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Solubility, Porosity, Dimensional and Volumetric Change of Endodontic Sealers

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The aim of this study was to evaluate physical properties of endodontic sealers (AH Plus, MTA Fillapex and Endofill), by conventional and micro-CT tests. Dimensional stability was evaluated after immersion of materials in distilled water for 30 days. Solubility and volumetric change were evaluated after 7 and 30 days of immersion in distilled water. Solubility was evaluated by means of mass loss and volumetric change was assessed by micro-CT. Porosity was evaluated under a microscope after 7 days of immersion in distilled water, and by using micro-CT after setting and immersion in distilled water for 7 and 30 days. Statistical analysis was performed by ANOVA and Tukey's test with 5% significance level. MTA Fillapex presented the highest solubility ($p<0.05$), showing values above the ISO/ADA recommendations. MTA Fillapex presented higher volumetric and dimensional changes, followed by Endofill and AH Plus ($p<0.05$). Dimensional stability of the MTA Fillapex and AH Plus did not follow ISO/ADA standards. The highest total porosity was observed for MTA Fillapex ($p<0.05$). Endofill had higher total porosity than AH Plus according to microscope evaluation ($p<0,05$), and both sealers were similar in micro-CT assessment ($p>0,05$). In conclusion, MTA Fillapex presented higher solubility, dimensional and volumetric change besides porosity compared to the other evaluated sealers. The assessed physical properties of sealers are related, and the different tests provided complementary data. Micro-CT is a valuable method for assessment of physical properties of endodontic materials.

Key Words: endodontics, x-ray microtomography, physicochemical analysis, silicate cement.

Introduction

The solubility of endodontic sealers may influence the success of treatment (1). According to American Dental Association (ADA) (2) and International Organization for Standardization (ISO) (3), root canal sealers should exhibit solubility less than 3%. Dimensional change of endodontic materials may compromise the sealing of the root canal (4). Based on ISO and ADA this change should not exceed 1.0% in contraction or 0.1% in expansion.

Another important physical property is the porosity, which may reduce the materials hardness and strength (5). The porosity of cements may be evaluated by using a high-pressure mercury intrusion porosimeter (6); by using immersion tests based on Archimedes principle defined in ASTM C830 Standard (7), or by (optical) light microscopy (8). However, these analyses often lead to conflicting results (9).

Root canal sealers are classified according to their chemical composition. AH Plus® (Dentsply, DeTrey GmbH, Konstanz, Germany) is an epoxy resin-based sealer that has been used in comparative studies (7). AH Plus presents low dimensional change and solubility (10). Calcium silicate-based sealers have been proposed in endodontics due to their biological properties. MTA Fillapex® (Angelus, Londrina, PR, Brazil), although including mineral trioxide aggregate (MTA) and presenting low toxic effects (11), this material is also a resin-based sealer and its main chemical component is salicylate resin. MTA Fillapex exhibits high solubility and dimensional change (12,13). Endofill (Dentsply Indústria e Comércio Ltda., Petrópolis, RJ, Brazil) is a well-established zinc oxide and eugenol-based sealer that has dimensional stability, but high solubility (14,15).

The in vitro methods proposed by ADA and ISO to assess solubility and dimensional stability do not replicate the clinical situation. New methodologies, such as micro-computed tomography (micro-CT) have been proposed to analyse dimensional stability and solubility of endodontic sealers (16,17). Micro-CT may also be used for evaluating the porosity and size of pores within a material (18).

Based on the limitations of conventional tests and lack of sufficient information in the literature regarding the association between the physical properties of endodontic sealers and the methodologies to assess these properties, the aim of this study was to evaluate the solubility, dimensional and volumetric change besides porosity of AH Plus, MTA Fillapex and Endofill by using conventional tests, complemented with micro-CT analyses.

