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Original article

Solubility and pH Value of 2 Different Root Canal Sealers: A Comparative Study

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ABSTRACT

Background and objectives. *The purpose of this study was to evaluate solubility and pH value of MTA-Fillapex compared to AH plus sealer.* ***Methods.*** *Forty sealer discs were prepared and incubated until complete setting of the sealers. The discs were divided into two equal main groups according to the tested material; Group I: 20 discs from MTA-Fillapex. Group II: 20 discs from AH Plus. Each group was further subdivided into 2 subgroups according to the evaluation test as follows; Subgroup A: used to evaluate the solubility of the sealer (10 disc). Subgroup B: used to evaluate pH of the sealer (10 disc). For solubility test, the discs were weighed using a precision balance to 0.0001 g. Then they were immersed in HBSS and left for 24 hours in incubator at 37°C, 95% relative humidity. The discs were removed from the containers and dried, reweighed. Solubility was calculated as percentage of mass loss. For pH test, the pH value was measured by a digital pH meter after (3, 24, 48, and 168 hours) from manipulation. Results were tabulated and statistically analyzed using a one-way ANOVA test followed by Pair-wise Newman keuls multiple comparison tests.* ***Results.*** *The result indicated that MTA-Fillapex showed mean percentage weight loss statistically significantly higher than AH Plus. MTA-Fillapex has shown an alkaline pH at all observation periods which was statistically significantly higher than AH Plus (p<.05).* ***Conclusion.*** *The current study concluded that the both sealers have solubility and pH in agreement with ISO 6876/2001 and ANSI/ADA specification no. 57.*

Keywords: Solubility, pH Value, MTA-Fillapex, AH Plus, Root Canal Sealer.

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الخلفية والأهداف. كان الغرض من هذه الدراسة هو تقييم الذوبان وقيمة الرقم الهيدروجيني لـ MTA-Fillapex مقارنة بـ AH plus sealer. **طُرق الدراسة**. تم تحضير أربعين قرص مانع للتسرب وتحضينها حتى تمام تماسك المادة المانعة للتسرب. تم تقسيم الأقراص إلى مجموعتين رئيسيتين متساويتين حسب المادة المختبرة؛ المجموعة الأولى: 20 قرصًا من MTA-Fillapex. المجموعة الثانية: 20 قرص من شركة AH Plus. تم تقسيم كل مجموعة إلى مجموعتين فرعيتين وفقًا لاختبار التقييم على النحو التالي؛ المجموعة الفرعية أ: تستخدم لتقييم ذوبان المادة العازلة (10 أقراص). المجموعة الفرعية ب: تستخدم لتقييم الرقم الهيدروجيني للمادة العازلة (10 أقراص). لاختبار الذوبان، تم وزن الأقراص باستخدام ميزان دقيق يصل إلى 0.0001 جم. ثم تم غمرهم في HBSS وتركوا لمدة 24 ساعة في الحاضنة عند درجة حرارة 37 درجة مئوية، ورطوبة نسبية 95٪. تمت إزالة الأقراص من الحاويات وتجفيفها وإعادة وزنها. تم حساب الذوبان كنسبة مئوية من فقدان الكتلة. بالنسبة لاختبار الرقم الهيدروجيني، تم قياس قيمة الرقم الهيدروجيني بواسطة مقياس الرقم الهيدروجيني الرقمي بعد (3، 24، 48، و 168 ساعة) من المعالجة. تم جدولة النتائج وتحليلها إحصائيا باستخدام اختبار ANOVA أحادي الاتجاه متبوعًا باختبارات المقارنة المتعددة لنيومان كيول. **النتائج**. أشارت النتيجة إلى أن MTA-Filapex أظهر متوسط نسبة فقدان الوزن أعلى بشكل ملحوظ إحصائيًا من AH Plus. أظهرت MTA-Fillapex درجة حموضة قلوية في جميع فترات المراقبة والتي كانت أعلى بشكل ملحوظ إحصائيًا من AH Plus (p<.05). **الخاتمة**. خلصت الدراسة الحالية إلى أن كلا المادتين العازلتين لهما قابلية ذوبان ودرجة حموضة متوافقة مع المواصفة ISO 6876/2001 ومواصفات ANSI/ADA رقم. 57.

