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RESEARCH ARTICLE

SOLUBILITY OF 5 DIFFERENT ROOT CANAL SEALERS IN WATER AND ARTIFICIAL SALIVA

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ABSTRACT

Aim: To compare the solubility of 5 different root canal sealers in water and in artificial saliva in 24, 48 and 72 hours.

Methodology: For standardized rings were filled with epoxy resin (AH-Plus), Silicon based (Roekoseal), Calcium hydroxide based (Apexit plus), MTA based (MTA Fillapex), Zinc oxide eugenol (Prime Dental). These samples were then immersed in distilled water and artificial saliva for 24 hrs, 42 hrs and 72 hrs. The weight loss of the sample was determined by comparing the weight pre test and post test. The data was calculated and analysed using one way ANOVA test.

Result: Among all resin based root canal sealer (AH-Plus) has showed less solubility in water and artificial saliva after 24hr, 48hrs and 72 hrs followed by Silicon based sealer- Roekoseal, Calcium hydroxide based – Apexit Plus, MTA based – MTA Fillapex, Zinc oxide eugenol sealer – prime dental. Zinc oxide eugenol based sealer has shown more solubility in saliva than in water. MTA Fillapex has shown more solubility in water than all other sealers.

Conclusion: AH-Plus has shown less solubility than all other sealers independent of any medium used, MTA Fillapex has shown more solubility in water. Zinc oxide eugenol has shown more solubility in saliva.

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INTRODUCTION

Obturation materials are used in root canal therapy (RCT) to entomb the residual microorganisms or their toxins, fill the inaccessible areas, and seal the canal in order to prevent coronal leakage which is a major cause of RCT failure (Niloofar *et al.,* 2012). The physical properties necessary for materials meeting this function include insolubility or at least low solubility. Degradation of the sealer may cause gaps at dentin/sealer or gutta-percha/sealer junctions, which can facilitate bacterial proliferation and colonization (Niloofar *et al.,* 2012). These spaces may provide an environment for bacterial colonization and passage of microoganisms and their products into the periapical tissues. Therefore, low water solubility of sealers has a major impact on success, longevity, and prognosis of RCT (Niloofar *et al.,* 2012). The quality of the seal obtained with gutta-percha and conventional zinc oxide eugenol (ZOE) sealers is not perfect. Hence, several new resin cement sealants have been developed to be used instead of ZOE and to improve the root canal seal beyond that currently possible with conventional materials (Niloofar *et al.,* 2012).

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The calcium hydroxide containing sealer is also believed to be soluble over time, but little experimental work is available to confirm this. On the contrary it has been demonstrated that silicon & epoxy resin based root canal sealers have a relatively low solubility in water (Schafer and Zindbiglari, 2003). Mineral trioxide aggregate (MTA) was initially introduced in Endodontics for the sealing of root and retrofilling perforations because of its favourable physical, chemical, and biological properties. Not withstanding, to be used as endodontic sealer, its formulation has to be upgraded to improve its flowing, setting time and bond strength (Meiryelen *et al.,*). MTA Fillapex (Angelus, Londrina, PR, Brazil) is a MTA-based endodontic sealer currently launched in Brazilian dental market and little studies on its physical-chemical properties have been conducted. Therefore, the aim of this study was to evaluate pH and solubility of MTA Fillapex and to compare its results with those of other endodontic sealers that have been used in clinical practice (Sealer 26, Sealapex and AH Plus) (Meiryelen *et al.,*). Evaluation of the solubility of the root canal sealers has, in general been based on studies in which the weight loss of set sealers in distilled water has been determined. To date, no other solubility media have been used in order to mimic inflammatory exudate, tissue fluids or saliva. Low solubility of root canal sealers has been introduced as a requirement in the International Standard 6876 (2001) for root canal sealing materials. According to this standard, the

solubilitiy of the set sealer shall not exceed 3% mass fraction after immersion in water for 24h (Schafer, 2003). Therefore, the aim of this in vitro study was to apply the method proposed to compare the solubility of all other 5 root canal sealers.