Materials and Methods

The evaluated materials were AH Plus (Dentsply, DeTrey GmbH, Konstanz, Germany), Endofill (Herpo Dental Products Ltda, Petrópolis, RJ, Brazil) and MTA Fillapex (Angelus, Londrina, PR, Brazil). The sealers were manipulated according to the manufacturers.

Solubility

Solubility test was evaluated based on a previous study (14). Specimens of each material measuring 1.5 mm high and 7.75 mm in internal diameter were fabricated (n=6) with a nylon thread and kept in an oven at 37 °C for 7 days. The specimens were weighed on a precision balance (Adventurer AR2140, Ohaus Corporation, Parsippany, NJ, USA). Then, the samples were suspended and fixed by means of nylon threads inside plastic flasks containing 7.5 mL of distilled, and kept in an oven at 37 °C for 7 days. The specimens were removed from the distilled water, dried with absorbent paper, and placed in a dehumidifying chamber until the mass was stabilized. New samples were prepared and kept immersed in distilled water for 30 days (n=6). The loss of mass was expressed as a percentage of the original mass. The percentage of solubility was calculated as follows: (IM-FM)/IM*100

where IM is the initial mass and FM is the final mass of the specimen after 7 and 30 days of immersion in distilled water.

The test was repeated 3 times. In accordance with ISO and ANSI/ADA, the solubility should be less than 3%.

Dimensional Stability

Dimensional stability of the materials was evaluated as previously described (14). Eight specimens measuring 3.58 mm in height and 3 mm in diameter were made from each material. Their surfaces were polished with 600-grit wet sandpaper. The initial length of each specimen was measured with a digital caliper (Mitutoyo). The specimens were then stored in flasks containing 2.24 mL distilled water at 37 °C for 30 days. Afterwards, they were removed from the flasks, dried with absorbent paper, and their final lengths were determined. The percentage of dimensional change was calculated as follows:

[(L30-L)/L] x 100

where L is the initial length of the specimen and L30, the length after 30 days.

The test was repeated 3 times. In accordance with ISO and ANSI/ADA, the results must not exceed 1.0% of contraction or 0.1% of expansion.

Volumetric Change

Volumetric change of the sealers was analyzed using micro-CT (SkyScan 1176, Bruker-MicroCT, Kontich, Belgium), based on a previous study (17). Transparent acrylic resin-based models were fabricated using metal molds with cavities measuring 3 mm deep and 1 mm in diameter (n=6). The cavities were filled with each material by a single operator, who was previously trained and calibrated. The samples were kept in an oven at 37 °C and relative humidity for three times the duration of their setting time, and scanned by using micro-CT (Bruker-MicroCT, Kontich, Belgium). The samples were scanned again at 7 and 30 days, and were kept immersed in distilled water between these experimental time intervals. The scanning procedure was performed using 50 kV X-ray tube voltages and 500 µA anode current; aluminum filter of 0.5; isotropic voxel of 18 µm; and an evolution cycle of 360°. Each scanning operation consisted of 721 images in TIF format. These images were used for quantitative analysis of the samples, allowing the total volume of material to be calculated in mm^3.

Reconstruction of the images was performed using NRecon software (V1.6.4,7; (Bruker-MicroCT, Kontich, Belgium). The correction parameters for smoothing, beam hardening and ring artefacts were defined for each material (the parameters for AH Plus were 1 for smoothing, 80 for beam hardening correction and 2 for ring artefacts correction, for Endofill were 0 for smoothing, 47 for beam hardening correction and 1 for ring artefacts correction and for MTA Fillapex, 0, 57 and 1, respectively). The same parameters were used for the same materials in the different periods. The reconstructed images were superimposed in the different periods and saved in the coronal, sagittal and transaxial planes by using the Data Viewer program (V1.5.2.4; Bruker-MicroCT, Kontich, Belgium). The images were analyzed using CTAn software (V1.11.8; Bruker-MicroCT, Kontich, Belgium). The volume filled by the sealers was calculated at each time interval.

