Universidade Estadual Paulista “Júlio de Mesquita Filho”
Faculdade de Odontologia de Araraquara

KATIA CRISTINA KEINE

IMPACTO DA INCORPORAÇÃO DE SUBSTÂNCIAS QUÍMICAS NAS PROPRIEDADES FÍSICO-QUÍMICAS DOS CIMENTOS ENDODÔNTICOS

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Dissertação apresentada ao Programa de Pós-graduação em Odontologia, Área de Endodontia, da Faculdade de Odontologia de Araraquara da Universidade Estadual Paulista "Júlio de Mesquita Filho" como requisito para obtenção do título de Mestre em Odontologia.
ORIENTADORA: Profa. Dra. Gisele Faria

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COMISSÃO JULGADORA

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DADOS CURRICULARES

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“O conhecimento serve para encantar pessoas, não para humilhá-las”

Mário Sérgio Cortela

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RESUMO

Avaliar os efeitos da incorporação do iodofórmio ou do hidróxido de cálcio (HC) respectivamente no cimento Sealer 26 ou MTA Fillapex (MF). Na avaliação com o Sealer 26, as proporções de 1.1g, 0,55g or 0.275g foram incorporadas ao cimento e o de tempo de presa, escoamento, solubilidade, pH e liberação de cálcio foram avaliados. Na análise do MF puro, acrescido de 5% (MF5) ou 10%(MF10) em massa de HC, o escoamento, pH e liberação de cálcio foram comparados com AH Plus. A adição do iodofórmio ao Sealer26 aumentou o tempo de presa do cimento ($p<0,05$), mas os demais resultados foram similares ($p>0,05$). Em 24hs, a mistura com 1.1g: 0.55g de iodofórmio demonstrou menor pH que o cimento puro ($p<0,05$). O MF teve maior escoamento que os demais cimentos ($p<0,05$). O MF10 apresentou menor escoamento que MF ($P<0,05$). O MF10 apresentou maior pH que o AH Plus ($p<0,05$).A liberação de cálcio foi similar ao MF e o MF10 ($p>0,05$). O MF5 demonstrou menor escoamento que o MF e maior que MF10 ($p<0,05$), tendo maior valor de pH que o MF ($p<0,05$) e similar liberação de cácio ao MF10. A adição do iodofórmio interfere no tempo de presa e solubilidade do Sealer 26 e a adição de 5%HC no MF apresentou-se como alternativa para reduzir o escoamento, sem interferir no potencial de alcalinização do MF.

Palavras Chaves: cálcio, endodontia, escoamento, propriedades físicas

Keine KC. Impact of incorporation of chemicals on the physico-chemical properties of root canal sealers [Dissertação de Mestrado]. Araraquara: Faculdade de Odontologia da UNESP; 2014.

ABSTRACT

To evaluate the effects of incorporation of iodoform or calcium hydroxide (CH) respectively in cement Sealer 26 or MTA Fillapex (MF) . In review with the Sealer26, the proportions of 1.1g, 0.55g or 0.275g were incorporated into the cement and the setting time, flow, solubility, pH and calcium release were evaluated. The analysis of pure MTA Fillapex (MF) or containing 5% (MF5) or 10% (MF 10) calcium hydroxide (CH), in weight, in comparison with AH Plus sealer .The addition of iodoform to Sealer26 cement increased setting time($p < 0.05$), but the other results were similar ($p > 0.05$). In 24 hours, the mixture with 1.1g : 0.55g iodoform showed lower pH than the pure cement ($p < 0.05$) . The MF had greater flow than the other cements ($p < 0.05$) . The MF10 showed lower flow that MF ($P < 0.05$) . The MF10 showed pH greater than AH Plus ($p < 0.05$). Calcium realise was similar to MF and MF10 ($p > 0.05$). The MF5 showed less than the flow MF , and greater than MF10 ($p < 0.05$), and higher pH value than the MF ($p < 0.05$) and calcium release similar to MF10 . The addition of iodoform interferes with the setting time and solubility of Sealer 26 and the addition of 5% CH in the MF presented as an alternative to reduce the flow without interfering with potential alkalization.

Key Words: calcium, endodontics, flow, physical properties

SUMÁRIO

1 INTRODUÇÃO

Os cimentos endodônticos possuem a finalidade de complementar a obturação dos canais radiculares, que juntamente com outros materiais, tais como cones de guta percha ou poliéster, irão preencher o espaço anteriormente ocupado pelo tecido pulpar, evitando a infiltração microbiana para o interior da cavidade pulpar (Holland et al.[13] ,1990).

Para que exerça seus objetivos, é importante que o cimento endodôntico apresente propriedades físicas, químicas e biológicas adequadas, a fim de favorecer ou não interferir negativamente sobre o processo de reparo apical (Caicedo, Von Fraunhofer [3],1988). Entretanto, as propriedades desejáveis para estes materiais não são observadas em um único cimento endodôntico.

Na Endodontia, os cimentos para uso na obturação dos canais radiculares são principalmente derivados de: a. óxido de zinco e eugenol, b. resinas, tais como epóxi, metacrilato ou salicilato, c. silicatos (MTA). Inúmeras outras formulações já foram propostas, porém em função de seus efeitos adversos praticamente não são recomendados para uso clinico rotineiro (Mutoh, Tani-Ishii [18], 2011; Morgental et al.[17], 2011; Bodanezi et al.[2], 2010).

O AH Plus (Dentsply DeTrey GmbH, Konstanz, Germany), um cimento que contém resina epóxi em sua composição é atualmente um dos mais utilizados. No Brasil, o Sealer 26 (Denstply, Petrópolis, Rio de Janeiro, Brasil) que também contém resina epóxi, é o mais utilizado na clinica odontológica (Guerreiro-Tanomaru et al.[12], 2009).

Paralelamente, devido aos excelentes resultados obtidos com o uso do MTA (mineral trioxide aggregate) em Endodontia, foi desenvolvido o MTA Fillapex (Angelus, Londrina, Paraná, Brasil), um cimento endodôntico que contém MTA na sua composição (Gomes-Filho et al.[11], 2012).

Para que um cimento endodôntico tenha um adequado comportamento clínico é fundamental que possua radiopacidade, tempo de presa, escoamento, potencial de alcalinização e liberação de cálcio adequados, dentro das normas técnicas estabelecidas pelos órgãos de regulamentação (Duarte et al.[6,7] ,2009, 2010; ASTM C266-03: 2000[1]; ISO 6876: 2001[15]).

