THE INFLUENCE OF ULTRASONIC ACTIVATION ON THE SETTING TIME AND FLOW OF FOUR ENDODONTIC SEALERS

Recebido em: 27/06/2023
Aceito em: 27/07/2023
DOI: 10.25110/arqsaude.v27i7.2023-045

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ABSTRACT: The aim of the present study was to evaluate the influence of ultrasonic activation (UA) on the setting time and flow of four endodontic sealers: AH Plus (AH), Sealer Plus (SP), MTA Fillapex (MTAF), and BioRoot RCS (BIO). Properties were evaluated as required by ANSI/ADA Specification N° 57 (2008); only the size of the specimens was modified. UA was applied using a smooth tapered ultrasonic tip coupled to a piezoelectric ultrasonic device (30% power) on the freshly mixed materials in two cycles of 20 seconds. The results were statistically analyzed using the ANOVA and Kruskal-Wallis tests, followed by the Tukey and Dunn posthoc tests, respectively, depending on the normality of the data. The shortest setting times, initial and final, were, 115 (BIO/UA) and 148.6 (BIO/UA) min, whereas the longest were 1215 (AH) and 1928 (AH) min. The MTAF sealer did not set throughout the experimental period (2880 minutes). Significant differences were observed between BIO and MTAF and the other sealers, with or without UA, both in the initial and final setting time ($P < 0.05$). UA did not change the initial setting times; however, it reduced the final setting of BIO ($P < 0.05$). The highest and lowest flow values observed were 25.52 mm (AH/UA) and 18.66 mm (BIO/UA), respectively. The AH sealer, regardless of UA, exhibited higher flow values compared to the other sealers ($P < 0.05$), except for the MTAF/UA group, which was the only sealer in which UA promoted a significant flow increase ($P < 0.05$). Under the conditions of the study, it can be concluded that the BIO, under UA, presented the lowest setting time; however, it exhibited the lowest flow values. The MTAF sealer did not reach its final setting. Moreover, the SP groups exhibited intermediate results in all analyses. In summary, only the final setting time of the BIO group and the flow values of the MTAF group were influenced by UA.
KEYWORDS: Root Canal Filling; Physico-Chemical Properties; Endodontics; Ultrasonic Energy.

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A INFLUÊNCIA DA ATIVAÇÃO ULTRASSÔNICA NO TEMPO DE AJUSTE E NO FLUXO DE QUATRO VEDADORES ENDODÔNTICOS

RESUMO: O objetivo do presente estudo foi avaliar a influência da ativação ultrassônica (UA) no tempo e fluxo de ajuste de quatro selantes endodônticos: AH Plus (AH), Sealer Plus (SP), MTA Fillapex (MTAF) e BioRoot RCS (BIO). As propriedades foram avaliadas conforme exigido pela Especificação N° 57 (2008) da ANSI/ADA; apenas o tamanho dos espécimes foi modificado. O UA foi aplicado usando uma ponta de ultrassom suave cônica acoplada a um dispositivo ultrassônico piezoelétrico (30% de potência) nos materiais recém-misturados em dois ciclos de 20 segundos. Os resultados foram analisados estatisticamente usando os testes ANOVA e Kruskal-Wallis, seguidos pelos testes póstticos de Tukey e Dunn, respectivamente, dependendo da normalidade dos dados. Os tempos de ajuste mais curtos, iniciais e finais, foram 115 (BIO/UA) e 148,6 (BIO/UA) min, enquanto os mais longos foram 1215 (AH) e 1928 (AH) min. O selador da MTAF não foi colocado durante todo o período experimental (2880 minutos). Foram observadas diferenças significativas entre o BIO e o MTAF e os demais seladores, com ou sem UA, tanto no tempo de ajuste inicial quanto final ($P < 0,05$). A UA não alterou os tempos de ajuste inicial; no entanto, reduziu o ajuste final da BIO ($P < 0,05$). Os valores de fluxo mais alto e mais baixo observados foram 25,52 mm (AH/UA) e 18,66 mm (BIO/UA), respectivamente. O selador AH, independentemente do UA, apresentou valores de fluxo mais elevados em comparação com os outros seladores ($P < 0,05$), exceto para o grupo MTAF/UA, que foi o único selador no qual o UA promoveu um aumento significativo do fluxo ($P < 0,05$). Nas condições do estudo, pode-se concluir que o BIO, sob UA, apresentou o menor tempo de ajuste; no entanto, exibiu os menores valores de fluxo. O selador MTAF não atingiu seu ajuste final. Além disso, os grupos de SP apresentaram resultados intermediários em todas as análises. Em resumo, apenas o tempo de ajuste final do grupo BIO e os valores de fluxo do grupo MTAF foram influenciados pelo UA.
PALAVRAS-CHAVE: Enchimento do Canal Raiz; Propriedades Físico-Químicas; Endodontia; Energia Ultrassônica.

