group HFU- Canals are partially dried with a capillary tip coupled to a vacuum pump for 5 seconds + 1 absorbent paper tip and filled with HiFlow;

HFM group- Canals are suctioned with an endodontic suction device at the mouth of the canal for 5 seconds and filled with HiFlow;

BCS group- canals are completely drained with absorbent paper tips and filled with Bio-C Sealer;

BCU group - the canals are partially dried with a capillary tip connected to a vacuum pump for 5 seconds + 1 absorbent paper tip and filled with Bio-C Sealer;

BCM group - the canals are suctioned with an endodontic aspirator at the mouth of the canal for 5 seconds and filled with Bio-C Sealer;

Root canals were obturated with the Tagger hybrid technique (using the Mcspadden instrument #40 at 3 mm from the apex). Subsequently, the specimens were stored in a humid oven at 37°C and 100% humidity for 7 days to ensure curing of the materials.

Resistance evaluation

Each root was cut into 2.00-mm-thick slices from the coronal to the apical third by cross-sectioning with a diamond disk at a speed of 300 rpm under cooling to avoid burning the specimens. The first and last slices were discarded, and the second slice of each third was selected for the push-out test. The specimens were positioned next to the EMIC DL200 testing machine (EMIC - São José dos Pinhais / PR /Brazil). After the mechanical test, the program generated the maximum force data in N and in KgF when the root canal filling material was displaced from the apical to the coronal direction. The bond strength (RU) at fracture was calculated in megapascals (MPa) by dividing the load in Newtons (N) by the area of the bonded surface. The bonded area of each section was calculated using the following formula: σ=F/ SL, and the force in kilonewtons (KN) required to displace the filler material was converted to stress (σ) in megapascals (MPa) by dividing the value of the displacement force (F) by the bonded area of the filler material (SL) in mm 2. To calculate the approximate value of the area, the formula SL = π (R + r) g was used, where: SL = cement adhesion area; π= 3.14; R= average radius in millimeters of the coronal part of the disk; r= average radius in millimeters of the apical part of the disk; g= relative disk height in millimeters.

Analysis of the fault modules

The defects of each slice were analyzed with a microscope at up to 40X magnification. The observed defects were classified according to a study by Zmener et al. (Zmener et al. 2008) to one of five subtypes: (a) Adhesive to dentin-when the filling material detached from the dentin; (b) Adhesive to filling material-when the gutta-percha detached from the endodontic cement; (c) Mixed-when the cement detached from both the dentin and the gutta-percha; (d) Cohesive in dentin-when a fracture occurred in the dentin; (e) Cohesive in filling material-when a fracture occurred in the endodontic cement.

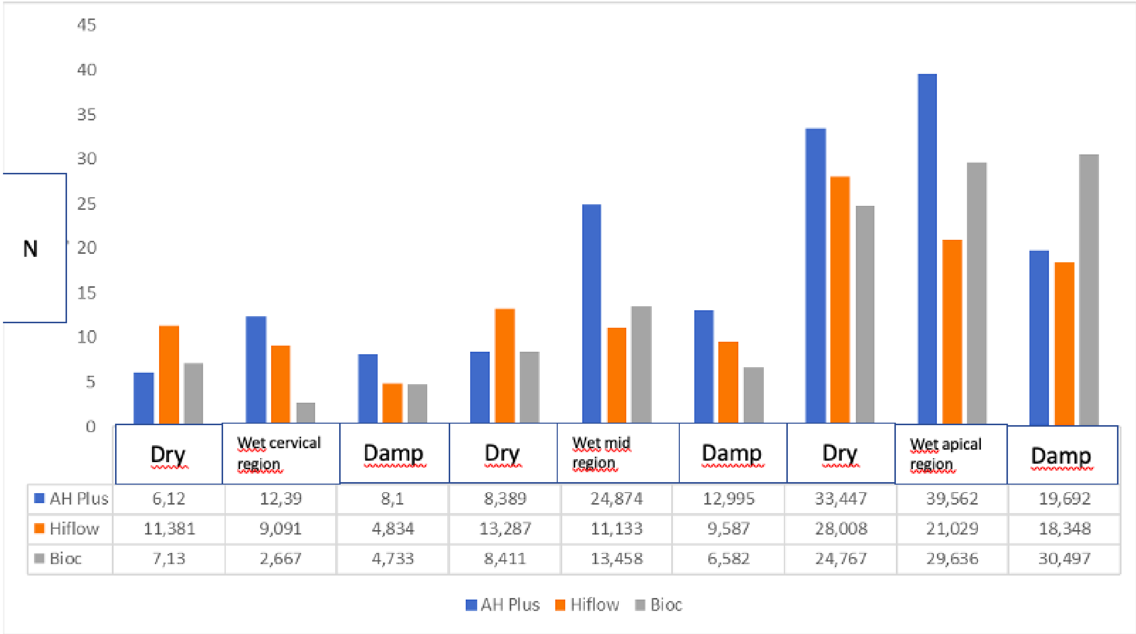
Statistical analysis

The Kolgomorov-Smirnoff test for normal distribution was performed. All numerical variables showed a non-normal distribution. To compare means between groups, Mann Whitney U analysis was used. The standard error set was 5% at a significance level of 95%.

