Solubility of root canal sealers: A comparative study

Claudio Poggio¹, Carla Renata Arciola², Alberto Dagna¹, Marco Colombo¹, Stefano Bianchi¹, Livia Visai³

¹Department of Operative Dentistry, University of Pavia, Pavia - Italy
²Research Unit on Implant Infections of the Rizzoli Orthopedic Institute of Bologna and Experimental Pathology Department of the University of Bologna, Bologna - Italy
³Department of Biochemistry and Center for Tissue Engineering (CIT), University of Pavia, Pavia - Italy

ABSTRACT

Introduction: Root canal sealers used in odontological applications are capable of providing an apical seal, thus inhibiting potential bacterial penetration into the periradicular tissues. Since insolubility is the ideal characteristic for a root canal sealer material, in dentistry the sealability of the material depends on its solubility.

Purpose: This study tested the solubility of six different root canal sealers: two ZnOE-based sealers (Endomethasone C and Argoseal), two calcium hydroxide-containing sealers (Bioseal Normal and Acrosseal), and two resin-based sealers (AH Plus and MM Seal).

Methods: Ten specimens of each material were prepared using stainless steel ring molds with the same internal diameter and the same height in order to obtain similar samples, and immersed in water. Solubility was determined by using sample weight loss (%) after 24 hours and after 2 months. The test was performed according to the International Standards Organization 6876 standard and the American Dental Association specifications No. 30 and No. 57. Data were statistically analyzed with a one-way analysis of variance test (ANOVA).

Results: The ANOVA test showed significant differences between the various groups. The post-hoc test showed that resin-based root canal sealers had significantly lower solubility percentages than other groups (p<0.05), and showed no difference between them (p>0.05).

Conclusions: All the materials fulfilled the requirements of International Standard 6876 and ANSI/ADA specifications demonstrating a weight loss of less than 3%. Under the conditions of the present study, all root canal sealers were of low solubility, but the lowest solubility percentages were recorded in resin-based root canal sealers, which can be considered virtually insoluble, and thanks to this physical property, ideal for use as endodontic sealer.

KEY WORDS: Endodontic root canal sealers, Solubility, Weight loss

Accepted: July 28, 2010

INTRODUCTION

Endodontic root canal sealers are used in association with gutta-percha and have several functions to perform. The first is to guarantee the seal of the root canal system of the tooth, thereby entombing residual microorganisms and preventing ingress of any bacteria from the oral environment and their passage to periapical tissues (1-4). They further aid the seating of the master gutta-percha cone and act as a binding agent between the gutta-percha and the canal wall, filling residual spaces (5). Sealers accomplish the objective of providing a fluid tight seal: they must always be used in conjunction with the filling material, regardless of the technique or material used (6). Root canal sealers, even if used only as adjunctive materials in the obturation of root canal system, have been shown to influ-
ence the result of root canal treatment (7).

In 1988 Grossman (8) outlined the criteria for an ideal sealer, even though none of the endodontic sealers currently available possess all these properties: the sealers and their components should cause neither tissue destruction nor cell death, so they could not be toxic; they should remain dimensionally stable on setting; they should provide adequate working time for manipulation and placement into the root canal; they should have adhesion, performing bonding between dentin and obturation material; sealers also should be visible on radiographs; they should not cause staining of the crown of the teeth; they should not encourage bacterial growth; they must create and maintain a seal apically, laterally and coronally; they should be soluble in a solvent (when retreatment is necessary) but they should contemporaneously not disintegrate when in contact with tissue fluids.

As we said, none of the root canal sealers available today possess all these ideal characteristics, but some have more than others. The physical properties for filling materials include insolubility, or at least the lower solubility (9); this property is of primary importance for a root canal sealer, since successful results of endodontic therapy depend mostly on integrity of the sealer component (1, 2). Degradation of the sealer may cause gaps and voids along the sealer/dentin or the sealer/gutta-percha interface: these spaces can provide a pathway for microorganisms and their toxic products into periapical tissues (10, 11). Therefore insolubility of root canal sealers may have a great impact on the success rate of root canal treatment.