LA INFLUENCIA DE LA ACTIVACIÓN ULTRASÓNICA EN EL TIEMPO DE AJUSTE Y FLUJO DE CUATRO SELLADORES ENDODÓNTICOS

RESUMEN: El objetivo del presente estudio fue evaluar la influencia de la activación ultrasónica (AU) sobre el tiempo de ajuste y flujo de cuatro selladores endodónticos: AH Plus (AH), Sealer Plus (SP), MTA Fillapex (MTAF) y BioRoot RCS (BIO). Las propiedades se evaluaron según lo requerido por la especificación ANSI/ADA N° 57 (2008), sólo se modificó el tamaño de los ejemplares. El AU se aplicó utilizando una punta ultrasónica cónica lisa acoplada a un dispositivo piezoeléctrico ultrasónico (30% de potencia) sobre los materiales recién mezclados en dos ciclos de 20 segundos. Los resultados se analizaron estadísticamente mediante las pruebas ANOVA y Kruskal-Wallis, seguidas de las pruebas postcográficas de Tukey y Dunn, respectivamente, dependiendo de la normalidad de los datos. Los tiempos de fraguado más cortos, inicial y final, fueron 115 (BIO/UA) y 148,6 (BIO/UA) min, mientras que los más largos fueron 1215 (AH) y 1928 (AH) min. El sellador MTAF no se ajustó durante todo el período experimental (2880 minutos). Se observaron diferencias significativas entre BIO y MTAF y los demás selladores, con o sin AU, tanto en el tiempo de ajuste inicial como final ($P < 0,05$). La AU no modificó los tiempos de ajuste inicial, pero redujo el ajuste final de BIO ($P < 0,05$). Los valores más altos y más bajos de caudal observados fueron 25,52 mm

(AH/UA) y 18,66 mm (BIO/UA), respectivamente. El sellador AH, independientemente del AU, presentó valores de caudal más altos en comparación con los demás selladores (P < 0,05), excepto para el grupo MTAF/AU, que fue el único sellador en el que el AU promovió un incremento significativo del caudal (P < 0,05). Bajo las condiciones del estudio, se puede concluir que el BIO, bajo AU, presentó el menor tiempo de fraguado, sin embargo, presentó los menores valores de caudal. El sellador MTAF no alcanzó su ajuste final. Por otra parte, los grupos SP presentaron resultados intermedios en todos los análisis. En resumen, solo el tiempo de ajuste final del grupo BIO y los valores de flujo del grupo MTAF fueron influenciados por el AU.
PALABRAS CLAVE: Llenado de Canales Radiculares; Propiedades Fisicoquímicas; Endodoncia; Energía Ultrasónica.

1. INTRODUCTION

Endodontic therapy comprises a series of interdependent events (ESTRELA et al., 2017) aimed at both biological and clinical success. In this scenario, root canal fillings must provide adequate sealing to prevent fluid percolation and canal reinfection, contributing to the repair of the apical and periapical regions of the root canal system (RCS) - (GUIMARÃES et al., 2014; OSKOCAK et al., 2015).

Endodontic filling materials are used to eliminate the interface between gutta-percha cones and/or between the gutta-percha and the root canal walls, rendering the filling more homogeneous and reducing the risk of infiltration (REIS-ARAUJO et al., 2009; ALMEIDA et. al, 2007). Moreover, it is known that adequate sealing after the chemomechanical preparation of the RCS contributes to the long-term success of endodontic treatment (BOUILLAGUET et al., 2008).

Variations in the chemical composition of endodontic filling materials create sealers with different physical-chemical properties. Therefore, the American National Standards Institute /American Dental Association (ANSI/ADA, Specification N° 57, 2008) established minimal standards regarding radiopacity, setting time, flow, solubility, film thickness, and dimensional change following setting that an ideal endodontic sealer should comply with (ANSI/ADA, 2008). Considering these characteristcs, resinous endodontic sealers are the most used and AH Plus is considered the gold standard (ALMEIDA, 2017; VIANA, 2023).

Among the main properties of an ideal endodontic sealer, flow and setting time stand out. The flow of an ideal sealer should enable the sealing of irregularities, and accessory and lateral canals. Thus, the lack of adequate flow could impair the effective filling of the RCS (BERNARDES et al., 2010). On the other hand, an ideal setting time should be neither too short, which could hinder clinical practice, nor too long, which could increase the release of irritating agents to the periapical tissues (ALLAN et al., 2001).