Porosity Analysis by Microscope

The porosity was evaluated based on a previous study (8). The material microstructure was observed by using an inverted digital microscope (MIC-D Olympus, Philippines) on rectangular specimens (n=6) measuring 8 x 10 mm and 5 mm high. The specimens were prepared and then stored for 7 days at 37°C and 95% humidity. The specimens were stored in distilled water for 7 days and sectioned in half along their cross section with a microtome cutter (Isomet 1000. Buehler Ltd, Lake Bluff, IL, USA). They were subsequently polished using fine grit silicon carbide abrasive paper. The specimen surfaces were observed using the inverted digital microscope at 50× and 200× magnification. The images were captured and analyzed qualitatively and quantitatively for the presence of pores. Quantitative analysis of pores was performed by means of the software Image Tool version 3.0 (UTHSCSA, San Antonio, TX, USA). The surface

of the material was divided into four parts, and each part was individually analyzed at the two magnifications used.

Porosity Analysis by Micro-CT

Porosity analysis by micro-CT was performed, based on previous study (18). Cylindrical test-specimens measuring 4.0±0.1 mm thickness and 7±0.1 mm in diameter were fabricated (n=6). The sealers were mixed in accordance with the manufacturer's instructions, placed in the molds and stored for 7 days at 37 °C and 95% humidity.

The samples were examined by means of micro-CT (SkyScan 1176) after setting and after immersion in distilled water for 7 and 30 days. The initial porosity of the materials and after the contact with the aqueous solution was evaluated. The scanning parameters were: voltage 80 kv, 313 µA current, pixel size 9 µm and 360° rotation using a Cu + Al filter. These images were used for quantitative analysis of the samples, allowing the porosity of the material to be calculated in mm^3 and percentage. Reconstruction of the images was performed using NRecon software (V1.6.4,7; Bruker-MicroCT).

The correction parameters for smoothing, beam hardening and ring artefacts were defined for each material (the parameters for AH Plus were 7 for smoothing, 70 for beam hardening correction and 8 for ring artefacts, for Endofill were 3 for smoothing, 70 for beam hardening correction and 8 for ring artifact correction, and for MTA Fillapex they were 1, 97 and 7, respectively). The same parameters were used for the same materials in the different periods. The reconstructed images were superimposed in the different periods and saved in the coronal, sagittal and transaxial planes by using the Data Viewer program (V1.5.2.4; Bruker-MicroCT). The images were analyzed using CTAn software (V1.11.8; Bruker-MicroCT). Open, closed and total porosity values were evaluated. A 3D model of the filled cavities was obtained by the CTAn software and visualized and saved using CTVol program (V2.0; Bruker-MicroCT).

Statistical Analysis

The results obtained for all the tests were submitted to a normality test, and then to the parametric ANOVA statistical test and the Tukey multiple comparison test, with 5% significance level.

Results

Dimensional Stability And Solubility

MTA Fillapex exhibited the highest dimensional change value, and AH Plus, the lowest ($p<0.05$). The AH Plus expansion and the contraction of the MTA Fillapex were higher than the limits set by the ISO standard. Endofill complied with the ISO limits. At 7 and 30 days, the solubility was higher for MTA Fillapex ($p<0.05$). The data is shown in Table 1.

Volumetric Change

MTA Fillapex presented the highest volumetric change after 7 and 30 days, followed by Endofill and AH Plus ($p<0,05$). After 7 days, AH Plus presented a volume increase while Endofill and MTA Fillapex showed a volume reduction. All the evaluated materials presented a decrease in volume at 30 days. The data is shown in Table 2.

Porosity by Microscopy

The porosity values were higher for MTA Fillapex, followed by Endofill, and lowest for AH Plus ($p<0.05$) (Table 3). Images captured at 50x magnification may be observed in Figure 1.