MATERIALS AND METHODS

5 chemically different root canal sealers were included in this study.

Stainless steel ring moulds having an internal diameter 20.0±0.1mm and a height of 1.6±0.1 mm were used for sample preparation. All moulds were cleaned with acetone for 15 min. All moulds were weighed thrice prior to use. The moulds were placed on a glass slab and filled to slight excess with the mixed sealer using a 2ml syringe avoiding air entrapment. All sealers were mixed in accordance with manufacturer's instructions. As it is well known that Apexit require moisture for setting, these sealers were mixed with a spatula moistened with tap water. All samples were left to set in a cabinet at 37°C for 24hrs and

Materials	Description	Manufacturer
GROUP 1: AH-PLUS	Paste A	DENTSPLY, GERMANY
	Bisphenol-A epoxy resin	
	Bisphenol-F epoxy resin	
	Calcium tungstate	
	Zirconium oxide	
	Silica	
	Iron oxide pigments	
	Paste B	
	Dibenzyldiamine	
	Aminoadamantane	
	Tricyclodecane-diamine	
	Calcium tungstate	
	Zirconium oxide	
	Silica	
	Silicone oil	
GROUP 2: ROEKOSEAL	Polydimethylsiloxane	COLTENE WHALEDENT,
	Silicon oil	GERMANY
	Paraffin-base oil	
	Platinum catalyst	
	Zirconium dioxide (radiopaque material)	
GROUP 3: MTA FILLAPEX	Salicylate resin	ANGELUS, LONDRINA, PR, BRAZIL
	Diluting resin	
	Natural resin	
	Bismuth Trioxide	
	Nanoparticulated silica	
	MTA	
GROUP 4: APEXIT PLUS	Calcium salts (hydroxide, oxide, phosphate)	IVOCLAR VIVADENT
	Hydrogenized colophony	
	Disalicylate	
	Bismuth salts (oxide, carbonate)	
	Highly dispersed silicon dioxide (silanized)	
	Alkyl ester of phosphoric acid	
	Zinc oxide	PRIME DENTAL
GROUP 5: ZINC OXIDE EUGENOL	Sodium borate	
	Barium sulphate	
	Bismuth subcarbonate	
	Hydrogenated Rosin	
	Eugenol	

Solubility was determined in distilled water and in artificial saliva, which was prepared from Nikhil analytical Labrotary. The artificial saliva used in this present study were prepared according to Macknight – Hane and Whitford (1992) formula (Artificial saliva preparation).

The composition of artificial saliva (grams per litre):

Methyl – p – hydroxybenzoate	2.00
Sodium carboxymethyl cellulose	10.00
KCl	0.625
$MgCl_2.6H_20$	0.059
$CaCl_2.2H_2O$	0.166
K_2HPO_4	0.804
KH_2PO_4	0.326

The pH of artificial saliva was adjusted to 6.75 with KOH

100% relative humidity. Prior to the immersion of the samples, all sealers in their moulds were weighed thrice and the average reading was recorded. All weight measurements were in grams, recorded to four decimal places. All samples containing sealers were immersed in a fresh solutions of artificial saliva and distilled water. Samples were immersed in solutions in distilled water and artificial saliva for 24h, 48h and 72h. There was no agitation of the dish. Samples of sealers were removed from the dish after the specified immersion period using a pair of tweezers, touching only the metal mould. Samples were washed with 3ml of distilled water. The samples were weighed thrice and the mass of the sealer was determined. The difference in mass was calculated as a percentage of the original weight of the sealer. The difference in the solubility of each sealer in the different liquids was assessed by analysis of variance. Differences between the eight sealers with respect to their solubility in each liquid were analysed using a one-way ANOVA.

RESULTS

Group 1: The epoxy-based materials AH-Plus were of low solubility. The weight loss of AH-Plus range from 0.10% - 0.21%. Thus, AH-Plus was virtually insoluble in all liquids. There were no significant differences between the weight loss of AH-Plus in distilled water and artificial saliva ($P > 0.05$).