In addition to the enhancement of physical-mechanical properties through chemical modification, new utilization protocols have been tested to increase the performance of these materials. In this field, the use of ultrasonic activation (UA), which is based on the transmission of acoustic energy from an ultrasonic tip to the material, has been suggested (VAN DER SLUIS et al., 2007).

The UA of sealers generates a more uniform and homogeneous mixture of its components, thereby achieving a reduction in particle size, which promotes greater interaction between the sealer and the dentin (LOPES et al., 2019; SAGHIRI et al., 2020; ALCALDE et al., 2018). Hence, it minimizes the formation of voids inside the filling material and enables a greater adaptation of the sealer to the canal walls and irregularities, as well as greater penetration in the lateral accessory canals (GUIMARÃES et al., 2014; ARSLAN et al., 2016; JIANG et al., 2016; WIESSE et al., 2017).

Given the above, the present study aimed to evaluate the influence of UA on the setting time and flow of four endodontic sealers [AH Plus Jet (Denstsply/DeTrey GmbH, Konstanz, Germany), Sealer Plus (MK Life, Porto Alegre, RS, Brazil), MTA Fillapex (Angelus Ind. Prod. Odont. S/A, Londrina, PR, Brazil) and BioRoot RCS (Septodont, Saint Maur-des-Fosses, France)]. The null hypothesis was that UA would not affect the flow and setting time of these materials.

2. MATERIALS AND METHODS

Four endodontic sealers of different bases were tested: AH Plus Jet (AH), based on epoxy-amine resin, Sealer Plus (SP), based on epoxy resin, MTA-Fillapex (MTAF), based on salicylate resin, and BioRoot RCS (BIO), based on tricalcium silicate. Each sealer was individually allocated to groups based on the use or not of UA, thus resulting in 8 experimental groups (n= 5/group). Information on the composition of the tested sealers is shown in Table 1. The testing procedures were performed according to the ANSI/ADA specification No. 57, 2008.

Table 01. Composition of the endodontic sealers.

Sealer	**Paste A / Powder**	**Paste B / Liquid**
AH PLUS	Bisphenol-A epoxy resin	Dibenzyldiamine
	Bisphenol-F epoxy resin	Aminoadamantane
(Dentsply DeTrey GmbH,	Calcium tungstate	tricyclodecane diamine
Konstanz, Germany)	Zirconia oxide	Calcium tungstate

	Silica Iron oxide pigments	zirconia oxide Silica Silicone oil
SEALER PLUS (MK Life, Porto Alegre, RS, Brazil)	Bisphenol A-co-epichlorohydrin Bisphenol-F epoxy resin Zirconia oxide Silicone and siloxane Iron oxide Calcium hydroxide	Hexamethylethylene tetramine Zirconia oxide Silicone and siloxane Calcium hydroxide Calcium tungstate
MTA FILLAPEX (Angelus, Londrina, PR, Brazil)	Salicylate resin Natural resin Calcium tungstate Nanoparticulate silica Pigments	Thinner resin Trioxide mineral aggregate Nanoparticulate silica Pigments
BIOROOT RCS (Septodont, Saint Maur-des-Fosses, France)	Tricalcium silicate Povidone Zirconia oxide	Calcium chloride Polycarboxylate Water

Source: Own Authorship

2.1 Setting Time

The determination of the setting time followed the parameters established in the specification, in which only the dimensions and the base material of the matrix were adapted. Two acrylic plates (50 mm x 50 mm x 2 mm), 20 wells 4 mm in diameter were prepared and arranged in 4 rows of 5 wells each. Another plate with the same dimensions was coupled and fixed to the perforated plate to serve as the bottom layer.

The sealers were mixed according to the manufacturer's instructions and 0.05 ml of the mixture was poured into the wells using an insulin syringe (BD Ultra-Fine II, Becton Dickinson, Juiz de Fora, MG, Brazil). In the subgroups receiving UA, the mixtures were agitated while still inside the syringe, without touching its walls, using a piezoelectric ultrasonic device (Piezon Master PM100; EMS, Nyon, Geneva, Switzerland) equipped with a smooth conical tip (E5; Helse Dental Technology, Santa Rosa do Viterbo, SP, Brazil) and power setting at approximately 30%. One syringe was used for each specimen. Two activation cycles of 20 seconds were performed, totaling 40 seconds immediately after the sealers were poured into the wells.

