A COMPARISON OF THE PHYSICOCHEMICAL PROPERTIES OF AH PLUS JET WITH SAMPLES TAKEN FROM THE BEGINNING, MIDDLE AND FINAL PORTIONS OF THE AUTOMIX SYSTEM

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ABSTRACT

Aim: To evaluate physicochemical properties and semi-quantitative elemental analysis of AH Plus Jet with samples from the beginning, middle and final portions of the automix syringe system. Methodology: Three experimental groups based on the source of the material used (beginning, middle and final portion) were established for each of the evaluated properties. Setting time, flow and radiopacity were evaluate following ANSI/ADA n. 57 specification. Set specimens was used in the semi-quantitative elemental analysis in an energy-dispersive X ray spectroscopy and scanning-electron microscopy (EDS/SEM). Statistical analysis was performed using one-way ANOVA followed by Tukey test ($P<0.05$). Results: Flow, setting time, solubility and EDS/SEM tests showed no significant differences among the three portions of the automix syringe ($P>0.05$). Radiopacity test showed significant differences in the beginning of the syringe comparing to the middle and final portions ($P<0.05$). EDS/SEM analysis identified the presence of C, O, Al, Ca, Zr and W. The element Al, however, was found only in the final portion of the syringe. Conclusions: The results of AH Plus Jet suggested an adequate ratio of the components, without segregation between organic and inorganic components, since the results of setting time, flow, solubility, and EDS/SEM analysis presented similar values regardless of the portion of the syringe from where the sealer was taken.

KEYWORDS: AH Plus Jet. physicochemical properties. radiopacity. root canal sealer. solubility.
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INTRODUCTION

AH Plus (Dentsply DeTrey, Konstanz, Germany) is one of the most used and tested root canal sealer, presenting physicochemical properties compatible with international standards[1-3]. It consists of a two-tube system with Paste A - containing epoxy resins- Bisphenol A/F and paste B - containing polyamines Dibenzyldiamine, aminoadamantane and tricyclodecane-diamine. Such as other resin-epoxy-based materials, during mixing, the reaction between the amines and the epoxy monomers is

responsible for the formation of polymers (polymerization). The level of conversion of monomers into polymers achieved in that process influences the physical and mechanical properties of the material[4-5].

It has been previously suggested that the segregation between the organic and inorganic components of the two-tube system AH Plus might affect the physicochemical properties of this sealer according to the portion where the material was taken: from the beginning, middle or final of the tubes in which they were supplied[6]. In fact, the results demonstrated a variation in the values of setting time, flow and radiopacity of AH Plus sealer among the different portions of the sealer tube[6]. Besides that, the mixing of the components of a sealer during the handling of the material is an operator-induced factor, which can result, many times, in disproportionate mixing of the sealer components[7-9]. A better-balanced ratio of sealer components can be provided through a device with automatic proportioning and mixing[9]. The AH Plus Jet (Dentsply DeTrey, Konstanz, Germany) is commercially sold as an auto-mixing syringe with disposable tips, overcoming any operator-induced variability[6,9], resulting in a sealer that, in accordance with the manufacturer, have better final physicochemical properties. However, the physicochemical properties of this sealer, with samples from the different parts of the tube has never been evaluated.

Therefore, the aim of this study was to evaluate the setting time, flow, radiopacity and solubility of AH Plus Jet. The semi-quantitative elemental analysis provided by an energy-dispersive X ray spectroscopy and scanning-electron microscopy (EDS/SEM) was also evaluated. All the results were correlated with the portion of the syringe from where the sample was obtained: from the beginning, middle or final portion of the automix syringe system. The null hypothesis tested was that there would be no differences in the physicochemical properties and in the semi-quantitative elemental analysis of AH Plus Jet regardless of the portion of the tube used.

Table 1. The content of AH Plus Jet.