Porosity by Micro-CT

The porosity values observed in micro-CT are shown in Table 4. The open and total porosity values were higher for MTA Fillapex until 7 days ($p<0.05$). After 30 days, MTA

Table 1. Means and standard deviation of dimensional stability and solubility (%) of the root canal sealers

	AHP	EDF	MTAF
Dimensional stability	0.56 (0.17)[c]	-0.76 (0.14)[b]	-1.69 (0.24)[a]
Solubility 7 days (mass loss)	-0.11 (0.13)[b]	1.59 (0.17)[b]	22.03 (2.00)[a]
Solubility 30 days (mass loss)	-0.40 (0.27)[b]	2.49 (0.62)[b]	25.63 (5.84)[a]

[a,b,c]Different letters indicate statistically significant difference between experimental groups ($p<0.05$).

Table 2. Means and standard deviation of volumetric change (%) for the evaluated materials after 7 and 30 days by means of micro-CT

	AHP	EDF	MTAF
Volumetric Change 7 days	0.32 (0.13)[c]	-0.90 (0.45)[b]	-11.14 (1.10)[a]
Volumetric Change 30 days	-0.60 (0.31)[c]	-6.84 (2.40)[b]	-13.66 (2.55)[a]

[a,b,c]Different letters indicate statistically significant difference between experimental groups ($p<0.05$).

Table 3. Means and standard deviation of porosity (number of pores) of the root canal sealers

	AHP	EDF	MTAF
Number of pores	16.17 (4.36)[c]	42.67 (12.60)[b]	103.7 (32.54)[a]

[a,b,c]Different letters indicate statistically significant difference between experimental groups ($p<0.05$).

Fillapex disintegrated and exhibited numerous cracks (Fig. 2) making it difficult to measure the porosity. AH Plus and Endofill exhibited similar open and total porosity values for all their time frames ($p>0.05$). Endofill exhibited the highest closed porosity values ($p<0.05$). MTA Fillapex and Endofill maintained their porosity values during the time ($p>0.05$). AH Plus showed its greatest values of open and total porosity at 7 days and its closed porosity decreased after 30 days ($p<0.05$). The 3D models created in the CTAn program with the different materials at initial time are presented in Figure 3.

Discussion

The current study evaluated three root canal sealers with different chemical compositions regarding their physical properties. Smaller specimens were used to assess solubility and dimensional stability (14), without changing the accuracy of the method. According to ISO and ADA specifications, the solubility should be less than 3%, and dimensional change must be a maximum contraction of 1.0% or 0.1% of expansion. Solubility is evaluated using conventional tests after 24 h. However, longer periods of analysis can be used to provide information about the behaviour of the materials (19). Furthermore, the conventional tests for evaluating the solubility and dimensional stability of the sealers have some limitations. Regarding the solubility, the sealers may exhibit degradation during storage or absorb water (17). The main limitation of dimensional stability test is that this method is based on a linear measurement (13). Thus, the use of micro-CT in the present study provided three-dimensional volumetric analysis (in mm^3), allowing correlation of volumetric change with the properties of solubility and dimensional change (16,17). This non-destructive methodology allows standardized and reproducible analysis, complementing the conventional tests (17). Since different materials were evaluated and their radiopacity could interfere in the analyses of the images obtained by micro-CT, we performed a careful selection of the reconstruction parameters for each sealer.