To measure the initial and final setting time, Gillmore needles were carefully lowered vertically onto the surface of the sealers. The time between the mixing of the materials until needle indentations ceased to be visible was recorded. The sealers were evaluated after 2 h at intervals of 15 min and later at intervals of 10 min once the indentations started to become less visible. This test was performed under controlled temperature (37±1°C) and relative humidity (95±5%).

2.2 Flow

Immediately after mixing the sealers according to the manufacturer's instructions, 0.05 ml of the filling material was poured on the center of a glass plate (40 mm x 40 mm x 5 mm) using one insulin syringe for each specimen. In the UA subgroups, this procedure was performed with the sealer still inside the syringe, similar to the setting time test.

After 180 s, another smooth and flat glass plate with the same dimensions was placed over the sealer, with a total weight of 120 g. After 10 min, the plate was removed and the largest and smallest diameters of the sealer were measured using a digital caliper (Carbografite, Petrópolis, RJ, Brazil). Specimens in which the difference between the measured diameters was >1.0 mm were replaced. Three repetitions were performed for each experimental group.

2.3 Statistical Analysis

For statistical analysis, the parametric or non-parametric nature of the data was determined. The initial setting time data were classified as non-parametric; therefore, the Kruskal-Wallis and Dunn tests were used. The data resulting from the other analyses were considered parametric and, therefore, ANOVA and Tukey tests were used. Regardless of the test, the significance level was set at $P < 0.05$.

3. RESULTS

The median, minimum, and maximum values of the initial setting time and the mean and standard deviation values of the final setting time and flow are shown in Table 2. Regarding the setting time, the AH sealer had the best results for the final and initial setting, 1215 and 1928 minutes, respectively. For the final setting, significant differences ($P < 0.05$) were observed between AH, SP, and BIO, with and without UA. It was not possible to record the final setting time of the MTAF sealer during the tested period of the study. UA did not affect the initial setting times; however, it reduced the final setting of the BIO sealer ($P < 0.05$).

The highest flow measurement was 25.52 mm (AH/AU) and the lowest was 18.66 mm (BIO/AU). The AH group, regardless of UA, exhibited the highest values compared to the other sealer ($P < 0.05$), except for the MTAF/AU group.

Table 02. Physical properties of endodontic sealers without and with ultrasonic activation.

MATERIAL	UA	Setting time (min) INITIAL MEDIAN	INITIAL MINIMUM	INITIAL MAXIMUM	FINAL MEAN (SD)	FLOW (mm) MEAN (SD)
AH PLUS	W/O	1215 $^{a.B}$	1155	1217	1928.8 (29.8) $^{a.B}$	23.68 (1.31) $^{a.A}$
	W	1190 $^{a.B}$	1149	1210	1920.4 (18.6) $^{a.B}$	25.52 (0.32) $^{a.A}$
SEALER PLUS	W/O	200 $^{a.A}$	195	210	246.6 (3.2) $^{a.A}$	19.96 (0.03) $^{a.B}$
	W	179 $^{a.A}$	175	185	227.8 (5.4) $^{a.A}$	20.96 (0.55) $^{a.B}$
MTA FILAPEX	W/O	1134 $^{a.B}$	1119	1160	-	18.89 (0.92) $^{a.B}$
	W	1068 $^{a.B}$	1059	1084	-	25.34 (0.41) $^{b.A}$
BIO ROOT RCS	W/O	167 $^{a.A}$	160	175	263.0 (4.0) $^{a.A}$	19.53 (0.89) $^{a.B}$
	W	115 $^{a.A}$	105	127	148.6 (11.7) $^{b.A}$	18.66 (1.01) $^{a.B}$

^{a,b} Different lowercase letters represent significant differences within the same sealer group with and without UA, according to the ANOVA and Tukey tests for final setting time and flow, and Kruskal-Wallis and Dunn tests for initial setting ($P < 0.05$); A,B Different uppercase letters represent significant differences between different sealer groups with and without UA, ($P < 0.05$). W= with; W/O= without.

Source: Own Authorship

4. DISCUSSION

According to Versiani et al. (2006), different types of endodontic sealers have been used in combination with gutta-percha to fill the root canal after biomechanical preparation. Furthermore, the use of ultrasonic energy in endodontic practices has been increasing, prompting research on the use of UA in endodontic sealers (ALCALDE et al., 2017), which could improve not only the properties of these materials but also the filling of irregularities and dentinal tubules.

Therefore, the present study evaluated the influence of UA on the setting time and flow of four different endodontic sealers. Our findings indicated that the use of UA affected some of the evaluations, notably the final setting time of BIO and the flow of MTAF. Thus, the null hypothesis was partially rejected.