3. Results

Graph 1 shows the results of the average maximum force (in N) of the RU test performed on the specimens with the different cements and different drying protocols.

Graph 1- Results of the average maximum force (in N) of the RU test performed on the specimens with the different cements and different drying protocols.



Source - survey data.

When comparing the average values of AH plus between dry and wet media, a statistically significant difference was observed in the cervical region (p = 0.04), the middle region (p = 0.001). There was also a statistically significant difference between the mean values of wet and moist environments in the middle region (p = 0.03). For the Hiflow, there was a statistically significant difference between the mean values of force in the cervical region between the dry and wet environments (p = 0.023). For the Bio-C sealer, no statistically significant differences were found in the mean forces between the canal drying protocols (p > 0.05). The results that showed statistical differences between the media in the same cement are marked with an asterisk in the value boxes.

When comparing the different cements but under the same moisture conditions, a significant difference in strength was found when analyzing the dry and wet media. In the wet group, there was a statistically significant difference in the mean values of the strengths between the AH Plus and the Hiflow (p = 0.01) and between the AH plus and the Bio-C Sealer (p = 0.01). These data were presented in Graph 1. The results that showed statistical differences between the cements with the same drying protocol are marked with an asterisk in the columns of the graphs.

Table 1 shows the classification of fractures at different drying protocols and different cements in all samples of the study. In the analysis of the AH Plus cement, the most common fracture was mixed and cohesive in the dentin (n=27 - 30%).

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For the Hiflow cement, the most common fracture was mixed fracture with 38 cases (42.2%). Finally, for the Bio-C cement sealer, the most common fracture was also mixed fracture, occurring in 37.8% of cases (n = 34).

Table 1 – Distribution of fractures of specimens subjected to resistance test in different endodontic cements.

Fracture	**AH Plus**		**Hiflow**		**Bio-C**	
	N	**%**	**N**	**%**	**N**	**%**
Dentin adhesive	22	24.4	17	18.9	17	18.9
Adhesive mixed with the restorative material	2	2.2	6	6.7	7	7.8
Adhesive to restorative and cohesive to dentin	22	24.4	38	42.2	34	37.8
Adhesive and cohesive in dentin	1	1.1	1	1.1	2	2.2
Adhesive to the restorative and cohesive to the filling material	8	8.9	3	3.3	5	5.6
Mixed and cohesive in dentin	4	4.4	9	10.0	10	11.1
Mixed and cohesive in the filling material	27	30.0	16	17.8	14	15.6
Fracture	4	4.4	0	0	1	1.1

Source: survey data.

4. Discussion

Moisture conditions affected the bond strength values of the endodontic cements evaluated by the push-out test. Therefore, the null hypothesis was rejected. For the failure modules, the null hypothesis was accepted because mixed fractures occurred in all groups. The results showed that the AH Plus had a greater push-out force in the middle third in the "wet" condition than the Hiflow and Bio-C Sealer.

This result can be explained by the low solubility of AH Plus, which has better long-term dimensional stability and microretention in dentin compared to other cements, thereby increasing bond strength. For these and other properties, AH Plus is considered the gold standard (Resende et al. 2009; Borges 2012; Almeida et al. 2017). In addition, a previous study suggests that the adhesion of AH Plus to dentin is not only due to its mechanical microretention, but also to its potential to chemically bind to the collagen matrix of the dentin tissue (Fisher et al. 2007).

Another factor could be the swelling of the epoxy component of AH Plus after water absorption, which may increase the resistance to displacement during the push-out test (Fernández-García & Chiang, 2002). This result is consistent with previous studies showing that AH Plus has higher bond strength than various endodontic cements, including some bioceramics (Almeida et al. 2017; Vilanova et al. 2012).

Different amounts of residual moisture in root canals alter the sealing properties of conventional cements, epoxy-based cements, and calcium silicate-based cements. On the other hand, even though bioceramic cements are hydrophilic, there are no clear instructions from any manufacturer to achieve an ideal level of residual moisture in the canal (Dias et al. 2014; Nagas et al. 2012). Excessive drying may remove the water present in the dentinal tubules, which in turn may hinder the effective penetration of hydrophilic cements and affect the quality of adhesion (Nagas et al. 2012; Padro et al. 2013).

In the push-out test, the inner root dentin is used as a substrate and the filling material provides the bond strength of the filling cement as it moves (Fisher et al. 2007; Goracci et al. 2004; Taşdemir et al. 2014; Gade et al. 2015). The bonding potential of AH Plus to dentin is satisfactory under intracanal moisture conditions, but under extreme moisture conditions - wet and dry - cement bonding is severely compromised (Nagas et al. 2012). Hiflow showed lower bond strength in the wet environment than the dry group in the middle third. This result contrasts with some previous studies that showed that the removal of water reduces the bond strength of bioceramic cements to root dentin (Nagas et al. 2012; Taşdemir et al. 2014; Paula et al. 2016). Although bioceramic cements are hydrophilic, their hydrophilicity is not sufficient to displace water in a fully moist root canal, and the resulting entrapment of water droplets between the cement-dentin interface would lead to fracture of the bond, reducing the strength of the bond (Zmener et al. 2008).