Entretanto tem-se observado que o Sealer 26 apresenta radiopacidade inferior a de diversos cimentos endodônticos e da guta percha, o que dificulta a interpretação visual da qualidade da obturação do canal radicular. Por outro lado, também tem-se observado que o MTA Fillapex apresenta um escoamento excessivo, o que pode favorecer a extrusão apical do cimento (Figueiredo et al.[10], 1997; Keine et al.[16], 2012).

Com vistas a sanar estes problemas detectáveis clinicamente, cirurgiões-dentistas incorporam empiricamente substâncias químicas aos cimentos endodônticos, tais como o iodofórmio no Sealer 26 e o hidróxido de cálcio no MTA Fillapex. No entanto, não há estudos avaliando o efeito da incorporação destas substâncias nas propriedades dos cimentos endodônticos.

Sendo assim, é importante a investigação inicial dos efeitos ocasionados pela incorporação destas substâncias químicas nos cimentos endodônticos, através de análises físico-químicas, a fim de esclarecer os riscos e/ou benefícios deste procedimento.

2 PROPOSIÇÃO

Capítulo 1: Avaliar a influência da adição de iodofórmio no tempo de presa, escoamento, solubilidade, pH e liberação de cálcio do Sealer 26.

Capítulo 2: Avaliar o escoamento, pH e liberação de cálcio do MTA Fillapex puro ou contendo 5% ou 10% de hidróxido de cálcio, em massa, em comparação ao AH Plus.

3 CAPÍTULOS

3.1 CAPÍTULO 1

The impact of the addition of iodoform on physico-chemical properties of an epoxy-based endodontic sealer*

**Artigo aprovado para publicação no Journal of Applied Oral Science*

ABSTRACT

Introduction - because of the low radiopacity of Sealer 26, iodoform has been empirically added to this sealer. Thus, the interference of this procedure in the physico-chemical properties of Sealer 26 must be evaluated.
Objective – this study evaluated the influence of the addition of iodoform on setting time, flow, solubility, pH, and calcium release of an epoxy-based sealer.
Materials and Methods – the control group was pure Sealer 26, and the experimental groups were Sealer 26 added of 1.1g, 0,55g or 0.275g of iodoform. Setting time evaluation was done in accordance with ASTM C266-03 specification. The analysis of flow and solubility was in accordance with ISO 6876-2001 specification. For the evaluation of pH and calcium ion release, polyethylene tubes were filled with the materials and immersed in flasks with 10ml of deionized water. After 24h, 7, 14, 21, 28, and 45 days pH was measured. In 45 days, the calcium released was evaluated with an atomic absorption spectrophotometer.
Results – the addition of iodoform increased the setting time in comparison to pure sealer ($P<0.05$). For flow, solubility, and calcium release, the mixtures presented results similar to pure sealer ($p > 0.05$). In the 24h period the mixture with 1.1g and 0.55g of iodoform showed lower pH than pure sealer or then sealer added with 0.275g of iodoform ($P<0.05$).
Conclusions – the iodoform added in Sealer 26 interferes in its setting time and solubility properties. However its clinical significance needs further studies.

Keywords: Endodontics; Root canal filling materials; Physical and chemical properties

INTRODUCTION

Endodontic sealers should have an antimicrobial activity[28] and the capacity to stimulate the healing of periapical tissue[19]. These properties are related to the release of hydroxyl and calcium ions[13] and the increasing of pH[15].

Besides these, endodontic sealers should have adequate flow, setting time and low solubility[7].

In Brazil, the epoxy-based sealer, Sealer 26, is one of the most used in root canal filling and widelly commercially available[6]. However, its radiopacity is lower than other endodontic sealers, such as methacrylate-based (Epiphany)[16] or even another epoxy-based (AH Plus)[26], and gutta-percha point[5]. This difference of radiopacity may be inadvertently interpreted as failure in root canal filling.

A way to improve the radiopacity of the Sealer 26 is to add iodoform, in a minimum ratio of 1:3, to the total volume of the sealer[14]. Materials containing iodoform have been sugested for root canal filling in primary teeth[8]. When added to Sealapex, the histological results do not change in relation to pure Sealapex[17], and the mixture presented clinical applicability as an alternative radiopacifier to endodontic procedures.

Iodoform (CHI_3) is commonly used as radiopacifier to primary tooth root canal filling[8,24,22]. The mixtures with iodoform have low antibacterial activity[3]. But its use in endodontic is controversial, because of their potential for causing toxic effects[24,4]. Iodoform has similar radiopacifying action to bismuth subnitrate and higher than zinc oxide, barium sulfate, zirconium oxide, and calcium tungstate[11].

Because of the radiopacity of Sealer 26, endodontists have empirically added the iodoform to this sealer. But there are no studies evaluating the interference of this procedure in physico-chemical properties of the Sealer 26. Since some essential properties, such as solubility and setting time are under the control of the powder/resin ratio of sealer, preparing the material with varying consistency may result in variations in the properties of sealer.[7] This may have a clinical consequence, such as difficulty in handling and/or interference in the antimicrobial action[7,20].

Flow is important as it determines the ability of the sealer to penetrate into small irregularities, ramifications and lateral canals[1]. When the flow of the sealer is not in accordance with the ISO (6876) or ADA (57) norms its use in root canal obturation is contraindicate. The maintenance of alkalization potential and calcium ion release properties of the sealer after its manipulation are important to their biological and microbiological actions[10].

Therefore, studies that assess the interference of the addition of several proportions of iodoform in properties of the Sealer 26 are necessary. This study aims to evaluate the hypothesis that the addition of iodoform interfere negatively in its physico-chemical properties.

The aim of this study was to evaluate the initial and final setting time, flow, solubility, pH and calcium release from an epoxy-based sealer (Sealer 26), both pure and added with 1.1g; 0.55g or 0.275g of iodoform.

MATERIALS AND METHODS

The epoxy-based sealer used for this study was the Sealer 26 (Dentsply, Maillefer Instruments AS, Ballaigues, Suíça). The main constituents of this material, according manufacturer, are: a. powder (bismuth trioxide, calcium hydroxide, tetramine hexametilene, titanium dioxide); b. resin (Bisphenol-epoxy resin).