	AH Plus Jet
Paste A	Bisfenol-A epoxy resin, bisfenol-F epoxy resin, calcium tungstate, zirconium oxide, silica, iron oxide pigments
Paste B	Dibenzyldiamine, aminoadamantane, tricyclodecane-diamide, calcium tungstate, zirconium oxide, sílica, silicone oil

MATERIALS AND METHODS

The syringe-mixed sealer AH Plus Jet (**Table 1**) containing 15 grams of sealer was used in all of experiments and divided in three parts, according to the portion of the sealer the was used: beginning considered as the first 5 grams of sealer taken from the syringe, middle considered as the next 5 grams and final of the syringe considered as the remaining 5 grams. The samples were weighed on the analytical balance after the removal of the automix syringe. These groups were established for the analyses of flow, setting time, radiopacity, solubility and EDS/SEM analysis.

Flow test

The flow test was determined according to the American National Standards Institute/American Dental Association Specification 57 (ANSI/ADA 2000)[10]. Specimens were fabricated from the samples obtained from each portion of the automix syringe (n = 3). The initial, middle or final portion of syringe- mixed AH Plus Jet, were placed onto the center of a polish glass slab (0.5 [± 0.05] mL). A glass plate was placed on the sealer, and a weight (120g of mass) was applied. After 10 minutes of mixing, the weight waslifted and the diameters (maximum and minimum) of the resulting sealer discs were measured by a digital caliper (Mitutoyo Corp, Tokyo, Japan).

Setting time

Setting time was determined according to the American National Standards Institute/American Dental Association Specification 57 (ANSI/ADA 2000)[10]. Metal molds rings measuring 10 mm of diameter and 2 mm of height, were filled with the materials obtained from the different portions of automix syringe (n =3). The rings were filled and maintained under controlled temperature 37 (±1) °C and 95 (±5) % humidity. After the manipulation (10 min), Gilmore needles (113.5g and flat end of 2.0 mm of diameter) were placed on the surface of each specimen. Every 10 min, this procedure was repeated until no more markings could be produced by the tip of the Gilmore needle on the sealer surface.

Radiopacity

The method used was based on the study of Duarte et al.[11]. According to ANSI/ADA Specification 57[10], metallic rings measuring 10 mm of diameter and 1 mm of thickness were filled with the sealer obtained from the different portions of the automix syringe (n =10). Once filled, the rings were stored at 37 (±1) °C and 95 (±5) % humidity, until setting was complete. Once set, the specimens were checked with a digital caliper to determine thickness. Then, the specimens were placed on five occlusal films (Insight; Kodak Company, Rochester, NY, USA), along with a graduated aluminum step-wedge varying in thickness from 2 to 16 mm

(in 2 mm increments). Radiographs were obtained using a radiographic unit (Gnatus XR6010; Ribeirão Preto, SP, Brazil) operating at 60 kV and 10 mA, with a 0.3s exposure time and 30cm focus-film distance. Once processed the radiographs were digitized using a Nikon-D40 with a Nikon 50- mm macro lens (Nikon D40, Tokyo, Japan) and imported into the software Digora 1.51 (Orion Corporation Soredex, Helsinki, Finland). The aforementioned procedure was repeated thrice for each specimen and aluminum step-wedge thickness to determine radiopacity, radiographic density was converted into milimiters of aluminum (mm Al). The final value recorded was the average among the three results.

Solubility

The solubility of the AH Plus Jet sealer was verified according to ANSI/ADA[10] and with the modifications proposed by Carvalho-Jr et al.[12]. Thirty specimens were used for this test (n=10). The cylindrical Teflon molds were filled with the sealers obtained from the different portions of the automix syringe and stored. The samples were removed from the molds and weighed. Each sample was placed inside a plastic container filled with distilled and deionized water (DDW), with the help of a nylon thread (suspension). The containers were sealed. After 24 hours of storage at 95% relative humidity and 37°C, the samples were removed from the containers, rinsed with DDW, and dried with absorbent paper. The DDW was evaluated by means of atomic absorption spectrometry and the samples were dehumidified for 24 hours and weighed once more. This procedure was repeated 5 times for each sealer. The solubility results were determined by the recorded weight loss of each sample.