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INTRODUCTION

In endodontic obturation, the use of a thermoplastic core filling material, such as gutta-percha (GP), in conjunction with an endodontic sealer is considered conventional. Because of its good physical and biological features, gutta-percha (GP) is frequently utilized, but its lack of adhesiveness and flow necessitates the use of endodontic sealers [1]. Endodontic sealants should flow and fill the gaps between the dentinal wall and the GP core, as well as the accessory canals, and bond to both the GP and the dentin. The root filling's sealing ability is determined by the sealer's bonding to dentin and resistance to breakdown by bodily fluids. Endodontic sealants should be biocompatible because they will be in contact with living tissues for a long time [2]. Because sealer dissolution might jeopardize the overall quality of the root canal therapy, endodontic sealers should have low solubility. The degradation of root canal sealers may release chemical substances that cause inflammatory changes in the periapical tissue [3, 4]. Furthermore, root canal sealers should have a low solubility rate to retain sealing ability and/or resist reinfection caused by gaps between root canals and filling materials [5].

In general, epoxy resin-based root canal sealers, which are regarded the gold standard sealers, have a low solubility, as defined by ISO 6876:2012 and ANSI/ADA 57:2000 criteria [6, 7]. One of these sealers is AH Plus, which has been extensively evaluated for its physical and mechanical properties. Bioceramics (BCs) are a type of endodontic material that is primarily made up of synthetic tricalcium silicate [8]. Endodontics originally used bioceramic-based materials in the 1990s as retrograde filling materials, then as root repair cements and root canal sealers (RCS) [9]. Bioceramic materials have physico-chemical and biological properties that make them ideal for endodontic treatments. Within the biological milieu, bioceramics are non-toxic, biocompatible, dimensionally, and chemically stable [10]. The hydraulic and hydration capabilities of bioceramics have sparked the most interest since their introduction [8]. MTA-Fillapex (Angelus, Londrina, Brazil). is the first generation of paste MTA-containing root canal sealer which is based on salicylate resin and other resinous components [11]. It is supplied as a 4 g dual syringe with automixing tips for intra-canal application. After mixing it consists of Salicylate resin, diluting resin, natural resin, calcium tungstate, bismuth oxide, nanoparticulate silica, pigments and MTA [12].

Microorganisms from treated and filled root canals (RC) leak into the periapical tissues, causing most endodontically treated teeth to fail [13]. The most important goal of root canal therapy (RCT) is to construct a fluid-tight seal along the length of the RC system to promote tissue healing, minimize microleakage and reinfection, and encase any microorganisms that may remain after cleaning and shaping [14]. The aim of this study was to evaluate solubility and pH value of MTA-Fillapex and compare it to AH plus sealer.

METHODS

Specimen Preparation

MTA-Fillapex (Angelus, Londrina PR, Brazil) and AH Plus (Dentsply, DeTrey, GmbH, Germany), were used as the experimental materials. Mixing of the MTA-Fillapex was performed by the self-mixing tip attached to the syringe according to the manufacturer's instructions. Equal volume units of the AH Plus paste sealer were mixed on a mixing pad using a sterile metal spatula to a homogeneous consistency according to the manufacturer's instructions. The mixed sealers were placed in stainless steel ring molds with dimensions according to each subgroup test. For solubility test, the dimensions were 20 mm internal diameter and 1.5 mm thick [15].

For pH test, the mold dimensions were 10 mm internal diameter and 2mm thick [6, 7]. The specimens were then removed after complete setting and the thickness was checked with a digital caliper. Forty discs were divided into two equal main groups according to the tested material; Group I: 20 discs from MTA-Fillapex.

Group II: 20 discs from AH Plus. Each group was further subdivided into 2 subgroups according to the evaluation test as follows; Subgroup A: used to evaluate the solubilty of the sealer (10 disc). Subgroup B: used to evaluate pH of the sealer (10 disc).