AH Plus and Endofill presented proper solubility after 7 and 30 days, while MTA Fillapex exhibited high values in both time intervals, in agreement with previous studies (12,13). Regarding the dimensional stability, only Endofill was in accordance with the standards. MTA Fillapex exhibited the highest dimensional change (shrinkage of

Table 4. Means and standard deviation of open, closed and total porosity results (%) observed for the materials at time intervals: initial, after 7 and 30 days

	AHP	MTAF	EDF
Initial open porosity	2.47 (0.74)^{a,A}	44.29 (9.30)^{b,A}	4.33 (0.85)^{a,A}
Open porosity 7 days	4.09 (1.04)^{a,B}	47.80(7.75)^{b,A}	4.01 (0.87)^{a,A}
Open porosity 30 days	3.42 (1.23)^{a,A}	-	4.16 (1.09)^{a,A}
Initial closed porosity	0.05 (0.02)^{a,A}	0.12 (0.07)^{a,A}	0.47 (0.09)^{b,A}
Closed porosity 7 days	0.10 (0.04)^{a,A}	0.10 (0.04)^{a,A}	0.47 (0.08)^{b,A}
Closed porosity 30 days	0.02 (0.01)^{a,B}	-	0.47 (0.09)^{b,A}
Initial Total porosity	2.70 (1.09)^{a,A}	44.31 (9.64)^{b,A}	4.96 (0.75)^{a,A}
Total porosity 7 days	4.30 (1.05)^{a,B}	49.94 (7.99)^{b,A}	4.37 (1.01)^{a,A}
Total porosity 30 days	3.10 (0.79)^{a,A}	-	4.51 (0.96)^{a,A}

^{a,b,c,d}Different letters in same line indicate statistical difference among experimental groups in the same period ($p<0.05$). ^{A,B}Capital letters in same column indicate statistically significant difference among the same group in different periods ($p<0.05$).

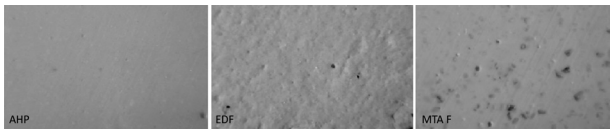

Figure 1. Micrographs showing porosity of sealers AH Plus, Endofill and MTA Fillapex at 50× magnification observed under inverted digital microscope.

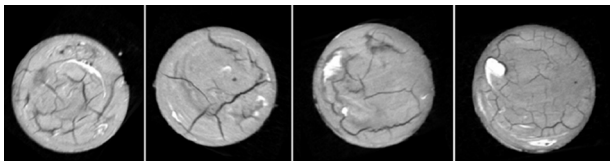

Figure 2. Images captured from CTAn software illustrating microtomographic images of different MTA Fillapex specimens after 30 days of immersion in distilled water.

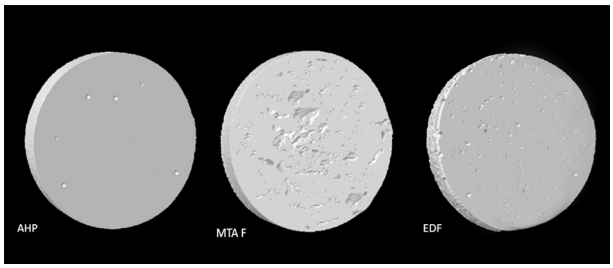

Figure 3. 3D models illustrating microtomographic images of the porosity of AH Plus, MTA Fillapex and Endofill, respectively.

1.69%). In previous research, AH Plus (13,15) and MTA Fillapex (13) were shown not to comply with the ISO/ADA recommendations, while Endofill was in accordance (15), according to the results of present study. MTA Fillapex and Endofill presented a volumetric reduction after immersion, probably due to their solubility and dimensional contraction. AH Plus presented a gain of mass and length, besides a volume increase at 7 days, also showing a direct relation between the tests.

The salicylate resin in MTA Fillapex composition lead to a high dissolution in addition to increasing the contraction factor (13), which could justify the high solubility, contraction and the volumetric loss of this material. These properties may compromise the root canal sealing (20). Epoxy resin-based sealers have crosslinks in its resin polymers, which cause a low contraction and some expansion during setting (10,13). This factor would explain the expansion that occurred in AH Plus in the dimensional and volumetric change after 7 days, in addition to the increase in weight. For Endofill, the solubility observed after 7 and 30 days, besides the dimensional and volumetric reduction may occur due to the continuous loss of eugenol, causing a leaching effect that could lead to disintegration of the material (21).

