Research Article

Comparative in-Vitro Evaluation of Material Properties of Commonly Used Root-End Filling Materials

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Abstract

Background: Standard guidelines for performing conventional endodontic procedures have undergone a number of modifications recently. An ideal characteristics of a root end filling materials include dimensional stability; radio-opacity; non-resorbability; compressibility, adequate working and quick setting time; biocompatibility especially in peri-radicular area; bioactivity and easy handling properties.

Objectives: This study evaluated and compared different root-end filling materials by determining their solubility, sorption and fluid uptake after immersion in deionized water over a period of 28 days.

Methods: Materials were mixed a per manufacturer's instructions and disc-shaped specimens were made (15mm×1mm). After 24h curing, discs were immersed in deionized water and incubated at 37±1◦C. At specified time intervals, measurements for mass and volume of discs were taken to calculate fluid sorption, solubility and uptake.

Results: Fluid uptake of MTA was 0.029±0.0025g at day 1 and 0.066±0.004g at day 28 whereas, for Acroseal it was 0.0006±0.0001g at day 1 and 0.006±0.001g at day 28. Fluid uptake of MTA and Acroseal increased with passage of time but for Biodentine fluid uptake decreased from day 1 (0.017±0.005g) to day 28 (0.008± 0.005g). Acroseal exhibited lowest values of fluid sorption (0.006±0.0005%) and solubility (0.0006± 0.00005%) whereas bio-dentine exhibited maximum values of fluid sorption (0.06±0.007%) and solubility (0.04±0.03%).

Conclusions: Percent water solubility and sorption of Biodentine is higher than MTA and Acroseal, whereas its fluid uptake decreases over a period of 28 days in comparison to MTA and Acroseal, indicating its stability and thus making Biodentine a better root-end filling material.

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Introduction

Standard guidelines for performing conventional endodontic procedures have undergone a number of modifications recently.[1] According to a study, of all the endodontic procedures performed, 5.5% end up in having either root end surgery or perforation repair.[2] The class of materials to be used in these circumstances should not only be able to form and maintain an efficient water-resistant seal but also should have the ability to perform in presence of tissue fluids, unlike most restorative materials. An ideal characteristics of a root end filling materials include dimensional stability; radio-opacity; non-resorbability; compressibility, adequate

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working and quick setting time; biocompatibility especially in peri-radicular area; bioactivity and easy handling properties.[2,3]

Several materials have been used by the clinicians over ages for root repair and as root end filling materials including amalgam, resin, composites, ethoxybenzoic acid cements, Cavit™, glass ionomer cements, gutta-percha, zinc oxide eugenol cements, polycarboxylate cement and biomaterials like calcium hydroxide-based cements and calcium silicate based cements.[2,4] In tissue fluids, Root repair cements should exhibit little solubility in tissue fluids to avoid leakage into the root canal system to prevent treatment failure. Solubility evaluation is carried out using standardized material samples, weighed pre and post immersion in distilled water as per ANSI/ADA specification No. 57 or ISO6876:200216.[5]

Mineral Trioxide Aggregate (MTA) and Biodentine are commercial bioactive calcium silicate-based endodontic cements.[6] MTA is widely accepted material as a retrograde filling material in endodontic therapy since its introduction in 1993 by Dr Torabinejad.[2,3,5] However, like any other biomaterial, MTA is far from being ideal due to its difficult handling and long setting times of about 175mins approx. which may result in loss of marginal integrity and ingress of bacteria into the peri-radicular tissue.[1,2,7] Biodentine, a bioactive restorative and reparative cement, has similar indications and properties as MTA but with superior setting time.[5,7] Biodentine's liquid component comprises of a water reducing agent and calcium chloride for accelerating the setting reaction.[1,7]