Today the major types of endodontic sealers are Zinc Oxide Eugenol (ZnOE) based sealers, resin-based sealers, and calcium hydroxide-containing sealers; other variations and compounds have been proposed or are marketed as sealers, but these should be considered experimental (6). The ZnOE-based sealers represent the gold standard in endodontics (12), because of their long history of successful usage, making their positive qualities outweigh their negative aspects, like staining, slow setting time and solubility (13). Resin-based sealers were introduced some years ago and today are accepted worldwide; epoxy-based cements are the primary ones, with many useful properties like antimicrobial action, adhesion to dentin walls, ease of using and mixing, good sealability and relative insolubility (6). Calcium hydroxide-containing sealers, in which the calcium hydroxide is incorporated in a ZnOE or is resin-based, have also been introduced. These sealers are claimed to have antimicrobial effects and biologic properties that stimulate a calcific barrier at the apex, although these characteristics have not yet been conclusively and completely demonstrated (6).

Low solubility of a root canal sealer has been introduced as a requirement in the International Standard 6876 (14) for root canal sealing materials. According to this standard and the ANSI/ADA Specifications No. 57 (15), and No. 30 (16), the solubility of a sealer shall not exceed 3% mass fraction after immersion in water for 24 hours.

The purpose of this in vitro study was to compare solubility at 24 hours and at 2 months of six different root canal sealers: two ZnOE-based sealers (Endomethasone C and Argoseal), two calcium hydroxide-containing sealers (Bioseal Normal and Acroseal) and two resin-based sealers (AH Plus and MM Seal). The null hypothesis tested was that there is no significant difference in solubility percent-ages among the six different endodontic sealers.

MATERIALS AND METHODS

Six commercially available endodontic root canal sealers representing three different classes of materials were tested in this study. Table I shows the chemical composition and characteristics of the tested sealers. The solubility of Endomethasone C, Argoseal, Bioseal Normal, Acroseal, AH Plus and MM Seal was determined in accordance with American Dental Association (ADA) specifications No. 57 (15) and No. 30 (16) and with the International Standards Organization (ISO) 6876 method (14). This International Standard specifies requirements for “materials used for permanent obturation of the root canal with or without the aid of obturating points”. Stainless steel ring molds with an internal diameter of 20 ± 0.1 mm and a height of 1.5 ± 0.1 mm were used for sample preparation. All molds were cleaned with acetone in an ultrasound bath for 15 minutes. All molds were weighed 3 times before use (accuracy ± 0.0001 g) on a Mettler AE-163 balance (Mettler, Toledo, Spain), which was used throughout the experiment. The molds were placed on a glass plate and filled to slight excess with the mixed materials. All root canal sealers were mixed by the same operator in accordance with manufacturer’s instructions. After filling the molds, another glass plate covered with a Mylar strip was placed on top of the molds, exerting a light pressure in order to remove any excess material. Ten sets of specimens for each mate-

ISSN 0391-3988
© 2010 Wichtig Editore - ISSN 0391-3988 677
Solubility of root canal sealers

The resins were prepared in one operation. All samples were left to set for 24 hours on a grating in a cabinet at 37°C and 100% relative humidity. The samples in their molds were then exposed to air for 15 minutes, weighed 3 times and the average reading was recorded to 3 decimal places. The specimens of each material were individually placed in tarred bottles, containing 5 mL of distilled water. The bottles were then transferred to an oven at 37°C where they remained for 24 hours. They were removed from the oven and rinsed with distilled water, which was then collected in the same bottles. After that, the water was evaporated at a temperature slightly below boiling point. Bottles and residues were dried in an oven at 105°C, cooled down in the same desiccator and weighed. The differences found between this weight and the original bottle weight were divided by the initial dry weight of the specimens and multiplied by 100. The result was recorded as solubility (17-19). The solubility test was performed again at 2 months using the same method (19).

