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Effect of pH and Storage Time on The Solubility Behaviour of Different Endodontic Sealers

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The aim of this study was to evaluate the solubility of 5 types of root canal sealers in artificial saliva with 3 different pH values and at 5 different times. For standardized samples (n = 75 per group), ring moulds were filled with epoxy resin (AH 26, AH Plus), polyketone (Diaket), silicone (RSA) and calcium hydoxide (Sealapex)-based sealers. The samples were immersed in distilled water and artificial Saliva (pH values: 4.5, 5.7 and 7.) 1, 4, 7, 14 and 28 days and dried in the desiccator for 0.5 h in order to re-weigh. Then, the mean weight loss was determined. The difference between the first and second measurements was recorded as the total amount of material removed. The results were analyzed by univariate ANOVA and Duncan test at $p < 0.05$. It was detected that the type of the sealer, storage media and storage time are statistically significant ($p < 0.001$). Results show that the silicone (RSA) based sealer and epoxy resin (AH Plus) had significantly less artificial saliva sorption than the other root canal sealers evaluated. There were significant differences among the materials ($p < 0.001$).

Key Words: Degradation, Endodontic sealers, Solubility.

INTRODUCTION

The use of a sealer during root canal obturation is essential for success and a basic concept is that sealer is more important than the obturating material. Sealer must be used in conjunction with the obturating material regardless of the technique or material used. This makes the physical properties and placement of the sealer important[1].

Sealers serve as filler for canal irregularities and minor discrepancies between the root canal walls or in the tubules[2,3] and may avoid an environment for bacterial colonization. One of the most important properties determining the durability of canal sealers in the root canal is resistance against dissolution and disintegration. When degradation of sealers consists, gaps along the sealer-dentine or the sealer-gutta percha interface appear. These gaps may be blaze passage of microorganisms and their products in to the periapical tissues[4]. Hence, insolubility of root canal sealers may have a major impact on the success rate of root-canal treatment. The pH of oral fluids may vary from pH 4 to pH 8.5 representing a range from mildly acidic to mildly alkaline. Highly acidic soft drinks and use of chalk-containing

tooth pastes extend this range from a lower end of pH 2 up to pH 12. It is possible for a material to be stable at near neutral pH values but erode rapidly at extremes of either acidity or alkalinity[5].

Literature shows that few studies have been carried out on the solubility of root canal sealers. The calcium hydroxide- containing sealer Sealapex is also believed to be soluble over time, but little experimental work is available to confirm this observations[6]. Nonetheless zinc oxide eugenol-based sealers are generally associated with certain degree of weight loss after storage in water, ranging from *ca.* 7 % to less than 1 %[7,8].

On the contrary, it has been demonstrated that silicone[7], epoxy resin[8] and polyketone[7,8] based sealers have a relatively low solubility in water. Material should not be dissolve, erode or corrode. The solubility of a material is simply a measurement of the extent to which dissolve in a given fluid like water or saliva[5].

The aim of this study is to evaluate that pH and storage time have any influence on the water sorption and solubility behaviour of 5 root canal sealers.

EXPERIMENTAL

In this study, 5 root canal sealers, determining 4 different chemical classes of materials were used: epoxy resin (AH 26, AH Plus), polyvinyl resin (Diaket), silicone (RSA) and calcium hydroxide (Sealapex)-based sealers (Table-1). Sealers were mixed in accordance with the manufacturers' instructions. Because it is well known that calcium-hydroxide-based sealer requires moisture for setting[9], Sealapex was mixed with a spatula moistened with tap water. Freshly mixed sealers were placed in moulds. Standardized stainless steel moulds with 8 mm diameter and 2 mm height were used and a 4 mm diameter hole drilled centrally for specimen placement (4 mm diameter × 2 mm thick). All molds were cleaned with acetone for 15 min and weighed 3 times before use. All weight measurements throughout the study were in g recorded to 4 decimal places.