To manipulate the Sealer 26, 1.1g of powder was added to 1.0g of resin and mixed during 30s. This mixture was considered as the control group (G1). For the experimental groups, the mixture was prepared adding iodoform (Biodinâmica Ind. Com., Ibiporã, PR, Brazil) in proportions of 1.1g (G2), 0,55g (G3), and 0.275g (G4).

Determination of setting time

The initial and final setting times of the mixtures were determined according to the International Standards Organization (ISO) 6876 specification[18] and the ASTM C266-03 (Philadelphia: ASTM; 2008) specification[2]. All specimens were measured under controlled temperature and humidity: 37 $\pm$ 1°C and 95 $\pm$ 5% relative humidity. The sealers were mixed and inserted into metallic ring molds with 10mm of diameter and 2mm of thickness (n=3, each group).

At 180 seconds, each specimen was indented with a Gilmore needle (113.5g) until the initial setting time was determined. Then, a Gilmore needle weighing 456.5g was used to determine the final setting time. The setting times were determined as being the time elapsed from the beginning of the mixing to the time at which no indentation was detected on the surface of the mixtures. The initial and final setting times were determined when the Gilmore needle, with 113.5g and 456.5g respectively, failed to leave a definite mark in the surface of the sample. Data obtained was subjected to one-way ANOVA and Tukey tests, at a significance level of 5%.

Flow

All procedures were realized according to ISO 6876:2001[7,1,18]. The root canal sealers were mixed according to the conditions described before, and 0.05±0.005ml of mixture was placed on the centre of a glass plate with a graduated syringe. After 3 min, a second plate weighing 20g and a weight of 100g were placed on the sealer. After seven additional minutes, the average of the major and minor diameter of the disc of sealer was recorded in mm using a digital caliper with a resolution of 0.01mm (Mitutoyo MTI Corporation, Tokyo, Japan). The mean of three measurements for each sealer was taken as the flow of the material and the data submitted to Kruskal Wallis test at a significance level of 5%.

Solubility

Teflon ring molds (n=10, each group) measuring 1.5mm thickness and 20mm internal diameter were filled with the mixtures. Two glass plates, covered with incolor cellophane, were placed under and on the mold. The samples were stored for 45 days in 37 ± 1°C and 95 ± 5% relative humidity to set. After the removal of residues or loose particles, the specimens were placed in a dessicator with sílica by 24 h. In sequence, they were weighed on a precision scale (Shimadzu AW-220; Shimadzu, São Paulo, SP, Brazil) and immersed individually in 50ml of deionized water in closed flasks. Special care was taken to keep the specimens hung in the water. All specimens were again stored for 7 days in 37 ± 1°C. After this period, the specimens were again placed in a dessicator for 24h and subsequently weighed, in similar conditions as described above. The solubility and disintegration between the original mass of the specimen and its final mass were calculated to deduce the percentage of original mass loss of the specimen. All procedures were in accordance to Vivan et al.[27] The data obtained was submitted to Kruskal Wallis test at a significance level of 5%.

pH and calcium release

Forty polyethylene tubes measuring 0.5cm in length and 1.0mm in internal diameter (n=10, each group) were filled with the mixtures using a Lentulo spiral (Dentsply, Maillefer Instruments AS, Ballaigues, Suíça).

The tubes filled with fresh mixtures were weighed in order to check the standardization of the amount of sealer. They were placed in polypropylene flasks (Injeplast, São Paulo, SP, Brazil) containing 10mL of deionized water and kept at 37^{O}C. Prior to immersion of the specimens, both pH and calcium concentration of deionized water were verified, with pH being 6.8 and calcium being totally absent. Evaluations of pH and calcium ion release were carried out after 24h, 7, 14, 21, 28, and 45 days. The specimens were kept in the same distilled water during all experiment. Measurement of pH was performed with a pHmeter (model Q400I; Quimis, Diadema, SP, Brazil), previously calibrated with solutions of known pH (4, 7, and 14), at constant temperature (25^{o}C). The flasks were placed in a shaker (Farmen, São Paulo, SP, Brazil) for 5 seconds before pH measurement. The control procedure included measuring the pH of the water in which no specimens had been immersed.

In 45 days, a Spectra 55B atomic spectrophotometer (Varian, Inc.,Palo Alto, CA, USA) was used to determine the calcium release concentration (in ppm) in the deionized water. Lanthanium oxide was added to all samples to eliminate ionic interference. Solutions containing calcium concentrations of 0, 1, 2, 3, 4 and 5 ppm were used to create a standard calibration curve[23]. Data obtained was subjected to one-way ANOVA and Tukey tests, at a significance level of 5%.

RESULTS

The initial and final setting times between the groups were statistically different ($P<0.05$). The mean and standard deviation of the initial and final setting times in minutes for Sealer 26 and experimental mixtures are showed in table 1.

Regarding the flow test, the experimental mixtures and the pure Sealer 26 presented similar results ($P>0.05$) (table 2). The flow of all mixtures is in accordance to ISO 6876:2001[18].

The experimental groups presented higher solubility than Sealer 26 (P<0.05), but no differences were observed between them (P>0.05). All the sealers released calcium and were similar (P>0.05) (table 3).

The mean and standard deviation for pH values, in several periods, of the Sealer 26 and the three experimental mixtures are presented in figure 1. In the 24h period, the addition of 1.1g (G2) or 0.55g (G3) of iodoform promoted reduction of pH values. Also, these values were lower than presented by G1 and G4 (P<0.05). In other periods, pH values between the groups were similar (P>0.05). The pH values reduced in the course of time

DISCUSSION

The results of the present study, particularly the setting time and solubility tests allow us to accept the hypothesis that the addition of iodoform, independently of the proportion added interferes in the properties of Sealer 26. Therefore this procedure should be avoided.

The addition of iodoform in proportion of 1.1g, 0.55g, and 0.275g to powder of the sealer provides total mass increase of 52.38%, 26.19%, and 13.09%, respectively. Despite this, the flow was similar in all groups. When 10% calcium hydroxide was added in another epoxy-based sealer (AH Plus) the flow reduced significantly in comparison to original consistency[12]. However, AH Plus is a paste/paste system and Sealer 26 is powder/paste system and the amount, in volume, of the iodoform aggregated was insufficient to promote alterations in the sealer flow.

