Water Uptake and Solubility of Acroseal Sealer in Comparison with Apexit and AH Plus Sealers in Hank's Solution

Djurica Grga, Bojan Dželetović, Vesna Miletić, Marija Damjanov

Department of Restorative Dentistry and Endodontics, School of Dentistry, University of Belgrade, Belgrade, Serbia

SUMMARY

Introduction The function of root canal fillings is to seal the root canal system. The important physical property necessary for sealers to achieve this is low solubility. However, any therapeutic effect of calcium hydroxide-based sealers is dependent on the calcium hydroxide being in ionized form, which implies that the material must be at least partly soluble.

Objective The objective of our study was to compare weight changes of Acroseal and Apexit, conventional calcium hydroxide-based sealer and AH Plus, epoxy-amine resin sealer in Hank's solution at different exposure times.

Methods The standardized samples of each material were weighed and immersed in the Hank's solution for 1 h, 24 h, 96 h, 14 days and 28 days. After these exposure times, they were removed, dried, and weighed again. Mean weight changes were determined and the differences between sealers were analysed statistically using a one-way ANOVA.

Results The highest differences in mass were observed in Apexit, 1.52%, and were significantly different from Acroseal, 0.93% ($p<0.05$) and AH Plus, 0.45% ($p<0.05$). There were no significant differences between Acroseal and AH Plus, except for 96 h period.

Conclusion Under the conditions of our study, it may be concluded that the Acroseal sealer presented the behaviour more like epoxy-based material, AH Plus, than calcium hydroxide sealer, Apexit.

Keywords: root canal filling materials; solubility; Acroseal; Apexit; AH Plus

INTRODUCTION

The primary goal of successful endodontic therapy is the complete obturation of the root canal. It is well established that the sealer cement is an extremely important component of the root canal filling necessary for achieving three-dimensional obturation of the canal space. The function of root canal fillings is to seal the root canal system and prevent microorganisms and/or their toxic products from reaching the periodontal tissues [1].

Possibly the most important physical property necessary for sealers to achieve this is low solubility. Low solubility of root-canal sealers has been introduced as a requirement in the International Standard ISO 6876 for root-canal sealing materials. This standard requires that endodontic sealing materials do not exceed the maximum weight loss of 3% after storage in distilled water for 24 hours. Moreover, the sealers should be of low solubility because the components leaching from the root-canal filling may have undesirable biological effects on the surrounding tissue [2, 3].

Since root-canal filling materials may be in direct contact with periapical tissues for a prolonged period of time, their biocompatibility is of primary importance. The use of improved "biologic" sealers based on calcium hydroxide has been proposed for permanent sealing of the root canal system [4]. The two most important reasons for using calcium hydroxide as a root-filling material are the stimulation of the periapical tissues in order to maintain health or promote healing and secondly for antimicrobial effects. Any therapeutic effect of this type of sealer is dependent on the calcium hydroxide being in ionized form. This implies that the material must be at least partly soluble [5, 6]. One study reported that the release of calcium and hydroxyl ions from the calcium hydroxide-containing sealers may be variable, and this could be attributable to the differences in the disintegration rate of the sealers as a consequence of their composition [7].

OBJECTIVE

The objective of this study was to compare the weight changes of Acroseal and Apexit, conventional calcium hydroxide-based sealer and AH Plus, epoxy-amine resin, which is frequently used as a control material in research after immersion in Hank's solution at 1 h, 24 h, 96 h, 14 days and 28 days exposure times.

METHODS

Three different root canal sealers were tested in this study: Acroseal (Septodont, Saint Maur des Fosses, France), Apexit (Ivoclar Vivadent,

Correspondence to:
Djurica GRGA
School of Dentistry
Dr Subotića 8, 11000 Belgrade
Serbia
djuricag@eunet.rs

Schaan, Liechtenstein) and AH Plus (Dentsply, Konstanz, Germany). Samples were immersed in the Hank's solution, whose ingredients are listed in Table 1.

The tests recorded weight differences before and after the immersion of test specimens. Stainless steel ring moulds of an internal diameter 20.0±0.1 mm and a height of 1.6±0.1 mm were used for sample preparation. All moulds were cleaned with acetone in an ultrasound bath for 15 minutes. All moulds were weighed three times prior to use (accuracy: ±0.0001 g). The mean values were calculated.

The mould was supported by a glass plate of larger dimensions, covered with a cellophane film, and filled by the sealer using the mixer tip, supplied by the manufacturer, according to manufacturer's instructions. All samples were left to set on a grating in a cabinet at 37°C for 24 h and 95% relative humidity. Twenty-five samples were prepared from each sealer and then divided into five groups of 5, ready for immersion in Hank's solution for 1 h, 24 h, 96 h, 14 days and 28 days. Prior to the immersion of the samples, all sealers in their moulds were weighed (Acculab ALC-110.4, Goettingen, Germany) three times and the average reading was recorded.