Bioceramic cements cure in the presence of intraradicular moisture. However, when the canal is completely dried and thus "dry", moisture may still be present in the dentin tubules, since dentin is 20% water (by volume), which is already sufficient for the setting reaction of dentin. bioceramic cements (Razmi et al. 2016). Another possible explanation for this result is that thermoplastic techniques, if used, may affect adhesion due to drying of the dentin tubules. Moisture is essential for cement to set because it is hydrophilic. However, according to the manufacturer, Hiflow cement was developed for use with thermal filling techniques. Therefore, the result of this study shows that thermoplasticization did not change the bond strength even in the "dry" state.

Comparing Endosequence BC, AH Plus, and Endomethasone-N cements using lateral condensation and thermoplasticization techniques, showed that the AH Plus group had higher push-out strength compared to the other cements with the two filling techniques (Gade et al.). The Bio-C Sealer cement did not show significant differences between the different moisture conditions with the thermoplastic technique, which may be beneficial in clinical practice due to the different degree of residual moisture in the different regions of the root canal (Nagas et al. 2012; Zmener et al. 2008). HiFlow, Endosequence and AH Plus can be used with thermal obturation techniques without abrupt changes in their physical properties, including setting time, weight loss and chemical properties (Aksel et al. 2021).

In this study, the push-out method was used to evaluate bond strength, as previous studies have shown that this is the test with significant precision values to measure the bond strength of restorative materials to root canal dentin (Fisher et al. 2007; Taşdemir et al. 2014; Gade et al. 2015). The AH Plus had a higher average bond strength than the bioceramic cements regardless of moisture status. It was hypothesized that bioceramic cements, being hydrophilic, would have better bond strength results under "moist" and perhaps even "wet" conditions. This may accurately represent the limitations of this study and previous studies and especially in clinical practice where the goal is to standardize residual moisture in all thirds of the root canal (Gesi et al. 2005). However, adhesion is necessary to maintain the integrity of the cement-dentin interface during mechanical stresses caused by tooth flexion, surgical procedures, or subsequent preparation of the post space, so that adhesion fractures do not occur, causing markings in the filling, which can lead to possible microleakage. and failures in endodontic treatment (Carvalho-Junior Jr et al. 2007; Camilleri et al. 2011).

Clinically, failure of the connection may occur due to mechanical stress from tooth flexion or during root preparation for post placement (Babb et al. 2009). The predominant fracture in all cements, regardless of moisture status in the cervical and middle thirds, was a mixed fracture in which the cementum was displaced by both dentin and gutta-percha. In the apical third, the predominant fractures were mixed and cohesive dentin for all cements and regardless of moisture, although dentin fractures also occurred.

Although the gutta-percha cone has a diameter and taper compatible with that of the instrument used to prepare the canal, there is a larger free area between the dentin and gutta-percha cone in the cervical area than in the other thirds when using the single-cone technique. Consequently, not only is the thickness of the endodontic cement greater in this space, but the distribution of this material along the other thirds becomes more difficult due to the smaller dimension, which promotes even distribution. In the same disk, there may be a greater amount of cement between the cone and the root canal than in other regions. It is therefore assumed that, due to the inadequacy of the drying process in general, which is associated with the irregular distribution of the cement along the dentin walls, in the same section the cement could be displaced from the dentin wall in one region and displaced in another of the gutta-percha cone (Babb et al. 2009).

In this study, when the push-out test was performed, the tips were not exchanged to evaluate the resistance of the filling material in the different thirds, which may have led to discrepant results because the apical diameter is smaller than the cervical and average diameters, so the force applied to the material was not as homogeneous as possible. The prevalence of cohesive fractures in the dentin in the apical third may be justified (Dias et al. 2014). In all the cements tested, the "wet" condition was found to have the lowest bond strength values. However, when the laboratory test was performed, it became clear that the capillary tip produced more efficient and faster drying than the paper tips. The greatest differences were observed in the middle and cervical thirds, which may further support this hypothesis since the capillary tip was not inserted into the apical third of the roots, but 4 mm anterior to working length. In conjunction with the absorbent paper tip, the residual moisture could be lower than in the 'dry' group itself, which was performed by visual inspection until the last paper tip was obviously dry. Only ready-to-use, one-paste filling cements were selected in this study. This may have had a positive effect on the results as it minimized possible inappropriate ratios of other cements such as paste/paste or powder/paste as used in other studies (Dias et al. 2014; Nagas et al. 2012; Paula et al. 2016).

5. Conclusion

Humidity affected the bond strength values of the endodontic cements evaluated with the push-out test. The AH Plus achieved better adhesion to dentin walls than the HiFlow and Bio-C Sealer. The prevalence of failure modules that occurred in all root thirds was mixed fracture, regardless of the cement used.

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