Energy-Dispersive X-ray Spectroscopy (EDS/SEM)

The samples were prepared according to the portion of the automix syringe from where the sealer was taken, for the EDS/SEM analysis. The sealer was poured inside Teflon discs with 10 mm of diameter by 1 mm of height and kept in an incubator at 37(±1)º C and relative humidity of 100 (±1)%. After setting, the specimens were fragmented and the internal portion of the sealer was analyzed in the SEM (ESEM Zeiss EVO 50, Carl Zeiss, Oberkochen, Germany) connected to an EDS detector (Oxford INCA 350 EDS, Abingdon, Oxfordshire, UK) using a dedicated software with ZAF correction.

The normal distribution of the data was confirmed by the Shapiro-Wilk test (P>0.05). The data was analyzed for statistical differences using the one-way ANOVA and the Tukey HSD post hoc test (P<0.05). All statistical procedures were performed by BioStat 2008 5.0.1 software (AnalystSoft, Walnut, CA, USA).

RESULTS

*Flow test*The results showed no significant differences among the three portions of the AH Plus automix syringe (P>0.05) (**Table 2**). All values found were greater than 20 mm in diameter, in accordance with the ANSI/ADA specification n. 57[10].

Setting time

The results of the setting time showed no significant differences among the three portions of the automix syringe (P>0.05) (**Table 2**).

Radiopacity

The findings showed significant differences in the values of radiopacity in the beginning of the syringe comparing to the middle and final portions (P<0.05) (**Table 2**). All specimens had radiopacity values above the 3-mm limit of Al, as recommended by ANSI/ADA Specification n. 57[10].

Table 2. Mean values and standard deviations of the flow, setting time, radiopacity and solubility for the beginning, middle and final portion of automix syringe.

	Flow (mm)	Setting time (min)	Radiopacity (mmAl)	Solubility
Beginning	40.72 ± 4.32^{A}	774.88 ± 42.47^{A}	15.76 ± 0.46^{A}	0.35 ± 0.41^{A}
Middle	40.54 ± 3.84^{A}	771.29 ± 69.19^{A}	15.04 ± 0.61^{B}	0.16 ± 0.15^{A}
Final	41.2 ± 4.53^{A}	827.86 ± 107.49^{A}	14.85 ± 0.66^{B}	0.38 ± 0.22^{A}

Different superscript letters indicate statistical significantly differences (P<0.05).

Solubility

The results showed no statistical differences among the different portions of the syringe (P>0.05) (**Table 2**). All specimens had solubility values as recommended by ANSI/ADA Specification n. 57[10].

Energy-Dispersive X-ray Spectroscopy (EDS/SEM)

EDS/SEM analysis identified the presence of C, O, Al, Ca, Zr and W. The element Al, however, was found only in the final portion of the syringe. The results showed no significant differences among the three portions of the automix syringe (P>0.05) (**Figure 1**).

DISCUSSION

One benefit of syringe-mixing is that the appropriate proportion of the components is provided by the manufacturer and, by design the mixing is performed in cleaner, quicker and a more consistent manner. This assumption is supported by the results of setting time, flow and solubility of AH Plus Jet sealer which demonstrated similar values regardless of the portion of the syringe from where the sealer was taken. The study of Baldi et al.[6] demonstrated a variation in the values of these properties of AH Plus sealer

depending on the portion of the tube from where the sealer was taken. This is explained by the possible segregation of the components, which can definitely alter the monomer-activator ratio, causing variations in the physico and chemical properties of the material, compromising clinical outcomes.