Differences in solubility percentages were calculated and a statistical analysis was performed with computer software (Stata 7; College Station, TX, USA). Analysis of variance (ANOVA) was applied to determine whether significant differences existed among the groups. For the

| TABLE I - CHEMICAL COMPOSITION AND CHARACTERISTICS OF TESTED MATERIALS |
|-----------------|-----------------|-----------------|-----------------|
| Type of sealer  | Sealer          | Manufacturer     | Chemical Ingredients                               |
| ZnOE-based      | Endomethasone C | Septodont, Saint-Maur, France | Powder Hydrocortisone acetate, thymol iodide, barium sulphate, zinc oxide, magnesium stearate |
|                 |                 |                 | Liquid Bidistilled eugenol |
| ZnOE-based      | Argoseal        | Dental Ogna, Milan, Italy | Powder Silver, zinc oxide, natural resin, diiodothymol |
|                 |                 |                 | Liquid Bidistilled eugenol, purified oleoresin |
| Calcium hydroxide-containing | Bioseal Normal | Dental Ogna, Milan, Italy | Powder Zinc oxide, calcium hydroxide, natural resins, barium sulphate, hydroxyapatite |
|                 |                 |                 | Liquid Purified oleoresin in bidistilled eugenol |
| Calcium hydroxide-containing | Acroseal       | Dental Ogna, Milan, Italy | Paste A Glycerethic acid (enoxolone), methenamine, radiopaque excipient |
|                 |                 |                 | Paste B Calcium hydroxide, DGEBA, radiopaque excipient |
| Resin-based (epoxy) | AH Plus        | Dentsply Maillefer, Konstanz, Germany | Paste A Bisphenol-A-diglycidylether, calcium tungstate, ZrO₂ |
|                 |                 |                 | Paste B Adamantine amine, diamines, silicon oil |
| Resin-based (epoxy) | MM Seal        | MicroMega, Besancon, France | Paste A Epoxy oligomer resin, ethylene glycol salicylate, calcium phosphate, bismuth carbonate, zirconium oxide |
|                 |                 |                 | Paste B Poly-aminobenzoate, triethanolamine, calcium phosphate, bismuth carbonate, zirconium oxide, calcium oxide |
post-hoc test, Tukey’s test was used. Significance for all statistical tests was predetermined at p<0.05.

RESULTS

The results of the solubility test after 24 hours and after 2 months are listed in Table II. The null hypothesis of the study was rejected. The ANOVA test showed significant differences between the various groups. The post-hoc test showed that groups 1 and 2 reported significant lower solubility percentages than other groups (p<0.05) for both observation periods, and showed no difference between them (p>0.05). The highest solubility percentages were recorded in groups 3, 4, 5 and 6, which showed no significant difference between them (p>0.05). All the materials fulfilled the requirements of the International Standard 6876 (12) and ANSI/ADA specifications No. 57 (15) and No. 30 (16), demonstrating a weight loss of less than 3%. The weight loss of the six root canal sealers after two months was not statistically significant, thus, the materials were virtually insoluble.

DISCUSSION

The purpose of inserting a root canal sealer into the canal is to provide an apical seal that is able to inhibit the leakage of irritant substances or pathogen microorganisms from the root canal system into the periapical tissues (19, 20). Sealability helps to hamper the creation of a locus minoris resistentiae prone to the colonization by microorganisms of oral microflora or by new emerging pathogens, thus preventing inflammation and infection (21-23).

Solubility is a very important factor in assessing the suitability of potential substances to be used as restorative materials in dentistry. 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 (24, 25). The solubility of the set material, when determined in accordance to International Standard 6876 (14) or ANSI/ADA Specifications No. 57 (15) and No. 30 (16), shall not exceed 3% mass fraction after immersion in water for 24 hours. The findings of this study demonstrated that all tested materials were within the recommended range.