Solubility Evaluation

A standard mix of the sealer was placed in a mold supported by a glass plate and filled to a slight excess. An impermeable nylon thread was placed inside 10 discs of each sealer before setting to provide convenient mean of holding the specimens. The assembly was placed in an incubator (37 ºC, 95% relative humidity) and left for a period corresponding to three times the setting time [11, 15, 16, 17]. As soon as the samples were removed from the mold, residues and loose particles were removed. Samples were weighed 3 times each in an analytical balance (accuracy, ± 0.0001 g), and the mean reading was recorded for determination of the initial weight. The samples were suspended by the nylon thread and placed inside a vessel with a wide opening containing 50 ± 1ml of distilled water, taking care to avoid any contact between the sample and the inner surface of the container. The containers were sealed and left 24 hours in an incubator (37 ºC, 95% relative humidity). After this period, the samples were removed from the containers, rinsed with distilled water and blotted dry using absorbent paper, and placed in dehumidifier for 24 hours [11]. Afterward, they were reweighted again for determination of the final weight. Solubility of the tested sealers was expressed as the percentage of the weight change (%) of each sample compared to the original weight and calculated according to the following equation [11, 15, 16, 18, 19].

Solubility = ([final weight – initial weight] / initial weight) x 100.

pH Evaluation:

Ten discs of each sealer were used for pH evaluation test. Each disc was put in a glass beaker containing 10 ml distilled water and stored at 37 ºC throughout the study periods. The distilled water was not changed during the observation period. After predetermined periods (3, 24, 48, 72 and 168 hours) [20. 21], the pH of the solution was measured. The pH was measured with a pH meter previously calibrated with solution of known pH (4, 7, 10). Before the immersion of specimens, the pH of the distilled water was measured to ensure its neutral pH of 7. At each observation period after removal of the specimens, the test tubes were shaken for 5 seconds before pH measurement.

Statistical Analysis

All the data was collected and tabulated. One-way analysis of variance (ANOVA) was used for comparison between means followed by Pair-wise Newman keuls multiple comparison tests to evaluate the significance between groups. Statistical analysis was performed using Instat 3.3 statistics software for windows. The significance level was set at $p \leq 0.05$.

RESULTS

Solubility

The mean percentage weight loss was 1.01 ±0.179 in subgroup IA of the MTA-Fillapex. The maximum percentage weight loss was 1.3 % and the minimum percentage weight loss was 0.8 %. While in subgroup IIA of the AH Plus, the mean percentage weight loss was 0.222 ±0.093. The maximum percentage weight loss was 0.35 % and the minimum percentage weight loss was 0.05%. MTA-Fillapex has shown higher mean percentage weight loss than AH Plus with a statistically significant difference (Fig. 1).

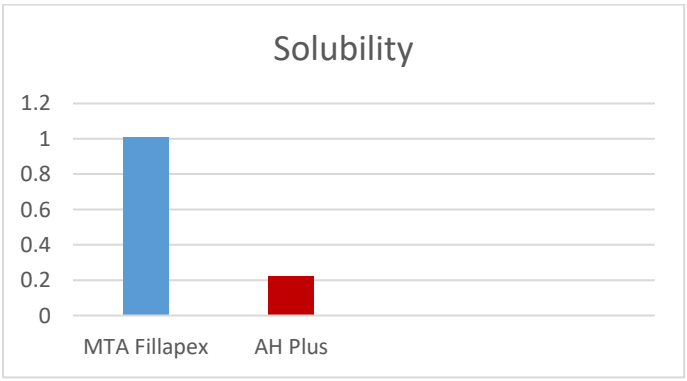

Figure 1. Bar chart representing mean solubility of both sealers as percentage weight loss.

pH value

The mean pH value in subgroup IB of the MTA-Fillapex after 3 hours was 9.52 ± 0.168, after 24 hours was 9.45 ±0.164. While at 48 hours, it was 8.44 ±0.259, after 72 hours it was 8.33 ±0.266, and was 7.63 ±0.156 after 168 hours. The mean pH value in subgroup IIB of the AH Plus after 3 hours was 7.04 ±0.126, after 24 hours was 7 ±0.115. While after 48 hours, it was 6.95 ±0.097, after 72 hours it was 6.92 ±0.103, and it was 6.93 ±0.115 after 168 hours. MTA-Fillapex showed higher mean pH value than AH Plus at all observation periods (3, 24, 48, 72 and 168 hours). This difference was shown to be statistically significant at all observation periods (Fig. 2).