Several studies related the importance that endodontic sealers present alkaline pH value and promote calcium ion release[15,10,21]. In 24h period, the addition of iodoform to G2 and G3 groups promoted a reduction of pH value probably because the amount of calcium hydroxide present in the powder sealer decreased in relation to the sealer original proportion (0.37g/g). In other periods there were reduction of the pH values, but specifically in each period the values were similar. The presence of some components acid hydrogen in the sealer, as bispehol-epoxy resin, may have reacted with hydroxyl ion causing with a consequent prevention of hydroxyl ion release may have also contributed to prevention of hydroxyl ion release[23].

In this study, the assessment of the calcium release by the mixtures was only dosage in the end of experiment and did not interfered in the release. The values corresponded to the release throughout the experiment. It is possible that the same chemical reactions may have been involved as partial neutralization of hydroxyl ions also occurred in the calcium chelation[23]. Other study with Sealer 26 were made in different conditions which makes the comparison with our results impossible.[10,25]

The addition of iodoform into sealer powder, in all proportions used, promoted increase of initial and final setting time. The setting of the sealer occurs because the reaction between the bisphenol-epoxy resin and tetramine hexamethylene[9]. Addition of iodoform decreased the amount of hexamethylene tetramine significantly affecting the setting time of the sealer. Probably as a consequence of the interference in setting time and of powder/resin ratio modification, the solubility of experimental mixtures increased when compared to original Sealer 26 sealer. Similarly when the calcium hydroxide was added into AH Plus (epoxy-based sealer) there was increased in solubility of the sealer[12]. The powder/liquid ratio of endodontic sealer influences the physic-chemical properties of endodontic sealers.[7]

Thus, the addition of iodoform in Sealer 26 interferes with the physico-chemical properties of the material. Other studies should be conducted to select a radiopacifier that do not interfere with the epoxy-based sealer properties.

CONCLUSIONS

Under the conditions of this study, it seems that addition of iodoform in the powder of Sealer 26 has an influence on its physico-chemical properties. Two properties are significantly altered: solubility and setting time. Only in 24h period, the addition of iodoform, in proportion of 1.1g and 0.55g, decreased the pH value of sealer. The flow and calcium release were not affected. Therefore, these alterations to Sealer 26 properties are likely to be clinically relevant and these mixtures are not recommended for clinical use.

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Table 1 – Mean and standard deviation of the initial and final setting times in minutes for Sealer 26 and experimental mixtures.

	G1 (1.1:0.0:1.0)	G2 (1.1:1.1:1.0)	G3 (1.1:0.55:1.0)	G4 (1.1:0.275:1.0)
Initial	470 (8.66)[a]	11,486 (0.1)[b]	9,136 (199)[c]	5,595 (0.1)[d]
Final	2,569 (2.88)[a]	15,536 (0.1)[b]	11,952 (509)[c]	7,675 (103)[d]

[a,b,c,d] Different letters in each line indicate significant difference ($P<0.05$). The proportion is in sequence powder:iodoform:resin (in weight).

Table 2 - Mean and standard deviation of flow and calcium release for Sealer 26 and experimental mixtures.

	G1 (1.1:0.0:1.0)	G2 (1.1:1.1:1.0)	G3 (1.1:0.55:1.0)	G4 (1.1:0.275:1.0)
Flow (mm)	22.99 (±0.83)	21.91 (±1.87)	22.00 (±0.58)	22.01 (±1.01)
Calcium release (ppm)	1.37 (±0.20)	1.12 (±0.27)	1.23 (±0.13)	1,180 (0.20)

* No difference statistical was observed between the groups ($P>0.05$). The proportion is in sequence powder:iodoform:resin (in weight).

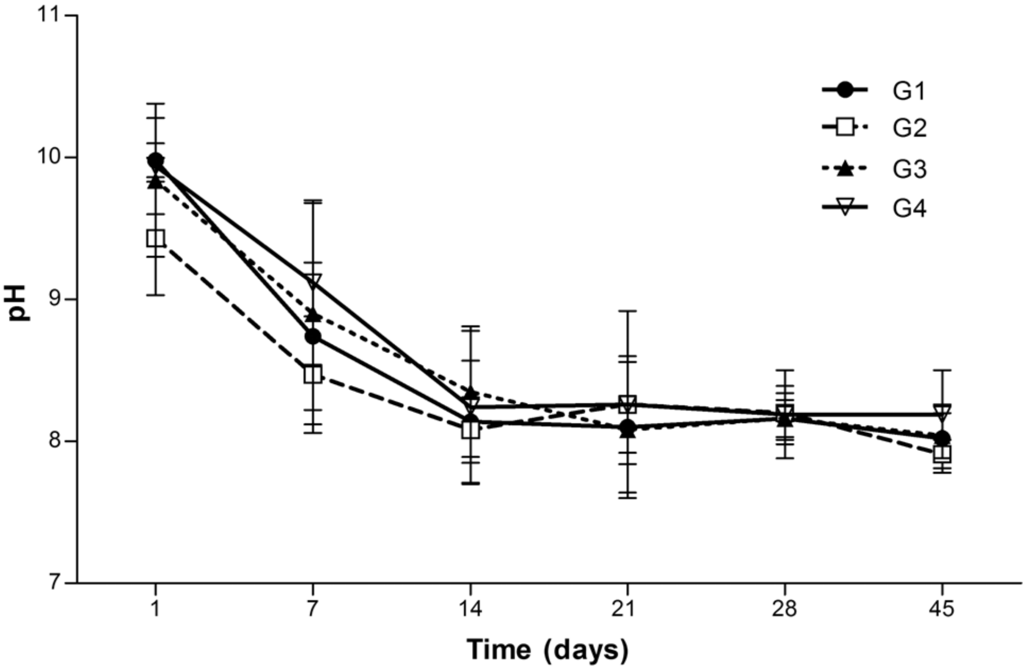
Table 3 - Mean of percentage of original mass loss for Sealer 26 and experimental mixtures.

	G1 (1.1:0.0:1.0)	G2 (1.1:1.1:1.0)	G3 (1.1:0.55:1.0)	G4 (1.1:0.275:1.0)
Mass loss	2.0[a]	10.98[b]	10.89[b]	9.38[b]

[a,b] Different letters in each line indicate significant difference ($P<0.05$). The proportion is in sequence powder:iodoform:resin (in weight).

FIGURE LEGEND

Figure 1 - pH changes of the Sealer 26 and mixtures according to different periods of times.