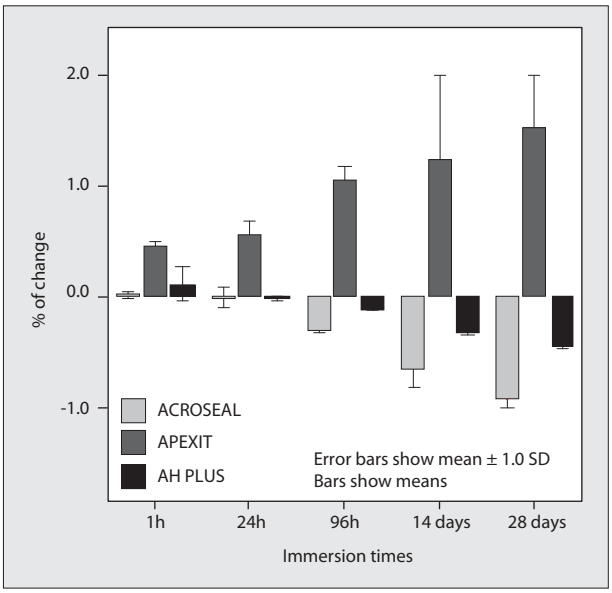
The samples were suspended by nylon thread and placed inside a plastic vessel, containing 50 mL of Hank's solution so that both surfaces of each sample were freely accessible to the liquid. The plastic vessels, in which the samples were placed, were sealed and left in an incubator at 37°C and 95% relative humidity for the specified immersion period. There was no agitation of the samples. Hank's solution was changed at weekly intervals. As controls, in terms of solvent action on metal moulds, 5 empty moulds were immersed in Hank's solution for 1 h, 24 h, 96 h, 14 days and 28 days and any changes in weight were recorded.

The samples of sealers were removed from the plastic vessels after the specified immersion period and rinsed with 3 mL of double-distilled water and allowed to dry for 24 h at 37°C in an oven, as described by Schafer et al. [8]. Thereafter, the samples were weighed three times and the mass of the sealer was determined. The differences in mass between the original weight of sealer and its final weight were calculated as a percentage of the original mass.

The differences in mass between sealers were assessed by analysis of variance (one-way ANOVA; $p<0.05$) using commercially available software (SPSS 10.0, SPSS, Chicago).

Table 1. Ingredients of the Hank's solution (filled up with distilled water to 1 kg)

Ingredient	Content
$CaCl_2 \cdot 2H_2O$	186 mg/L
KCl	400 mg/L
KH_2PO_4	60 mg/L
$MgSO_4 \cdot 7H_2O$	200 mg/L
NaCl	8000 mg/L
$NaHCO_3$	350 mg/L
$Na_2HPO_4 \cdot 7H_2O$	90 mg/L
Glucose	1000 mg/L



Graph 1. Weight changes of sealers for different immersion times

Table 2. Mean weight changes of sealers over time

Material	1 h	24 h	96 h	14 days	28 days
Acroseal	0.014	-0.004	-0.3	-0.6	-0.9
Apexit	0.46	0.56	1.05	1.2	1.5
AH Plus	0.11	-0.08	-0.12	-0.3	-0.4

RESULTS

There was no change in the weight of empty moulds after the immersion in solution at any exposure times. The results for all sealers, with respect to time, are shown in Graph 1. By the end of immersion time of 28 days AH Plus, the epoxy-based material, showed fewer mass differences than other sealers. The biggest differences in mass were observed in Apexit, 1.52%, and were significantly different from Acroseal, 0.93% ($p<0.05$) and AH Plus, 0.45% ($p<0.05$), (Table 2). While Apexit showed progressive weight loss throughout the experiment by indicating that dissolution exceeded water uptake, Acroseal and AH Plus showed a gain in mass, representing water sorption. Although the differences in mass recorded for Acroseal for immersion times up to 96 h were lower, and higher at exposure times greater than 96 h, there were no significant differences between Acroseal and AH Plus, except at exposure time of 96 h.

DISCUSSION

Property such as solubility of the root canal filling materials is associated with the integrity and stability of the canal wall/sealer or sealer/ gutta-percha, being directly related to the desired hermetical sealing [9, 10]. High solubility of root canal sealers is undesirable because it may result in increases in bacterial leakage [3, 5, 8, 9] and cause the release of materials that could irritate periapical tissues [3, 5]. No laboratory test can completely simulate in vivo conditions. However, Donnelly et al. [11] suggested that

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solubility studies should be regarded as a form of accelerated aging and laboratory studies remain useful screening techniques that must be properly interpreted. In such studies materials are prepared with very large surface areas that are directly exposed to excess solution, and if materials do not absorb water or loose dry mass under these conditions, then they might have better clinical performance.