TABLE-1
ROOT CANAL SEALERS USED IN PRESENT STUDY

Materials	Chemical nature	Company
AH 26	Epoxy resins	Dentsply, DeTrey GmbH, Konstanz, Germany
AH Plus	Epoxy resins	Dentsply, DeTrey GmbH, Konstanz, Germany
RSA	Silicone	Roeko, Langenau, Germany
Sealapex	Calcium hydroxide	Kerr,Salerno, Italia
Diaket	Polyketone	3M ESPE AG, Seefeld, Germany

All samples were left to set at room temperature for 48 h. Excess material was then trimmed level to the surface of the mould with a scalpel. From each sealer, 75 samples were prepared. Thus, a total of 375 samples were prepared for this study. Prior to immersion of samples, all sealers in their moulds were weight thrice and the average reading was recorded. The specimens were stored in artificial saliva[10]

(NaCl, 0.400 g; KCl, 0.400 g; $CaCl_2 \cdot H_2O$, 0.795 g; NaH_2PO_4, 0.69 g; $Na_2S \cdot 9H_2O$, 0.005 g; urea 1.0 g; distilled water 1000 mL) that has different pH value ranged 4.5, 5.7 and 7.0 at 37 °C. The pH was then adjusted with HCl and the volume made up to 1 L.

At room temperature, sealer samples were immersed in 20 mL of solution. The immersion was such that both surface of each sample were readily accessible to the solution. There was no agitation. After 1, 4, 7, 14 and 28 days consequently, samples of sealers were removed from the dish after the specified immersion period using a pair of tweezers, touching only the metal mould and dried in the desiccator for 0.5 h in order to re-weigh Thereafter, using an electronic analytical balance, the amount of sealer removed from the specimen was determined as difference between the original weight of the sealer and its final weight.

The mean and standard deviation values of weight loss were calculated at each time interval for each group of specimens. The values were compared by factorial analysis of variance using the software SPSS 10.0 (SPSS inc., Chicago, USA). Multiple comparison intervals were further performed to identify statistically homogeneous subsets ($p = 0.05$) using post hoc Duncan test ($p < 0.05$). Differences between the six sealers within each solution were analyzed with univariate ANOVA and Duncan test ($p < 0.01$).

RESULTS AND DISCUSSION

According to the variance analysis, the type of the sealers and storage time were found statistically significant, on the other hand, pH was not found to significant on solubility of experimental sealers. The silicone-based sealer RSA (RoekoSeal) and epoxy resin-based sealer AH Plus showed lower solubility than other sealers in each of three media. They presented statistical similarity in this solubility. The most soluble sealer was sealapex in all other 4 sealers. The solubility of different root canal sealers with different storage media was shown in Fig. 1. In addition, the solubility of cements in different period of time was shown in Fig. 2.

The orders with respect to solubility were found to be as follows: RSA = AH Plus < Diaket < AH 26 < Sealapex.

The sealers should be soluble in a solvent. Different sealers have different degrees of solubility in different solvents and with varying mechanical techniques[1].

International organization for standardization 6876:2001[11] is available that describes the procedure to determine the solubility of set sealer in water. According to the instructions given by this standard, ring moulds should have an internal diameter of 20 mm and a height of 1.5 mm. The method used in present investigation was oriented, to a great extent, by using these descriptions because in the present investigation the samples were 4 mm in diameter and 2 mm thick. This form is corroborated by Whitworth and Boursin[12]. According to ISO, 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 speciemens[11,13].