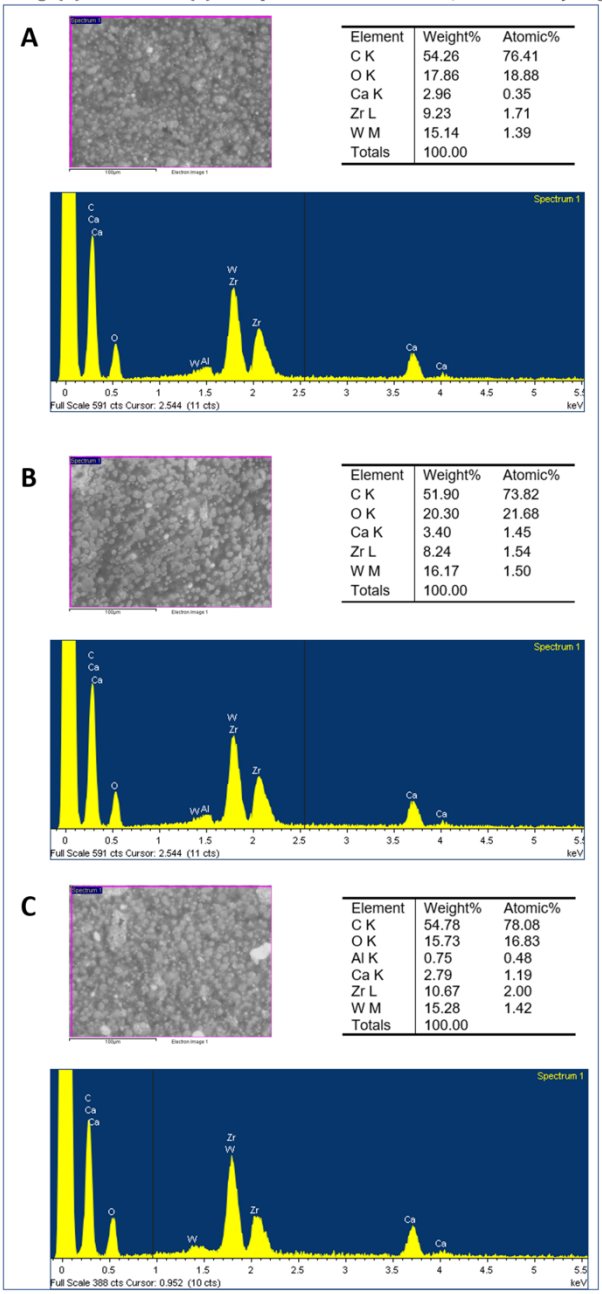
Flow is the physical property which allows the root canal sealer to fill spaces of difficult access. High flow, however, may increase the chance of extrusion of the material toward the periapical area occurring. Thus, the presence of variations in flow depending of the portion of the syringe from which the sealer is collected makes the standardization of the root canal filling technique difficult. The results of the flow test showed no statistical difference regardless of the portion of the syringe. The results differ, however, from the study of Baldi et al.[6], which did find statistical differences among the 3 portions of the AH Plus sealer tube.

While root canal sealers presenting very short setting time may pose a challenge in clinical practice, those with longer setting time are more susceptible to dissolution after root canal filling. Regarding setting time, the ANSI/ADA specification[10] determines a tolerance for variations of up to 10% from what the manufacturer indicates. The manufacturer indicates a setting time of 8h (480 min) for AH Plus Jet, and, therefore, the results obtained in this study (ranging from 774.88 min to 827.86 min) suggest a lack of compliance with the aforementioned norm. In the study of Baldi et al.[6], AH Plus also presented values higher than those prescribed by ANSI/ADA[10], however, the statistical analysis revealed a significant difference in the setting time between the beginning and the final portions of the AH Plus tube.

Although presenting the same chemical composition, the values of radiopacity of AH Plus Jet was higher and revealed less variability when compared to a previous study evaluating the radiopacity of AH Plus in the 3 portions of the tube[6]. Differently from the study of Baldi et al.[6], in which AH Plus sealer presented significant differences when comparing the initial, middle and final portions, in this study, the AH Plus Jet presented differences only in the beginning of the syringe comparing to the middle and final portions. Therefore, the null hypothesis was rejected. These differences among the portions of the syringe were minimal when compared to AH Plus[6].

