A Comparative Study of pH Changes, Releasing Elements and solubility of Bioceramic Sealers.

Abstract:

Introduction: Bioceramic technology has been introduced recently in endodontics to benefit from the hydroxyapatiteformation during the setting reaction in the presence of tissue fluid and establish a chemical bond at the dentin interface.

Aim: To comparethe physiochemical properties (pH changes, releasing elements and solubility)of different newerBioceramicsealer (Ceraseal) with mineral trioxide aggregate (MTA-Fillapex) sealers.

Material and Method: Samples were prepared by using three different injectable sealer placed in 20 ring moulds, 10 of each sealer. Each sample was prepared with an internal diameter of 10 mm and a height of 3mm.The samples were digitally weighed to register the mass of each specimen before and after immersion in distilled water. The pH value was measured by a digital pH meter, releasing elements measured in atomic absorption spectrophotometer, and solubility measured in digital weighed machine.After 1, 7, 14 and 28 days, pH changes, released elements and solubility was the data obtained calculated and statistically analysed.

Results: Ceraseal Sealer exhibited high alkaline pH over time and also showed significantly higher solubility ($P < 0.05$).

Conclusion: Bioceramic sealer shows more alkalinity and calcium ion release compared to the MTA sealer. The alkalinity nature of the bioceramic sealer, corresponds to the increase in solubility. So bioceramic sealer are more alkaline, calcium releasing and soluble.

Key-words: Ceraseal; MTA Fillapex; pH; solubility

Introduction:

One of themost important objectivesof endodontic filling is to acquire a proper seal after eliminating microorganisms from the root canal system, which prevents the re-entry of bacteria, fluid as well as the growth of the microorganisms[1,2]. To obtain appropriate seal, gutta-percha (GP) and root canal sealers are essential materials. An ideal sealer should offer specific properties [3,4] and amongst them, insolubility is one of the most desirable physical properties,[5] because it may have a great impact on the success rate of root canal treatment [6]. The dissolution of sealer may cause gaps along with the dentinsealergutta-percha interface and that might offer a pathway for bacteria and their by-products into periapical tissues. The pH change of sealers may be related to antibacterial outcome and deposition of mineralized tissue, thus playing an important role in the healing process. So, pH changes, calcium ion release, and solubility of sealer playimportant role in the success of root canal treatment.

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The alkaline pH is closely related to the antimicrobial effect and eradicates the residual microbes that survive on the dentinal wall[7,8]. The alkaline nature of the sealer also provides deposition of mineralized tissue, thus playing an important role in the healing process.

The calcium ion Ca+2 based sealers provide an antimicrobial effect, which inhibits the growth of residual microbes, and this enhances healing of periapical pathosis.[9,10]

The low solubility of a root canal sealer has been introduced in 2000 as a requirement in the ANSI/ADA specification No. 57 and in 2001 as a requirement in the International Standards Organization 6876 standard for root canal sealing materials. According to those standards, the solubility of a sealer should not exceed 3% mass fraction after immersion in water for 24 hours[11]. Different types of endodontic sealers are available like zinc-oxide eugenol (ZnOE), resin-based, calcium hydroxide containing, root canal sealers.But some disadvantages of sealer likeZinc oxide eugenol-basedsealer has producedcytotoxicity due to the presences of eugenol. MTA and bioceramic based sealerwere introduced, which has attractive physical, chemical, mechanical, andbiological properties.

Bioceramic and Mineral Trioxide Aggregate (MTA) root canal sealers are neweradvancements in sealers to provide benefit from the formation of hydroxyapatite during the setting reaction in the presence of tissue fluid and establish a chemical bond at the dentin interface.