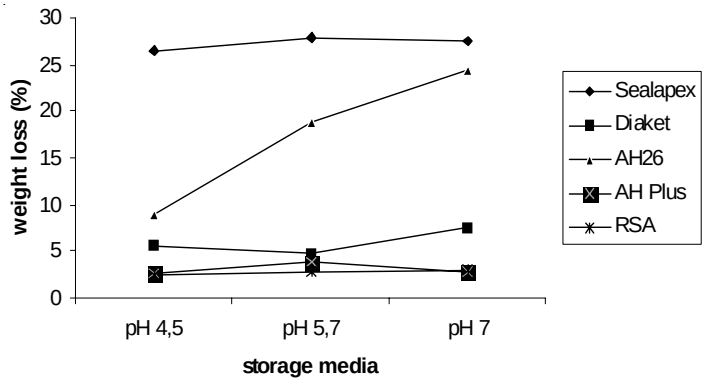

Fig. 1. Dissolution of sealers in different storage media

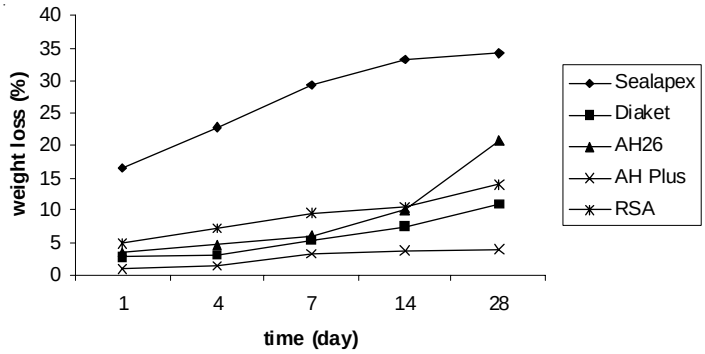

Fig. 2. Dissolution of sealers in different times

The specimens were weighed in order to avoid an under estimation of the material going in to solution. For enhancing the correctness of the measurements, one sample was used for just one immersion period.

It has been suggested that dilute acids should be used for solubility tests rather than distilled water in order to mimic tissue fluids. In current study, artificial saliva adjusted to 3 different pH values (4.5, 5.7 and 7.0) was used. Levine *et al.*[14] reported that the use of artificial saliva might produce a setting very much closer to an oral media and this might be of clinical significance.

Resin-filling materials have steadily gained popularity and are now accepted as a root canal filling. Resin cements such as AH Plus or AH 26 are renowned for their low solubility. Mc Michen *et al.*[15] demonstrated that the rate of dissolution of used materials in their study (AH Plus, Apexit, Endion, Roth 801 and Tubli-Seal EWT) is much higher than their ability to absorb fluid in all cases. Moreover, the sorption values recorded were lowest for AH Plus, indicating a small overall weight loss and the dissolution exceeded water uptake.

In present study, AH Plus and RSA showed the least weight loss of all sealers tested. However, AH 26 showed moderate solubility. This observation corroborates the results of previous studies[16]. According to Kazemi *et al.*[7], this may be a result of

polymer degradation of unreacted hexamethylene tetramine and its break down to ammonia and formaldehyde.

The addition of the calcium hydroxide to the sealers, thereby increasing the pH of the material, is claimed to create a therapeutic material that can be inductive of hard-tissue formation. Although an osteogenic response has been observed[17,18], the solubility of the calcium hydroxide sealers[6,9] have been questioned.

The calcium hydroxide-based sealer sealapex was significantly more soluble in artificial saliva than all other sealers used at all exposure times. The result obtained in this study largely confirmed those in previous reports[6,9,16]. In present study, the weight loss after final desiccation of the material shows the amount of material lost over the test period. The results of this study showed that there was no difference storage pH on solubility of 5 root canal sealers for all of exposure times.

Conclusion

Within the limits of this *in vitro* investigation, it is concluded that: (i) there were significant differences in the solubility profiles of 5 root canal sealers in artificial saliva, (ii) exposure time was important for the solubility of sealers tested, (iii) it was not statistically significant difference between the solubility values of stored solutions (acidic, basic and neutral artificial saliva), and (iv) the highest and the lowest solubility were presented by sealapex, AH Plus = RSA, respectively.

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