Comparative data regarding the behavior of root end filling materials upon coming in contact with fluid is limited. So, the basic aim of this study was to investigate and compare the solubility, sorption and fluid uptake of two most commonly used calcium silicate–based materials (Biodentine and MTA) and a conventional long-standing calcium hydroxide-based material (Acroseal).[4]

Methods

Materials used in this study were divided into three groups, the compositions and relevant product details are given in Table 1. Test disc were prepared using Teflon molds with an inner diameter 15±1mm and a thickness of 1±0.1mm as specified in ISO 4049; 2009 (9). The materials were mixed according to manufacturer's instructions, separating medium was applied to the molds and the mixed material was placed in them. This assembly was then placed in an incubator and allowed to set for 24h at 37±1°C. Three discs (15mm×1mm) were made for each material (n=3) for all the tests performed.

The samples were removed after 24h from the incubator, demolded carefully, dried and weighed on an analytical balance to record 'm1' as their dry mass, up to an accuracy of ±0.1µg. Vernier caliper was used to measure the diameter and thickness of each sample to an accuracy of 0.01mm. These readings were then used to calculate volume 'V' of each sample. The samples were dipped in 10mL of deionized water in labelled falcon tubes and placed at 37±1°C in an incubator. After one day samples were removed and dried using filter paper. These were then weighed 1 minute after being removed from the storage solution to an accuracy of 0.1µg. Their mass was recorded as 'm'. This process was repeated for all the samples at seven intervals i.e. 1, 7, 14, 21 and 28 days respectively.

The fluid uptake of each material was calculated at each specified time after taking the average of dry mass 'm1', average of mass at specified time 'm' and average of volume 'V' of the three readings obtained for each material. Equation 1 was then used for calculating the fluid uptake by the sample disc:

$$Fluid\,uptake(\%) = m - mL/V \times 100 \text{ \_\_\_\_ } equation\ 1$$

Evaluation of Sorption and Solubility: After 28 days, mass (m2) of the samples was recorded, which represented the fully saturated mass. The samples were stored at 23± 1°C for 24h in a desiccating jar containing silica gel until a stable reading for mass could be recorded. This constant mass (m3) was recorded as. Fluid sorption and solubility for each test sample was calculated after taking out the average of all the readings obtained. Equations 2 & 3 were used for calculating fluid sorption and solubility of the materials respectively:

$$Fluid\,Sorption(\%) = m2 - m3/V \times 100 \text{ \_\_\_ } equation\ 2$$

$$Fluid\,Solubility(\%) = m1 - m3/V \times 100 \text{ \_\_\_ } equation\ 3$$

Results

SPSS version 21 (SPSS software, IBM, USA) was employed for data analysis and interpretation. Means and standard deviations for fluid uptake, sorption and solubility were calculated for all the experimental groups. Repeated measure ANOVA with post-hoc tukey test was employed to calculate the statistically significant difference in fluid uptake at five different time intervals between and within the experimental groups respectively. One-way ANOVA with post-hoc tukey test was used for calculation of statistically significant difference in fluid sorption and solubility within and between the study groups respectively. $p \leq 0.05$ was taken as significant.

Table 1: *Experimental materials (n=3), Grouping and Relevant Product Details*

Group	Commercial Name	Company Name	Composition
(M)	MTA	Angelus	Tri-calcium silicate 51% Di-calcium silicate 10% Tri-calcium aluminate 11% Ferroaluminate tri-calcium 3% Calcium oxide 5% Bismuth oxide 20%
(B)	Bio Root RCS	Septodont	**Powder component:** Tricalcium silicate 80.1% Dicalcium silicate 14.9% Zirconium Oxide 5% Iron oxide traces **Liquid:** Calcium chloride (accelerator) Hydrosoluble polymer (water reducing agent)
(C)	Acro-seal	Septodont	**Base paste:** Hexamethylene tetramine Bismuth carbonate Hydrogenated Rosin Paraffin oil Venice terpentine Enoxolone **Catalyst paste**: Bismuth carbonate Calcium hydroxide Diglycidyl ether Bisphenol A Yellow iron oxide