MTA sealer was firstly introduced in Endodontics by Lee et al. [12] in 1993 as retro filling material and for the repair of perforations[13]. It has been indicated for pulp capping, apexification, and repairing of root resorption because of its favorable physical, chemical and biological properties[14,15] A new MTA-based endodontic sealer was launched in the Brazilian dental market – namely MTA Fillapex® (Angelus Soluções Odontológicas, Londrina, PR, Brazil) whose composition, according to the manufacturer, is basically MTA, silicate resin, natural resin and bismuth oxide[16].Viapiana et al.[17] found root canal sealers like MTA Fillapex to be highly soluble and Vitti et al.[18] reported the solubility of MTA Fillapex to be <3 %consistent with ISO 6876/2001

The bioceramic-based sealers containing calcium silicate and calcium phosphate attracted considerable attention because of their physical and biological properties such as their alkaline pH, chemical stability within the biological environment, and lack of shrinkage [19] Bioceramic materials contain calcium phosphate which enhances the setting properties of bioceramics and results in chemical composition and crystalline structure similar to tooth and bone apatite materials, thereby improving sealer-to-root dentin bonding. Ceraseal (Meta Biomed Co., Cheongju, Korea) is a newly launched premixed endodontic sealer containing calcium silicates, zirconium oxide, and thickening agent[20]. No study and clinical research find on pH changes, Ca+2 release and solubility of MTA fillapex and Ceraseal so that is the reason to perform this study .

The purpose of this in vitro study was to evaluate pH changes and calcium ion release and solubility of MTAFillapex and Cerasealsealers at different time of interval. The null hypothesis of this study is that the bioceramic root canal sealer Ceraseal will have high pH, releasing element and low solubility levels compared to the MTA Fillapex.

Methodology:

For sample preparation:

A Polyethylene tube was cut into 20 smaller tubes of equal sizes with each tube 10 mm in diameter and 3mm in height; the size of each sample was measured with a digital vernier caliper. The tubes were preweighed by a digital weighing balance machine (SEPTECHprecision balance digital machine) to select similar weighing tubes. The tubes were divided into three experimental groups in which they were filled with the material under study.

Injectable Ceraseal and MTA fillapex were used to prepare the samples. Twentey tubes were divided into two equal groups with each group consisting of ten tubes (n=10). According to manufacturer instructions injectable sealers were carried into polyethylene tubes. They were left in an incubator at 37 ˚C, 100% humidity until completely set. Each disc was tied with impermeable nylon thread so that sample will not damage while weighed. The initial weight (W0) of each disc was measured using a digital weighing machine.(SEPTECH precision balance digital machine). Each and every sample was immersed in different containers of 20 ml of deionised water for 1,7,14 and 28 days at 100% humidity at a constant temperature of 37˚ C.

Following are the two groups (n=10 each)

Group 1: MTA fillapex

Group 2: Ceraseal

For pH:

The pH of the storage solution at each immersion time period (1, 7, 14 and 28 days) was analysed using a pH meter (ENVTEC 0.001pH meter). Every time new solution was prepared for the next immersion time.

For solubility:

After each immersion timeperoid 1,7,14 and 28 days the discs were removed from the solution and then dried with blotting paper. The discs were left undisturbed for 24 hours for complete drying and then again weighed in SEPTECH precision balance digital machine. (Wf1, Wf7, Wf14 and Wf28). After measuring the Solubility, discs were placed in new solution for the next immersion time period.

The amount of solubility (%) was calculated by using the following equation:

Solubility (%) = Wo-Wf/Wf × 100

For calcium ion release:

The solutions after each immersion time period (1,7,14 and 28 day) were analysed for the amount of calcium ions (Ca^{+2}) released using anAtomic Absorption Spectrophotometer (AAS) (AA-6300 Shimadzu). At the 28th day, the solutions were analysed for other degradable elements, including phosphorus, aluminium, iron and manganese using the Atomic Absorption Spectrometer (AAS)(AA-6300 Shimadzu).

Statistical analysis:

According to the normality test, the data of pH changes, released elements andsolubility were statistically analyzed by the One-Way ANOVA test followed by Post-Hoc Tukey tests. The significance level p considered (<0.05), to compare thematerials under study.