All tests were performed in accordance with the ANSI/ADA Specification N°57 (2008), which is also in agreement with several other studies (ZHOU et al, 2013; GUIMARÃES et al., 2014; CAMARGO et al., 2017; LEE et al., 2017). However, new specimen dimensions have been proposed, as suggested by Carvalho-Júnior et al. (2007) when analyzing other physical-chemical properties. The modification in specimen size of this study aimed to favor UA, as the material volumes were reduced by approximately 80%. Conversely, the thickness of the specimens was maintained according to the recommendations of the specification.

The AH sealer presented long initial and final setting times, which somewhat contradicts what the manufacturer informs in its specifications, and this finding has been observed in other studies (BALDI et al, 2012; VIAPINA et al., 2014; ZAMPARINI et al., 2022). Using a base-catalyst paste system, its setting reaction occurs as a result of the slow polymerization of epoxy resin amines with high molecular weight, including bisphenol A and bisphenol F (RESENDE et al., 2009). Although the UA groups had a slightly shorter setting time, there was no statistically significant difference among the groups with and without UA ($P > 0.05$).

The use of UA did not affect the flow of the AH sealer, which has also been reported by other studies (ZHOU et al, 2013; LEE et al., 2017). Nevertheless, there was a statistically significant difference compared to SP and BIO with and without UA ($P < 0.05$).

The SP sealer exhibited relatively low setting times given its chemical polymerization, and the values were compatible with the manufacturer's information. The results of the present study also corroborate those of Vertuan et al. (2018), which introduced this sealer. The use of UA did not affect the setting time of the SP sealer ($P > 0.05$). The results of this study found that UA did not affect the flow of the SP sealer, which differs from the findings of a previous study (VERTUAN et al., 2018).

The initial setting time of MTAF was much longer than proposed by the manufacturer. These findings are in agreement with previous studies (FARAONI et al., 2013; ALMEIDA et al., 2020). According to Vitti et al. (2013), the setting reaction of this sealer occurs through two chemical reactions: the progressive hydration of the orthosilicate ions and the reaction between MTA and salicylate resin. In the present study, even in the presence of humidity, the MTAF sealer did not set properly after 48 h of observation, similar to another study (FARAONI et al., 2013). UA positively influenced the flow of MTAF and a statistically significant difference ($P < 0.05$) was observed. MTAF exhibited the highest flow rate and this finding has been reported by other studies (ZHOU et al, 2013; LEE et al., 2017; ALMEIDA et al., 2020; LOPES et al., 2019).

The BIO sealer exhibited the shortest final setting time among the tested materials. Its setting kinetics depends on the interaction between the aqueous vehicle and the tricalcium silicate grains (SILVA et al., 2022). UA significantly influenced its final setting time ($P < 0.05$), which is possibly due to a better mixture of its constituents, favoring greater contact between them and, consequently, accelerating their hardening (ORSTAVIK, 2005). This may explain the slightly shorter setting time for UA sealers in this present study. With UA, a reduction in BIO flow was observed, but without a statistically significant difference ($P > 0.05$). Silva et al. (2022) reported similar findings, corroborating those of the present study.

The UA of endodontic sealers allows polymerization to occur more homogeneously, providing better incorporation of filling particles. Moreover, the heat generated during this process reduces the viscosity of the sealer, increasing its fluidity and the rheological and mechanical properties of the material, mainly its cohesive resistance (ORAL et al., 2012; WIESSE et al., 2017). These characteristics, combined with the increased pressure of the sealer against the root canal walls, enable the filling of irregularities and greater penetration in accessory canals, thereby improving the sealing of the canals, especially in areas of difficult access such as lateral canals, isthmuses, and apical deltas (GUIMARÃES et al., 2014; ARSLAN et al., 2016).

In summary, given the limitations of this study, it can be suggested that the UA can influence the properties of endodontic sealers and thus improve the quality of RCS obturation. Nevertheless, further research should be carried out in this context to prove these results.

5. CONCLUSION

This study evaluated the influence of ultrasonic agitation on setting time and flow of 4 endodontic sealers. Under these conditions, it can be concluded that the BIO/UA had the shortest setting time, albeit it presented the lowest flow. On the other hand, AH showed the longest setting and highest flow with and without UA. MTAF did not reach the final setting; however, it had the highest flow with UA. Therefore, in view of the findings, it is suggested that new studies be carried out to evaluate changes in the physicochemical properties of endodontic sealers with the use of ultrasound, ensuring safety and to improve the filling quality.

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