The means and standard deviations for fluid uptake by the three study groups are shown in Figure 1. The observed fluid uptake at all time intervals i.e. day 1, 7, 14, 21 and 28, was highest for MTA and lowest for Acroseal. At day 1, fluid uptake of MTA was 0.029±0.0025g while at day 28 it was 0.066±0.004g whereas, for Acroseal it was 0.0006±0.0001g at day 1 and 0.006±0.001g at day 28. Fluid uptake of MTA and Acroseal increased with passage of time but for Biodentine the fluid uptake decreased from day 1(0.017±0.005g) to day 28(0.008 ±0.005g). A statistically significant difference in fluid uptake was observed within all experimental groups at different time intervals (p-value = .000). Fluid uptake at all time intervals was significant between Biodentine and MTA (p value = .000) and between MTA and Acroseal (p-value = .000). Fluid uptake at all time intervals was not significant between Biodentine and Acroseal (p value = .151).

The percentage sorption and solubility of the experimental groups are given in Figure 2. Acroseal exhibited lowest values of fluid sorption (0.006 ±0.0005%) and solubility (0.0006±0.00005%) whereas Biodentine exhibited the maximum values of fluid sorption (0.06 ± 0.007%) and solubility (0.04 ± 0.03%) amongst the tested materials. MTA showed fluid sorption of 0.04±0.002% and solubility of -0.015±0.002%. Acroseal and MTA demonstrated negative values for fluid solubility. One-way ANOVA shows a statistically significant difference between and within all experimental groups for both fluid solubility and fluid sorption. Post Hoc Tukey test shows a statistically significant difference in fluid solubility between Biodentine and MTA only and between all experimental groups for fluid sorption.