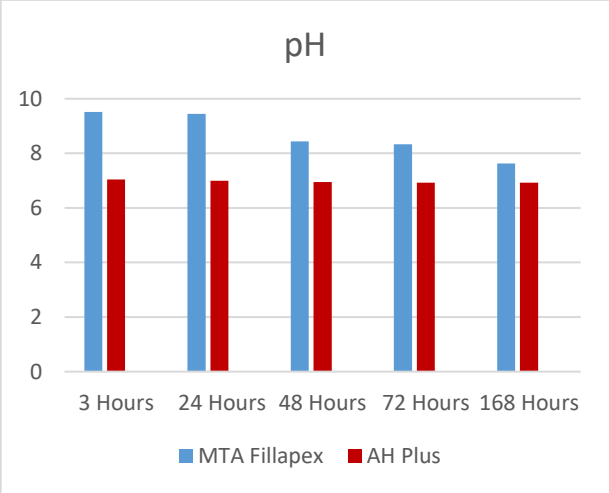

Figure 2. Bar chart representing mean pH values of MTA-Fillapex and AH Plus at all observation

DISSCUSION

Endodontic treatment effectiveness is dependent on multiple factors, including RC system shaping, cleaning, and a 3D RC obturation that can produce an excellent coronal seal, limit apical leakage, and encase any remaining bacteria [14,22]. This three-dimensional obturation could be achieved by mixing GP with a sealer, which functions as a binding agent between GP and the RC's dentin. The RC's long-standing bacteria-tight sealing relies mostly on the sealer's integrity, rather than the core material [23]. In last decade, calcium silicate cements have received greater attention in endodontics because they are able to set in the presence of biological fluids [24]. New endodontic materials have been developed based on the physicochemical properties of MTA. Dicalcium and tricalcium silicate cements (e.g., mineral trioxide aggregate [MTA]) were originally used in dentistry to treat lateral root perforations and retrograde root-end fillings [25]. Calcium silicate–based RCSs were created because they are highly biocompatible and bioactive. The clinical behaviour and handling of endodontic sealer changes are better understood thanks to preliminary laboratory studies of newly created endodontic sealers [26]. MTA-Fillapex is MTA-based sealer, which is mainly composed of MTA, resin (salicylate, diluting, natural), bismuth oxide, nano-particulated silica and pigments. Epoxy resin-based sealers were introduced in endodontics and current modifications of the original formula are widely used for root canal filling procedures. One of these sealers is AH Plus; which has been extensively evaluated for its physicochemical properties and biological response. The presented study was concluded to evaluate MTA-Fillapex and compare it to the well-known and extensively evaluated epoxy-resin based AH Plus.

The physical tests performed in this study were chosen to characterize the physical and handling properties of the tested sealers and were carried out by using methods based on ANSI/ADA no. 57 specification [7] and International Standards Organization (ISO) 6876/2001[6]. For the solubility test, a modification previously proposed by Carvalho-Junior et al [15] for the solubility test was used, which achieved similar results with a decrease in the material volume necessary for the production of the samples. The pH change of sealers may play a role in healing, because pH is associated with antimicrobial effects and deposition of mineralized tissue. It has been found that an alkaline pH of root canal sealers could neutralize the lactic acid from osteoclasts and prevent dissolution of mineralized components of teeth; therefore, root canal sealers, especially bioceramic-based sealers, can contribute to hard tissue formation by activating alkaline phosphatase [19]. Solubility is one of the most crucial physiochemical