CAPÍTULO 2

Effects of calcium hydroxide addition on the physical and chemical properties of a calcium silicate-based sealer*

**Artigo aprovado para publicação no Journal of Applied Oral Science*

ABSTRACT

Recently various calcium silicate-based sealers have been introduced for use in root canal fillings. MTA Fillapex is one of these sealers, but some of its physico-chemical properties are not in accordance with the ISO requirements. *Objective*: The aim of this study was to evaluate the flowability, pH level and calcium release of pure MTA Fillapex (MTAF) or containing 5% (MTAF5) or 10% (MTAF 10) calcium hydroxide (CH), in weight, in comparison with AH Plus sealer. *Material and Methods*: The flowability test was performed according to the ISO 6876:2001 requirements. For the pH level and calcium ion release analysis, the sealers were placed individually (n = 10) in plastic tubes and immersed in deionized water. After 24 hours, 7 and 14 days, the water in which each specimen had been immersed was evaluated to determine the pH level changes and calcium released. The flowability, pH level and calcium release data were analyzed statistically by the ANOVA test (α=5%). *Results*: In relation to the flowability: MTAF > AH Plus > MTAF 5 > MTAF 10. In relation to the pH level, for 24hrs: MTAF 5 = MTAF 10 = MTAF > AH Plus; in 7 and 14 days: MTAF 5 =MTAF 10 > MTAF > AH Plus. For the calcium release, in all periods: MTAF > MTAF5 = MTAF 10 > AH Plus. *Conclusions*: The addition of 5% CH in the MTA Fillapex (in weight), is an alternative to reduce the high flowability presented by the sealer, without interfering in its alkalization potential.

INTRODUCTION

Mineral trioxide aggregate (MTA) was developed to be used in endodontic complications, as well as in apical surgery[20,25]. Actually, it is also recommended to be used in pulp conservative treatments and in obturation of the apical portion of immature teeth[11,22].

MTA induces a new apical cementum deposition when used in root canals[15]. However, its handling is difficult for use in root canal obturations.[5] Several calcium silicate-based sealers were developed to be used as a root canal filling material, such as Endo-CPM-Sealer (EGEO, Buenos Aires, Argentina), ProRoot Endo Sealer (Dentsply Maillefer, Ballaigues, Switzerland) and an experimental cement, MTAS[3,13]. Recently, a new formulation of MTA-based cement (MTA Fillapex; Angelus Soluções Odontológicas, Londrina, PR, Brazil) was created to be used as a root canal sealer. The composition of MTA Fillapex after mixing, is MTA, salicilate resin, natural resin, bismuth and silica, according to the manufacturer.

MTA Fillapex has an antibacterial effect against *E. faecalis* before setting and adequate biocompatibility[11,21]. Despite these biological properties, its physicochemical properties are controversial. MTA Fillapex had the lowest push-out values to root dentine compared with AH Plus and IRoot SP sealers[26]. But in another study, MTA Fillapex presented acceptable resistance to dislodgement, which was similar to AH Plus[1]. A possible reason for this different result can be attributed to the difficulty in handling, due to excessive flowability. This observation was previously confirmed in a pilot study.

An adequate flowability is a necessary property of endodontic sealers[4]. Excessive flowability can favor apical extrusion and injure the periapical tissue, mainly in teeth with a wide foraminal opening[26,28]. A suggestion in order to decrease the flowability, is the addition of calcium hydroxide into the sealer[8].

On the other hand, the ideal endodontic sealer should be biocompatible and able to induce mineralized tissue formation. These properties are directly associated with the alkalization potential and calcium release of the materials[10]. Calcium hydroxide provides an alkaline pH and calcium ion release, leading to biochemical effects that culminate in the acceleration of the repair process[27]. The MTA Fillapex sealer (Angelus) has alkaline pH and promotes calcium release[19]. However, the effects of the addition of calcium hydroxide into MTA Fillapex with the objective to reduce sealer flowability are unknown.

The aim of this study was to evaluate the flowability, pH level and calcium ion release of MTA Fillapex, added with 5% or 10% calcium hydroxide powder (in weight), compared to pure MTA Fillapex and AH Plus.

MATERIAL AND METHODS

Root canal sealers

The sealers used in this study were: AH Plus (Dentsply DeTrey, Konstanz, GER) and the MTA Fillapex (MTAF) (Angelus Ind Prod Odontológicos S/A, Londrina, PR, Brazil). The MTAF was used pure or added with 5% (MTAF 5) or 10% (MTAF 10) of calcium hydroxide (Synth, Diadema, SP, Brazil), in weight. For AH Plus, the base and catalyst pastes were used in equal parts, in proportion of 1:1 (w / w).

Flowabilty

The root canal sealers were mixed according to conditions recommended by the manufacturer and the flowability test was performed according to ISO 6876:2001[4,18].

Sample preparation

Forty polyethylene tubes measuring 10 cm in length and 1.5 mm in internal diameter were filled with the sealers to be evaluated. For the pH level and calcium ion release evaluation, 10 samples were prepared from each material studied. Immediately after manipulating the materials, the tubes were filled and weighed, to check the standardization of the amount of sealer in each tube ($\pm$ 0.002 g) and placed in polypropilene flasks (Injeplast, São Paulo, SP, BR) containing 10 mL of deionized water. The tubes were kept at 37^{O}C (Farmen, São Paulo, SP, BR) during the entire study. After 24 hrs, 7 and 14 days, the water was assessed for pH levels and calcium release. Previous to the immersion of specimens, the pH level and calcium ion concentration of the deionized water were verified, attesting a pH level of 6.8 and a total absence of calcium ions. The tubes containing the sealers were placed in new flasks with 10 mL of deionized water for further analyses in the different time periods.

pH level test and calcium release analysis

Measurements were performed with a pH meter Q400AS (Quimis, Diadema, SP, Brazil) in constant temperature (25^{o}C). After 24 hrs of immersion, the tubes were carefully removed and placed into another flask with an equal amount of new deionized water. This procedure was repeated for a total of 336 hrs, with the solution changed at 24 hrs, 7 and 14 days. The pH values were compared by the ANOVA and Tukey tests, at a 5% significance level.