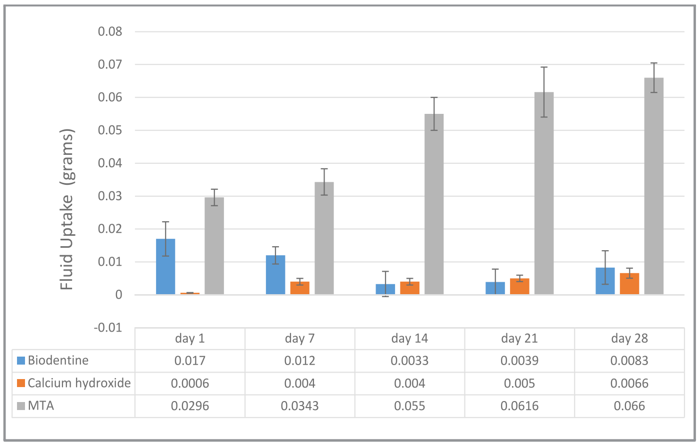

Figure 1: *Fluid Uptake of Test Materials at Different Time Intervals (Days)*

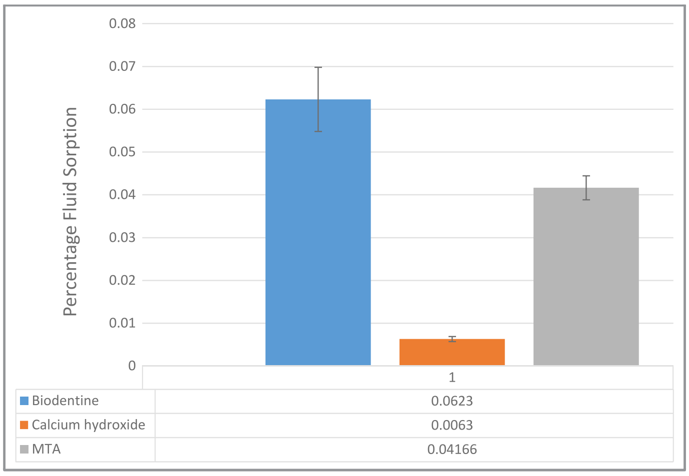

Figure 2: *Percentage Fluid Sorption of the Test Materials*

Discussion

The present study tested three commercially available root-end filling materials i.e. Biodentine, MTA Angelus and Acroseal, for sorption, solubility and fluid uptake over a period of one month. Retrograde root filling materials are in contact with peri-radicular tissue fluid following application, so it is vital to evaluate them for their solubility, sorption and fluid uptake characteristics over a longer periods of time as these properties may affect the sealing ability, bio-compatibility and overall success of the endodontic treatment.[2,3,8]

Solubility is defined as the amount of material that will dissolve in a given amount of solvent. American Dental Association (ADA) and International Organization for Standardization (ISO) have defined standards for required properties of root-end filling materials,[9] according to these solubility is determined by measuring the weight of the prepared samples before and after immersion in distilled water.[9] Loss of mass from that of initial mass is expressed as percentage solubility of the given material, which should not exceed 3% according to ISO standard 6876.[3,8,9] Water sorption and solubility were measured using ISO 4049; 2009.[10] Although this standard does not include fluid uptake investigation but it was calculated by modification of the standard following the work of Grech et. al in 2013.[10]

In the current study, percentage solubility of all the experimental groups were below 3% as specified in the international standard. Biodentine exhibited more solubility in comparison to the other two test materials in deionized water which is in accord with many previous studies.[2,8,11,12] In a study by Alzraikat, H., N.A. Taha, and A. Salameh, the reported 24-hour solubility of Biodentine was significantly higher than MTA (0.0347g, 2.91% vs. 0.0199g, 1.75% respectively)[2]. Another study by Kouzmanova, Y. and I. Dimitrova, reported greater 28-days solubility in distilled water by Biodentine than MTA Angelus which is in accord with this study findings[13]. This enhanced solubility of Biodentine can be attributed to higher dissolution of ions.[3] However, the finding of this study is in contrast to studies by Gandolfi, M.G., et al., and Torres, F.F.E., et al., where Biodentine showed solubility lower than MTA Angelus.[4,5]

Water sorption is defined as sum of water adsorbed on the surface and absorbed into the body of the material. In the current study, statistically significant water sorption and solubility were observed between all experimental groups. However, biodentine showed highest water sorption followed by MTA and least by Acroseal as is evident in Figure 2. Contrasting findings were reported by a study by Al-Sherbiny, I.M., et al., where MTA exhibited greater water sorption and solubility than Biodentine.[14]

Fluid uptake for MTA increased from day 1 to day 28, which may be due the grainy texture and porous structure as established by Alzraikat et al. in 2016.[2] Whereas, it remains relatively unchanged for Acroseal over one-month period. But for Biodentine fluid uptake gradually decreases as the time passes indicating that Biodentine becomes more stable over time as shown in Figure 1. In a study by Torres, F.F.E., et al., MTA exhibited higher fluid uptake than Biodentine which is in accordance with this study.[5]

The study was conducted in a small setup with minimum resources, so it has a number of limitations including small sample size and minimum number of tests conducted. But these findings pave a new path for further testing and evaluation of these root end filling materials based on their difference in compositions.

Conclusion

Solubility and water sorption of bio-dentine is higher than MTA and Acroseal but they still remain within the standardized limits. Whereas its fluid uptake over 28 days decreases in comparison to other two test materials indicating its stability. This property in addition to its easy handling properties makes it a better root-end filling material in comparison to other tested materials.

Ethical Approval: The Ethical review committee of Army Medical College Rawalpindi approved the study vide letter No. ERC/ID/101.

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Authors' Contribution:

MM: Study designing, Data collection, data analysis and interpretation, manuscript writing & Proof reading

HG: Idea conception, study designing, data collection, data analysis and interpretation, manuscript writing

MK: Data collection, manuscript writing & proof reading

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