The solubility test was carried out in the likeness of that from the study of Carvalho-Jr et al.[12], where it was demonstrated that smaller dimensions does not interfere in the results, allowing for lower amounts of material to be used. A previous study shows that the solubility of AH Plus Jet remains within the limits recommended by ISO 6876 even in alkaline pH[13]. The results of the

Figure 1 – Micrographs, graphs and tables representing the results obtained from EDX of the (A) beginning, (B) middle and (C) final portion of the AH Plus Jet automix syringe system.



present study showed no difference among the three portions of the syringe. Moreover, the present results confirm the low solubility of epoxy resins which can be explained by the characteristics of its resinous matrix, more resistant to the solubility[12].

EDS/SEM microanalysis allows for a quick mapping of the sample composition, in which all chemical elements are simultaneously identified and quantified. The results showed no significant differences among the three portions of the automix syringe ($P<0.05$), confirming their homogeneity. However, the element Aluminum was found only in the final portion of the syringe. Limitations of this technique include the possibility of interferences in electron interaction or the propagation of x-rays, which derives from the irregularities on the surface of the samples, along with the fact that small atomic number or small mass fraction chemical elements may not be properly identified, which can cause distortions in the results.

CONCLUSION

Based on the results thereby obtained, AH Plus Jet showed an adequate ratio of the components, since the results of setting time, flow, solubility and EDS/SEM presented similar values regardless of the portion of the syringe from where the sealer was taken.

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CONFLICTS OF INTEREST

No potential conflict of interest relevant to this article was reported.

REFERENCES

1. Marciano MA, Guimarães BM, Ordinola-Zapata R et al. Physical properties and interfacial adaptation of three epoxy resin-based sealers. J Endod. 2011 37:1417-21.

2. Lee JK, Kwak SW, Ha JH, Lee W, Kim HC. Physicochemical Properties of Epoxy Resin-Based and Bioceramic-Based Root Canal Sealers. Bioinorg Chem Appl. 2017 25:828-49.

3. Mendes AT, Silva PBD, Só BB et al. Evaluation of Physicochemical Properties of New Calcium Silicate-Based Sealer. Braz Dent J. 2018 29:536-40.

4. Ferracane JL, Greener EH. Fourier transform infrared analysis of degree of polymerization in unfilled resins–methods comparison. J Dent Res. 1984 63:1093–5.

5. Stansbury JW, Dickens SH. Determination of double bond conversion in dental resins by near infrared spectroscopy. Dent Mater. 2001 17:71-9.

6. Baldi JV, Bernardes RA, Duarte MA et al. Variability of physicochemical properties of an epoxy resin sealer taken from different parts of the same tube. Int Endod J. 2012 45:915-20.

7. Mitchell CA, Douglas WH. Comparison of the porosity of handmixed and capsulated glass-ionomer luting cements. Biomaterials. 1997 18:1127-31.

8. Akhavan H, Zahdabadi F, Mehrvarzfar P, Birjandi A. Comparative study on the microleakage of three root canal sealers. Iran Endod J. 2011 6:1-5.

9. De-Deus G, Scelza MZ, Neelakantan P, Sharma S, Neves Ade A, Silva EJ. Three-dimensional Quantitative Porosity Characterization of Syringe- versus Hand-mixed Set Epoxy Resin Root Canal Sealer. Braz Dent J. 2015 26:607-11.

10. Standard No. 57 for Endodontic Sealing Material. ANSI/ADA; 2000.

11. Duarte MA, Ordinola-Zapata R, Bernardes RA, et al. Influence of calcium hydroxide association on the physical properties of AH Plus. J Endod. 2010 36:1048–51.

12. Carvalho-Junior JR, Correr-Sobrinho L, Correr AB, Sinhoreti MA, Consani S, Sousa-Neto MD. Solubility and dimensional change after setting of root canal sealers: a proposal for smaller dimensions of test samples. J Endod. 2007 33:s1110-6.

13. Silva EJNL, Ferreira CM, Pinto KP, Barbosa AFA, Colaço MV, Sassone LM. Influence of variations in the environmental pH on the solubility and water sorption of a calcium silicate-based root canal sealer. Int Endod J. 2021 54:1394-402.