The calcium release was measured using an atomic absorption spectrophotometer AA7000 (Shimadzu, Tokyo, JPN), accordance to manufacturer's instructions. For the reading, 6 mL of the standard solutions or water samples were associated with 2 mL of lanthanum nitrate solution. For the white solution, 6 mL of deionized water was associated with the same amount of lanthanum nitrate solution. With the standard solutions, the white solutions, and the prepared sample, the reading was carried out using an atomic absorption spectrophotometer[29]. The calcium release was calculated by the equation of the line of the standard curve. The reading of the calcium release was taken in the same periods used for measuring the pH level. The calcium release values were compared by the ANOVA and Tukey tests, at a 5% significance level.

RESULTS

The representative mean and standard deviation to flowability values from each sealer are showed in Figure 1 (in mm). MTAF showed higher flowability (29.4 mm) than the other sealers ($p < 0.05$). The flowability of MTAF5 was lower than MTAF and AH Plus ($p > 0.05$), but it is in accordance to the ISO 6876:2001 requirements.

The MTAF10 showed the lowest flowability than the other sealers ($p<0.05$) and this value was below the ISO 6876:2001 requirements ($p < 0.05$). The flowability means and standard deviations (in mm) to MTAF, MTAF5, MTAF10 and AH Plus were 29.40 (1.05), 20.75 (0.89), 16.80 (0.71) and 21.91 (0.36), respectively.

The representative mean and standard deviation of pH values and calcium release from each sealer, in different periods are showed in Figure 2 and 3, respectively. For all periods, AH Plus showed the lowest pH value ($p < 0.05$). At 24 hrs, MTAF, MTAF5 and MTAF10 showed similar pH values ($p > 0.05$). In 7 and 14 days, the MTAF5 and MTAF10 showed similar pH values ($p>0.05$) and a higher pH level than MTAF ($p<0.05$).

For all periods, the AH Plus showed the lowest means of calcium release ($p < 0.05$). On the other hand, the MTAF5 and MTAF10 showed similar calcium release ($p>0.05$) and lower calcium release than MTAF.

DISCUSSION

The addition of 5% calcium hydroxide to MTAF provided a reduction of the flowability, in accordance with ISO requirements. Its pH level was alkaline in all periods but the calcium release was lower than pure MTA Fillapex. Despite the addition of 10% calcium hydroxide to MTA Fillapex, it provided a reduction in flowability, this mixture is in disagreement with ISO 6876:2001 requirements.

The flowability test results revealed that the addition of calcium hydroxide provides a reduction in flowability of the MTAF. This can be explained because of an increase in powder/liquid ratio also promoted, in accordance as observed in previous sutdy[23]. Except for MTAF10, all sealers were in accordance to ISO 6876:2001 requirements18.

It has been suggested that in order to stimulate mineralization, a material should have an alkaline pH level and release calcium[10]. The methodologies used for pH level and calcium release evaluations were similar to other studies[6,19,31]. The MTAF5 and MTAF10 presented similar pH levels to MTAF, only in a 24hr period. After this time, the pH values were higher than any other sealer.

For all periods, MTAF showed a higher calcium release, despite the fact that calcium hydroxide was added in MTAF5 and MTAF10. Hosoya et al.[17] (2004) observed that the addition of 10% calcium hydroxide in some sealers causes variations in setting time. Therefore, the addition of calcium hydroxide in MTAF could also have reduced the setting time of the sealer and consequently its solubility, and consequently reducing the calcium release. In contrast with this result, the addition of calcium hydroxide did not alter the AH Plus setting time[8]. However, the compositions of these sealers are different. AH Plus sealer presented lower calcium release than the other sealers, although it contains tungstate calcium in its composition. These results are consistent with other studies also carried out with atomic absorption spectrophotometry[7,12].

Through comparative analysis of compositions described by the manufactures, it was observed that MTAF and Sealapex (SybronEndo, Orange, CA, USA) contain several similar substances in their formulations. Thus, it is possible that many reactions and effects described in Sealapex also occur with the MTAF. Eldeniz, et al.[9] (2007) reports that a possible reason for the high calcium release provided by Sealapex is related with the presence of 24% calcium hydroxide. Because the sealer has a porous matrix with a low dimensional stability and high water absorption, the calcium is easily released[2,24].

On the other hand, the addition of substances with calcium in the MTA promotes a reduction of the setting time[30]. Thus, the addition of calcium hydroxide in MTAF could have interfered in the setting time and consequently in the calcium release. This could be a possible reason for MTAF showed higher calcium release when compared to MTAF5 and MTAF10.

The MTA has established properties and indications for use in several endodontic procedures[25]. The use as an endodontic sealer requires further adjustments, because its handling is very difficult. The MTAF is a new calcium silicate-based sealer, which flowability is in accordance with ISO 6876:2001 requirements[18], but clinically is very high, which can favor the extrusion of the material to the apical tissues. The purpose to add 5% (in weight) calcium hydroxide into the total weight of the sealer is an adequate alternative. Further studies are necessary to biologically evaluate this purpose.

CONCLUSION

Based on the methods and conditions employed in the present study, it is concluded that the addition of 5% calcium hydroxide (in weight) to MTAF, provides a reduction of flowability of the sealer, in conditions required by the ISO 6876:2001 specifications and favors a higher alkaline pH.

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FIGURE LEGENDS

Figure 1 - Flowability values from each sealers evaluated (in mm)**.** Differing letters indicate statistically significant differences ($p < 0.05$).