Group 2: The silicone-based sealer RSA Roekoseal showed a low solubility in artificial saliva and distilled water. It showed less solubility in artificial saliva as compared to distilled water. The solubility range from 0.71% - 1.23%. There was no significant differences in the solubility in either of the two liquids ($P > 0.05$).

Group 3: The MTA based sealer MTA Fillapex showed a less solubility in artificial saliva as compared to distilled water. The solubility range from 10.6% - 11.4%. There was no significant difference in the either of the two liquids ($P > 0.05$).

Group 4: The Calcium hydroxide-based sealerApexit Plus showed a less solubility in artificial saliva and distilled water. The solubility range from 1.1% - 1.45%. There was no significant difference in either of the two liquids ($P > 0.05$).

Group 5: The Zinc-oxide Eugenol based sealer showed a more solubility in artificial saliva and less solubility in distilled water. The solubility range from 12.6% - 14.7%. There was significant difference in either of the two liquids ($P < 0.05$).

DISCUSSION

Experimental set-up

The procedure to determine the solubility of set sealer in water is described in the International Standard ISO 6876 (2001) as well as British Standard BS 6934 (1998). The solubility tests performed in the present study followed to a great extent the methodology of this International Standard (Niloofar *et al.,* 2012). However, whilst weight loss of the test specimens was recorded by determining the decline in mass of the sealers after storage in the different liquids (Schafer, 2003). Furthermore, to our knowledge all other studies set the sealers in uniform conditions, while it might not comply with the manufacturers different instructions for different brands. Thus in this study, the setting time was determined exactly according to the manufacturers to reproduce *in vivo* conditions (Schafer, 2003). The low coefficients of variation calculated may indicate low sample dispersion and thus high reproducibility, consistency, and reliability of the methods used (Schafer, 2003).

The specimens were weighed in order to avoid an underestimation of the material going into solution. For instance, it is well known that when the residue method is applied to zinc oxide-eugenol cements, eugenol, the major constituent of the eluate, is lost by volatilization during the course of evaporation and hence is not estimated (Wilson, 1976). Moreover, it has been shown that the best indication of the extent of the disintegration can be obtained by weighing the specimens before and after the test (Wilson, 1976). However three of the experimental materials showed an increasing trend which seemed to have a possibility to exceed that level in delayed course. The solubility of the two newly tested epoxy resin-based sealers favorably ceased to increase, and their results were statistically similar. After the immersion period, all samples were washed with distilled water in order to remove loose debris of decomposition (Wilson, 1976). Moreover, sealers differing by less than 0.5% in solubility could be separated with statistical significance. Based on these findings, the experimental method seemed appropriate. Solubility of a solid is the situation, where a pure chemical compound is in thermodynamic equilibrium with its solution (Wilson, 1976). Moreover, it has to be taken into account, that measuring weight differences of the sealer specimens may also record disintegration processes that may not be the result of dissolution (Wilson, 1976, Orstavik, 1983). Lack of solubility has also been stated as an ideal characteristic for root-end filling materials. Solubility is the ability of a substance to dissolve in another, expressed as the concentration of the saturated solution of the former in the latter. For instance, filler particles of the material may fall out from the sealer structure during storage in the liquids. Furthermore, water uptake may compensate for dissolved material. This aspect might have an effect on the changes in weight of zinc oxide-eugenol and glass-ionomer based sealers, according to the findings reported by Wrbas et al; and finally, the drying process of the specimens after immersion in the different liquids may lead to evaporation of volatile components in the sealer (Schafer, 2003). It has been suggested that dilute acids or culture medium should be used for solubility tests rather than distilled water in order to mimic tissue fluids. In the present study, distilled water as well as artificial saliva solutions adjusted to PH 6.75 were used (Artificial saliva preparation). Saliva was included in the study in order to investigate the solubility of the root canal sealers in the case of coronal leakage under acid conditions developed in stagnation areas. This might be of clinical significance because acid conditions can occur orally either by ingestion of acidic food or by the degradation of polysaccharides to acids because of the action of various streptococci and bacilli. Distilled water was used as solubility medium in order to provide a baseline for solubility studies.