properties of a root canal filling material. High solubility of a root canal sealer might result in loss of structure to the oral environment and create lack of integrity in the sealer, dissolution may cause the release of material that could irritate the periapical tissue and may also permit gaps to form between root canals and filling materials. These gaps might provide a pathway for microorganisms and their toxic products into periapical tissues, damaging the endodontic seal [15]. The limitation in the present in vitro study is the use of distilled water as testing medium, in agreement with the international regulations, for the measurements of the chemical-physical properties in order to standardize the test conditions and hence allow a comparison of the data with other future studies. It is important to point out that the standard test methods for solubility recommend to immerse the materials in water only after complete setting (or at least 70% of the initial setting). However, this situation is impossible to be achieved clinically because the materials are immediately placed into contact with fluids and blood. Therefore, the solubility values in a clinical scenario are probably higher than the ones found in in-vitro tests [18].

The solubility results of both sealers comply with the requirements of ISO 6876/2001 specification [6] and ANSI/ADA Specification no. 57 [7], which defines solubility as the loss of mass during a period of immersion in water following srtting and should not exceed 3% by mass. During manipulation of AH Plus, diepoxide compounds and polyamines paste are mixed together. Each amine group can react with an epoxide group to form a covalent bond. The resulting polymer is heavily cross-linked and is thus rigid and strong [11, 27], which may explain the low solubility and the homogeneous and compact rough surface of AH Plus revealed in SEM analysis in the present study. The MTA-Fillapex had higher solubility. The degree of solubility of MTA is a matter of debate amongst investigators. The divergence between authors has been attributed to the powder-to-water ratio, that increases MTA porosity and solubility, as well as the addition of bismuth oxide, which is insoluble in water, and contributes to the insolubility of MTA [28]. Results of the present study came in accordance with Borges et al [11] and Zhou et al [19] who demonstrated higher solubility for the MTA-Fillapex than AH Plus. Our results contradicted those obtained by Vitti et al [18] who showed statistically significantly higher solubility for the AH Plus. They performed the solubility test at 1, 7, 14 and 28 days following a modification previously proposed by Gandolfi et al [29]. No logical explanation could be given for such contradiction. It is important to point out that the standard test methods for solubility recommend immersion of the materials in water only after complete setting (or at least 70% of the initial setting). However, this situation is impossible to be achieved clinically because the materials are immediately placed into contact with fluid and blood. Therefore, the solubility values in a clinical scenario are probably higher than the ones found in in vitro tests [18].

Results of the present study showed that MTA-Fillapex recorded an alkaline pH in all experimental periods on immersion in distilled water. AH Plus sealer showed a slightly neutral pH. MTA-Fillapex showed a significantly higher pH value than AH Plus up to the 7-day period. The high pH value recored by the MTA-Fillapex could be explained by its high solubility [19]. This indicated that MTA-Fillapex has a strong capacity of releasing hydroxyl ions. A high pH activates alkaline phosphatase, which is present in the tissues, is involved in the mineralization process, and requires a pH around 8.6 to 10.3 to operate. The high pH of this sealer may also neutralize the acids secreted by osteoclasts, and this may help prevent further destruction of mineralized tissue. One disadvantage of its alkaline pH is a possible high cytotoxicity according to Silva et al [20]. However, its initial cytotoxicity could also be considered as an advantage. The high pH usually has a destructive effect on bacterial cell membranes and protein structure, which seems interesting, especially knowing that microorganism can remain in the ramifications of the root canal system after chemomechanical preparation

and intracanal dressing. This came in agreement with Lovato et al [30] who stated that sealers having antimicrobial activity can act against such microorganisms, reducing their numbers and providing a better chance of successful root canal treatment. These results came in full agreement with Silva et al [20] and Zhou et al [19] who stated that MTA-Fillapex presented an alkaline pH in all experimental times which was statistically significantly higher than AH Plus. No disagreement was found between our results and previous studies [19, 20, 31] showing slightly neutral pH of AH Plus.

CONCLUSION

Under the condition of the present study, it can be concluded that both sealers have solubility and pH in agreement with ISO 6876/2001 and ANSI/ADA specification no. 57. Furthermore AH Plus are still superior to MTA-Fillaper regarding solubility.

Conflict of Interest

There are no financial, personal, or professional conflicts of interest to declare.

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