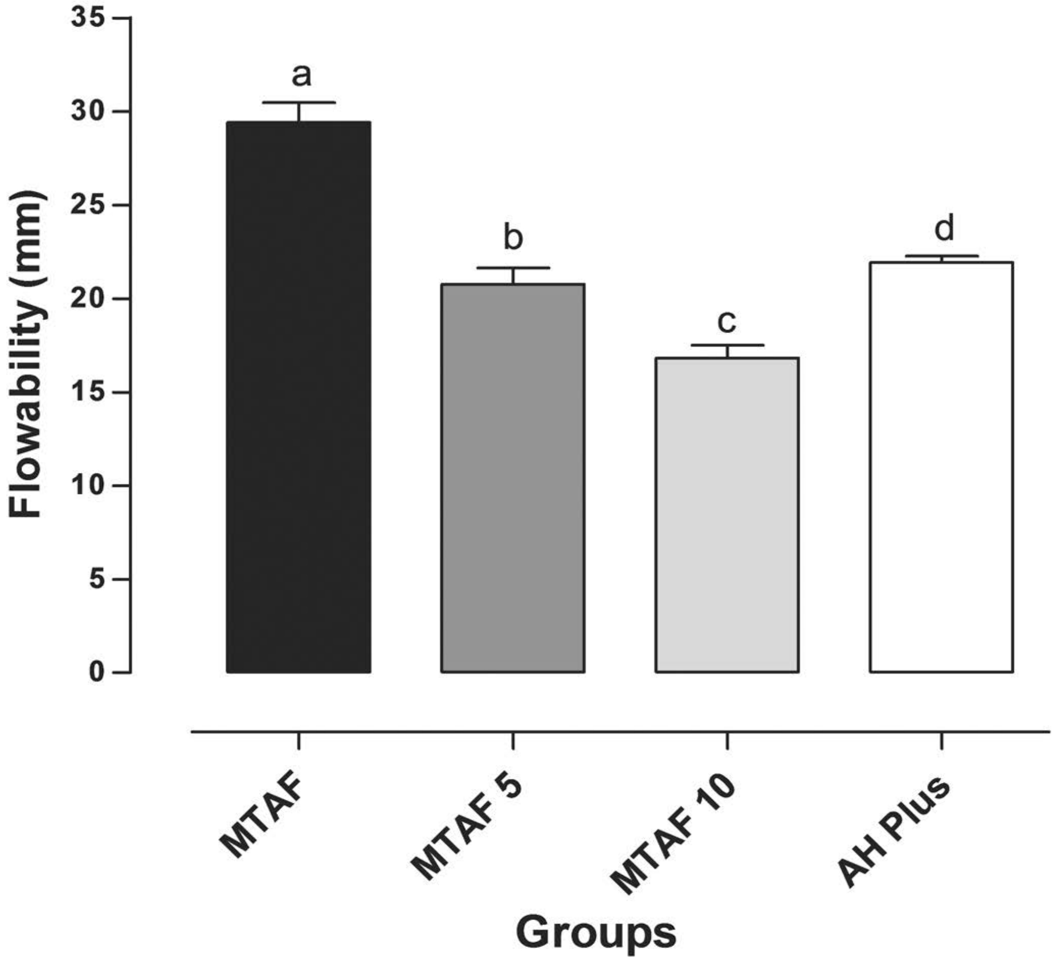


Figure 2 - pH from each sealer studied in the different experimental periods. Differing letters indicate statistically significant differences ($p < 0.05$).

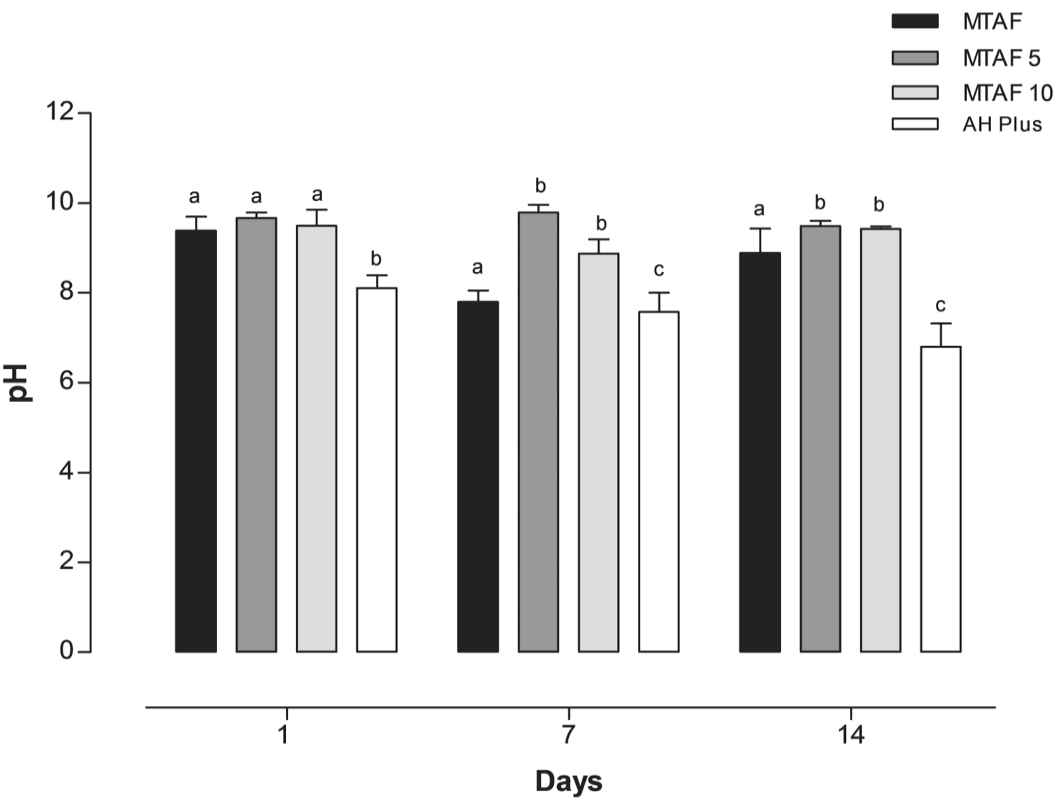
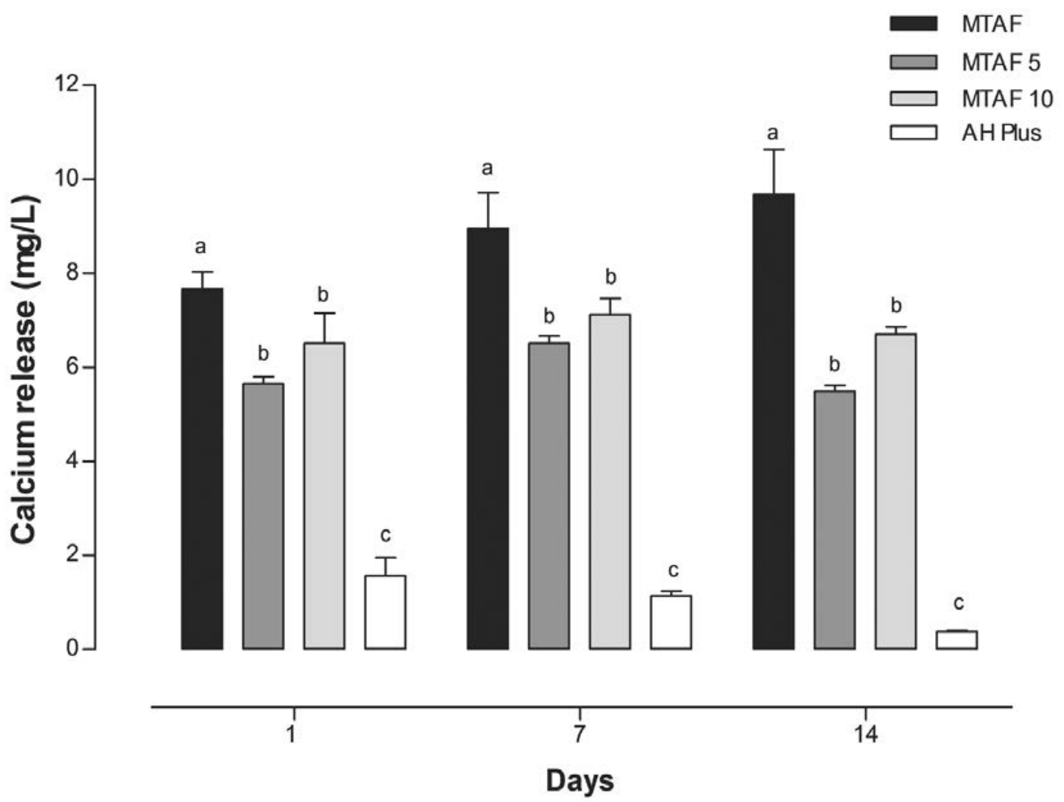