In the present study weight changes of test specimens were recorded by determining the difference in mass of the sealers samples after storage in Hank's solution, although the International Standard ISO 6876 (2001) suggests that the increase in weight of the dish in which the samples have been placed should be ascertained as the amount of material removed from the specimens. The specimens were weighed in order to avoid an underestimation of the material going into solution, if a constituent of the eluate is lost by volatilization during the course of evaporation [12]. Furthermore, the drying process of the specimens after immersion in Hank's solution may lead to evaporation of volatile components in the sealer [12]. According to Wilson et al. [12], the test only truly measures the extent of the decomposition of the material when the degradation products are water-soluble. In order to remove loose debris of decomposition, all samples were washed with double-distilled water after the immersion period [8]. The test immersion time described in the International Standard ISO 6876 (2001) was supplemented by longer immersion periods as it was suggested by Wilson et al. [12]. Also McMichen et al. [5] stated that in the root canal, the sealer may be exposed to tissue fluid and exudates and it is therefore necessary to determine the effects of prolonged exposure of the sealers to fluid. In the present study Hank's solution was used in order to mimic tissue fluids.

Wilson et al. [12] stated that solubility of a solid when strictly defined as a physicochemical term can only be applied to the situation where a pure chemical compound is in thermodynamic equilibrium with its solution and with regard to this definition the test used in the present study measured the elution of water-soluble material, but not the solubility.

It has to be taken into account that there are two competitive processes that take place; one is fluid uptake and the other dissolution, and the effect produced is dependent on the material type. Water uptake may compensate for dissolved material [13]. Our results demonstrate that the ability to absorb fluid of Acroseal and AH Plus is greater and oApexit is lower than the rate of their dissolution under the conditions of the present study. This could be explained by water sorption suffered by resins after polymerization of Acroseal and AH Plus [14], and zinc stearate in the content of Apexit, which is known to be highly hydrophobic and thus prevents water ingress [15]. The findings of the present study are in agreement with previously published results of Donnelly et al. [11] reporting in the case of AH Plus, a slow gain in mass over time. But according to the results of McMichen et al. [5] for AH Plus sealer, dissolution exceeded water uptake. This is probably the result of longer immersion period of 12 weeks. The progressive weight loss of Apexit obtained in this study was similar to that presented by McMichen et al. [5].

The differences in mass recorded were the lowest for Acroseal for immersion times up to 96 h but at exposure time greater than 96 h those values were the lowest for AH Plus. An explanation for this could be the finding of Eldeniz et al. [15] that Acroseal calcium ion release, as a result of solubility, was lower for experimental periods up to 96 h than experimental periods greater than 96 h.

CONCLUSION

On the basis of our results obtained under the conditions of the present study, it may be concluded that the Acroseal sealer presented the behaviour more like epoxy-based material, AH Plus, rather than calcium hydroxide sealer, Apexit.

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Поређење растворљивости и упијања воде ендодонтског силера *Acroseal* са пастама *Apexit* и *AH Plus* у Хенковом раствору

Ђурица Грга, Бојан Џелетовић, Весна Милетић, Марија Дамјанов

Клиника за болести зуба, Стоматолошки факултет, Универзитет у Београду, Београд, Србија

КРАТАК САДРЖАЈ

Увод Ендодонтски силери се користе за заптивање каналног система корена зуба. Важна физичка особина неопходна да се то постигне јесте мала растворљивост. Међутим, терапијски ефекат силера на бази калцијум-хидроксида зависи од јонизованости овог једињења, што значи да материјал мора бити бар делимично растворљив.

Циљ рада Циљ истраживања био је да се упореди промена масе пасти *Acroseal* и *Apexit*, као конвенционалних силера на бази калцијум-хидроксида, и пасте *AH Plus*, као силера који се састоји од епокси-амин смоле, у Хенковом раствору при различитим временима излагања.

Методе рада Стандардизовани узорци сваког материјала мерени су и потапани у Хенков раствор на један сат, 24 сата, 96 сати, 14 дана и 28 дана. После ових временских интервала узорци су извађени из раствора, сушени и поново мерени. Разлике између средњих вредности промене масе силера статистички су анализиране применом једносмерног *ANOVA* теста.

Резултати Највећа разлика у маси уочена је код пасте *Apexit* (1,52%), која је била статистички значајно већа него код пасти *Acroseal* (0,93%; $p<0,05$) и *AH Plus* (0,45%; $p<0,05$). Статистички значајне разлике између силера *Acroseal* и *AH Plus* није било, изузев за период од 96 сати.

Закључак Наше истраживање је показало да се *Acroseal* понаша сличније силеру базираном на епокси-амин смоли (*AH Plus*), него пасти на бази калцијум-хидроксида (*Apexit*).

Кључне речи: пасте за пуњење канала корена; растворљивост; *Acroseal*; *Apexit*; *AH Plus*

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