Micro-CT was used to evaluate the solubility and dimensional change of AH Plus and MTA Fillapex by using extracted human teeth in a previous study (16). The authors observed no difference in the reduction of volume of the materials after root canals filling and immersion in PBS, in disagreement with our results. The authors related that the solubility values observed for this sealer could be compensated by the absorption of fluids. The different methodologies and medium of immersion may justify the differences in the results, seeing that the solubility of calcium silicate-based cements tends to be greater in distilled water than in saline solutions (22).

Solubility and porosity may be associated (23) affecting the stability, integrity and durability of the cements (24). In this study, images captured in the inverted digital microscope at 50× and 200× magnifications were transferred to the program ImageTool version 3.0, making it possible to count the pores in the sealers and perform quantitative comparison. Evaluation by means of micro-CT does not require sectioning of the sample, which may influence the measurement of the number and size of pores (25). Furthermore, micro-CT provides data about open and closed porosity separately, since closed pores represented empty spaces completely surrounded by material, and open pores are those in which there is some type of contact with the outside surface.

The total porosity under microscopy and micro-CT showed high values for MTA Fillapex. After 30 days, porosity evaluation was not possible due to disintegration of the material and crack formation (Fig. 2), which probably occurred due to the high solubility of this sealer. A previous study (10) evaluated the solubility and porosity of MTA Fillapex. The authors observed a compact and homogeneous surface before the solubility test for MTA Fillapex, and the presence of cracks and porosities after the test. On the other hand, a more homogeneous surface with lower apparent porosity for AH Plus is observed in Figures 1 and 3. Barros et al. (7) observed low solubility and apparent porosity for AH Plus. Theses finding reinforce the correlation between the properties of solubility and porosity.

In conclusion, MTA Fillapex presented the highest solubility, dimensional and volumetric change, besides porosity, which could limit its clinical use. The assessed physical properties of the sealers are related, and the different tests provided complementary data. Micro-CT is an important non-destructive tool for analyzing the physiochemical properties of endodontic materials.

Resumo

O objetivo deste estudo foi avaliar propriedades físicas de cimentos endodônticos (AH Plus, MTA Fillapex e Endofill), por meio de testes convencionais e micro-CT. A estabilidade dimensional foi avaliada após imersão dos materiais em água destilada por 30 dias. A solubilidade e a alteração volumétrica dos materiais foram avaliadas após 7 e 30 dias de imersão em água destilada. A solubilidade foi avaliada por meio de perda de massa e a alteração volumétrica foi avaliada por micro-CT. A porosidade foi avaliada por microscopia após 7 dias de imersão em água destilada e por micro-CT após a presa e imersão em água destilada por 7 e 30 dias. A análise estatística foi realizada por meio dos testes ANOVA e Tukey, com nível de significância de 5%. MTA Fillapex apresentou a maior solubilidade ($p<0,05$), com valores acima das recomendações ISO/ADA. As alterações volumétricas e dimensionais foram maiores para MTA Fillapex, seguido por Endofill e AH Plus ($p<0,05$). MTA Fillapex e AH Plus não cumpriram os padrões ISO/ADA em relação à estabilidade dimensional. Uma maior porosidade total foi observada para MTA Fillapex ($p <0,05$). Endofill apresentou maior porosidade total que o AH Plus pela avaliação em microscopia ($p<0,05$), e ambos os cimentos foram semelhantes na avaliação por micro-CT ($p>0,05$). Em conclusão, MTA Fillapex apresentou maior solubilidade, alteração dimensional e volumétrica, além de maior porosidade em relação aos demais cimentos avaliados. As propriedades físicas avaliadas estão relacionadas, e os diferentes testes forneceram dados complementares. Micro-CT é um método valioso para avaliação das propriedades físicas dos materiais endodônticos.

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