Figure 3 - Calcium release (mg/L) from each sealer studied in the different experimental periods. Differing letters indicate statistically significant differences ($p < 0.05$).



4 DISCUSSÃO GERAL

A incorporação de substâncias químicas nos cimentos endodônticos avaliados alteraram algumas de suas propriedades físico-químicas. A adição do iodofórmio interferiu no tempo de presa e solubilidade do Sealer 26 e a adição de HC, na proporção de 5% em massa, no MTA Fillapex reduziu o escoamento do cimento para uma condição clínica viável e dentro das especificações técnicas pertinentes.

Diante do exposto no capítulo 1 do presente estudo, ficou evidente que a incorporação do iodofórmio no cimento Sealer 26 interferiu negativamente em seu tempo de presa, aumentando-o significantemente em relação ao cimento puro. Como o iodofórmio foi incorporado ao pó do cimento e a quantidade de resina foi mantida constante, houve a redução proporcional do hexametilenotetramina, o que possivelmente pode ter ocasionado tal fenômeno (Duarte et al.[8] , 1997).

Como consequência da alteração do tempo de presa do Sealer 26 e modificação na proporção pó:resina do cimento, a solubilidade das misturas experimentais foram maiores que a do cimento puro. Em situação análoga, quando o HC foi incorporado ao AH Plus, que também é um cimento que contém resina epóxi em sua composição, houve um aumento em sua solubilidade (Duarte et al.[7],2010). Por outro lado, qualquer alteração na proporção pó:liquido dos cimentos endodônticos pode ocasionar modificações em suas propriedades físico-químicas (Camps et al.[4],2004).

Como existiu uma relação direta entre a quantidade de iodofórmio incorporado com a do HC residual no pó do Sealer 26, houve uma redução nos valores de pH proporcionado pelas misturas, tanto quanto maior for a concentração do iodofórmio incorporado. Estudos prévios demonstram que a concentração de cálcio presente na mistura final repercute diretamente sobre o pH proporcionado pelo cimento endodôntico (Duarte et al.[5,7], 2000, 2010; Santos et al.[20],2005). Adicionalmente, como houve a alteração na composição do cimento, com a alteração no tempo de presa inicial e final, é possível que tenha ocorrido reações químicas que tenham interferido na liberação de íon hidroxila (Silva et al.[21] ,1997). Apesar destas observações em relação à alteração do pH, a liberação de cálcio proporcianada pelas misturas foi similar ao do cimento puro.

Em relação ao MTA Fillapex, a incorporação de 5% de hidróxido de cálcio (em massa) ao volume total do cimento proporcionou uma adequada redução do escoamento (ISO-6876: 2001; Camps et al.[4], 2004). A redução do escoamento pode ser explicado em virtude do aumento da relação entre os dois compostos do MTA Fillapex, estando de acordo com o observado em estudos prévios (Ostarvik[19],1983)

Apesar da redução do escoamento do MTA Fillapex ser um fator favorável, a incorporação de 5% de HC interferiu negativamente no pH, após 24 horas, e liberação de cálcio pelo cimento. Hosoya et al.[14](2004) observaram que a adição de 10% de HC em alguns cimentos ocasionam alteração no seu tempo de presa. É possível então deduzir que, a incorporação do HC ao MTA Fillapex tenha reduzido o seu tempo de presa e, por conseguinte também a liberação de cálcio.

Através da análise comparativa nas descrições dos componentes dos cimentos MTA Fillapex e Sealapex (SybronEndo, Orange, CA, USA) é possível observar semelhanças em suas composições. Portanto, diversas reações que ocorrem com o Sealapex podem também ocorrer com o MTA Fillapex. Eldeniz et al.[9] (2007) relataram que a presença de 24% de HC na composição do Sealapex proporciona uma alta liberação de cálcio pelo cimento.

Em contrapartida, a adição de substâncias com cálcio no MTA proporciona redução no tempo de presa do material (Wiltbank et al.[22],2007) Como este é um dos principais componentes do MTA Fillapex, é possível que a incorporação do HC tenha alterado o seu tempo de presa, interferindo na liberação de cálcio pelo cimento.

Portanto, é evidente que a incorporação de substâncias quimicas nos cimentos endodônticos puro interferem nas propriedades físico-químicas dos materiais, não sendo recomendável a utilização do iodofórmio juntamente com o Sealer 26. Porém, a adição de 5% de hidróxido de cálcio ao MTA Fillapex apresenta resultados favoráveis, melhorando seu escoamento, porém ainda são necessários estudos complementares para viabilizar a sua aplicação clinica.

5 CONCLUSÃO

Baseado nos métodos e condições deste estudo pode-se concluir que a adição do iodofórmio ao pó do Sealer 26 influencia algumas de suas propriedades físico-químicas, tais como o tempo de presa e solubilidade, as quais foram significantemente incrementadas. A incorporação de hidróxido de cálcio a 5% (em massa) no MTA Fillapex proporciona uma redução do escoamento do cimento, em condições exigidas pelas especificações ISO 6876:2001.

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