Some studies have been carried out on the solubility of root canal sealers (2). ZnOE-based sealers are generally associated with a certain degree of weight loss after storage in water, ranging from approximately 7% to less than 1% (9, 10, 26, 27); the calcium hydroxide-containing sealers are also believed to be soluble over time (28, 29). On the other hand, it has been demonstrated that resin-based sealers, and specifically epoxy resin-based ones, have a relatively low solubility in water (9, 10, 30).

Evaluation of the solubility of root canal sealers has, in general, been based on studies in which the weight loss of set sealers in distilled water has been determined. The ISO 6876 standard (14) describes the procedure for determining the solubility of set of root canal sealers in water. Because endodontic sealers in the apical region can be in direct contact with periapical tissues, the solubility test performed in the present study followed this procedure.

### TABLE II - SOLUBILITY PERCENTAGE AFTER 24 HOURS AND 2 MONTHS AND STANDARD DEVIATION (SD) OF WEIGHT LOSS FOR EACH MATERIAL AND FOR EACH IMMERSION PERIOD

<table>
<thead>
<tr>
<th>Group</th>
<th>Material</th>
<th>Solubility after 24 hours in percentage and SD</th>
<th>Solubility after 2 months in percentage and SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>MM Seal</td>
<td>0.38% (0.05)</td>
<td>0.41% (0.06)</td>
</tr>
<tr>
<td>2</td>
<td>AH Plus</td>
<td>0.32% (0.09)</td>
<td>0.33% (0.1)</td>
</tr>
<tr>
<td>3</td>
<td>Bioseal</td>
<td>0.85% (0.12)</td>
<td>0.88% (0.11)</td>
</tr>
<tr>
<td>4</td>
<td>Acroseal</td>
<td>0.81% (0.1)</td>
<td>0.87% (0.09)</td>
</tr>
<tr>
<td>5</td>
<td>Endomethasone C</td>
<td>0.73% (0.2)</td>
<td>0.75% (0.14)</td>
</tr>
<tr>
<td>6</td>
<td>Argoseal</td>
<td>0.77% (0.15)</td>
<td>0.81% (0.1)</td>
</tr>
</tbody>
</table>
In this study all root canal sealers demonstrated good results: none of the sealers exceeded 3% mass fraction after immersion in water for 24 hours, in accordance with ISO 6876 (14) and ANSI/ADA Specifications No. 57 (15) and No. 30 (16). The highest solubility percentages were recorded in samples obtained with ZnOE-based cements (Endomethasone C and Argoseal) and with calcium hydroxide-containing cements (Bioseal Normal and Acroseal), as confirmed in the literature (9, 10, 26-29).

While ZnOE-based sealers have represented the gold standard in endodontics for a number of years because of their long history of successful usage and their positive qualities (13), and calcium hydroxide-containing sealers have antimicrobial effects and biologic properties that have yet to be completely demonstrated (4), this in vitro study shows that epoxy resin-based sealers have significantly lower solubility percentages than other materials. Combined with their many confirmed useful properties (like antimicrobial action, adhesion to dentin walls, ease of use and mixing, and good sealability), epoxy resin-based sealers like AH Plus and MM Seal could be considered the new gold standard in endodontics.

CONCLUSIONS

Within the limitations of this in vitro study, root canal sealers showed minimal solubility, which is an essential physicochemical property of root-filling materials. The solubility test results of all the investigated endodontic sealers conformed to International Standards Organization 6876 (14) and American National Standards specifications No. 57 (15) and No. 30 (16) for endodontic filling materials: the solubility percentage of these materials were considered acceptable for ISO and ANSI/ADA specifications, but the best results were obtained for epoxy resin-based sealers (AH Plus and MM Seal), as confirmed by the literature.

Conflict of interest statement: The authors of this study have no conflict of interest to disclose.

Address for correspondence: Claudio Poggio Policlinico “San Matteo” Piazzale Golgi, 3 27100 Pavia, Italy e-mail: c.poggio@unipv.it

REFERENCES

16. ANSI/ADA. Revised American National Standard/American