Weight loss of sealers

Under the conditions of the present study, AH-Plus showed the least weight loss of all sealers tested, independent of the solubility used. AH-Plus was less soluble than all other sealers. On average, these sealers showed less than 2%, independent of the storage medium. AH-26 has showed more solubility as compared to AH-Plus in any other medium. According to the other authors, this may be a result of polymer degradation of unreacted hexamethylenetetramine and its break down to ammonia and formaldehyde (Schafer, 2003). This polymer degradation of unreacted hexamethylenetetramine may also be an explanantion for the higher solubility of AH 26 in comparison to AH-Plus (Schafer, 2003). This phenomenon and also the higher rate of solubility observed in this material might be attributable to degradation of unreacted hexamethylenetetramine polymer and its breakdown to ammonia and formaldehyde. It should be taken into consideration that the differences in surface-to-volume values of the specimens as well as other experimental configurations such as molds used and setting times might contribute to the differences in the results (Schafer, 2003). The calcium hydroxide containing sealers Apexit was significantly showed less solubility in artificial saliva than water. The reason for that might related to the content of zinc stearate in Apexit, which is known to be highly hydrophobic, thus preventing an ingress of water. In the study given by Schafer and Zandbiglari (2003)

Sealapex being compared with Apexit and has shown more solubility with Sealapex because it has a poorly formed matrix and demonstrated a very water sorption (Schafer, 2003). It was assumed that this porous material permits marked ingress of water over time that promotes continued reaction between powder and binder. This could be the cause of the high solubility of sealapex. As a physical property of a material, the insolubility can greatly impact on endodontic treatment success rate. Moreover, endodontic sealers must have low solubility because the leaching of their components can generate undesirable biological effects on the surrounding tissues (Claudio *et al.,* 2010). The endodontic filling materials are designed to be kept inside root canals to promote an impermeable sealing at long term and to eliminate any communication route between oral cavity and periapical tissues. Consequently, the low solubility level for these materials is of extreme importance (Claudio *et al.,* 2009). Borges *et al*. described that AH Plus and MTA Angelus sealer demonstrated to be soluble within the recommended range (Schafer, 2003), while MTA Fillapex and Sealapex sealers exhibited values higher than those recommended by the American National Standards Institute / American Dental Association results that corroborate with the findings of this present study, in which Sealapex sealer presented a higher level of mass loss, reaching 12.5%, while MTA Fillapex reached 11.4%.

The zinc oxide eugenol based sealer exhibited marked weight loss after 28 days, which correlates marked with the result of other authors. This high degree of solubility is probably a result of the leaching out of excess and non-reacted eugenol as well as the hydrolysis reaction of hardened zinc eugenolate. At exposure to saliva it showed more solubility in saliva than in distilled water. Zinc oxide eugenol based materials are hydrolytically decomposed according to the following equation (Schafer, 2003 Schafer, 2003):

$$Zn\,(C_{10}H_{11}O_2)_2 + 2H_2O = Zn(OH)_2 + 2C_{10}H_{12}O_2$$

Thus, in neutral solutions this water insoluble zinc hydroxide will adhere to the sealer and will not be transferred to the solution and measured. The requirement for compliance with the International Standard (2001) has been set at a weight loss of not more than 3% after storage in distilled water for 24h. Whilst sealers clearly exceeded the proposed maximum weight loss, the majority of materials tested met the standard (Claudio *et al.,* 2009).

Conclusion

Within the limitation of this laboratory study, most sealers were of low solubility, zinc oxide eugenol sealers followed by MTA Fillapex had a marked weight loss in all liquids. Thus, the method proposed by the ISO should be supplemented by the used of acidic test media if a more valid assessment is to be achieved.

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