Result:

pH changes:

The mean values of pH of both tested materials at different immersion time period are described in following line chart (Figure 1). An ANOVA test revealed a statistically significant difference among the groups (P<0.05). Group 1 exhibited significantly higher alkalinity on first day. On 14th and 28th day no significant difference was found between thegroups. Following Table 1 shows mean value of pH changes.The bioceramic sealer exhibited high alkaline pH over a period of time with the maximum valueon the 7th day.

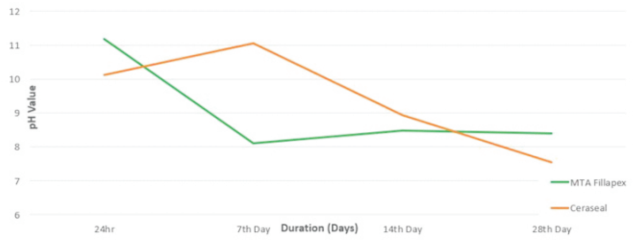

Figure 1. Plotlines representing the mean values of pH change over time for tested sealers

Duration	MTA fillapex	Ceraseal
1st day	11.17	10.13
7th	8.10	11.05
14th	8.48	8.94
28th	8.39	7.55

Table 1 Mean value of pH changes

Releasing element:

The Following bar chart (Figure2) describes the mean values of Ca^{+2} at different observation time period and cumulative amounts of degradable elements released after 28 days (mg/L)(Table 2). On initial day group 2 showed more calcium ion release compared to group 1. After 28th day Ca of group 1 and group 2 showed 13.66 and 11.63 respectively. Regarding Ca^{+2} released, there was a statistically significant difference among the two groups (P<0.05).

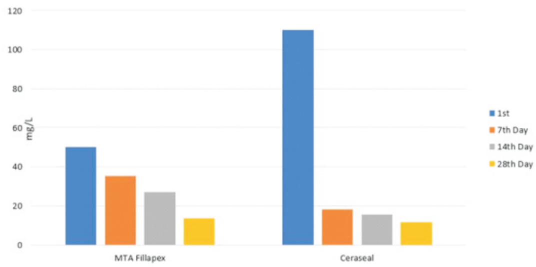

Figure 2 Mean values of released calcium ions (mg/L) over time for the tested sealers.

Duration	MTA fillapex	Ceraseal
1st day	50.25	109.93
7th	35.18	18.20
14th	27.06	15.35
28th	13.66	11.63

Table 2 mean value of releasing element (mg/l)

Solubility:

The following bar chart shows (Figure 3) the comparison between the mean values of the solubility of both the tested materials at different immersion time periods. The group 1 and group 2 exhibited an ongoing increase in solubility over a period of time exceeding the acceptable limit of ADA. A significant high value was obtained for group 2 followed by group 1 (Table 3).

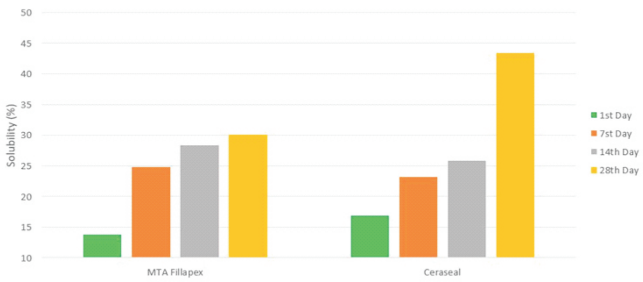

Figure 3 Mean values of solubility (%) over time among all tested sealers

Duration	MTA fillapex	Ceraseal
1st day	50.25	109.93
7th	35.18	18.20
14th	27.06	15.35
28th	13.66	11.63

Table 3 Mean value of solubility (%)of sealers

Discussion :

Choosing an endodontic sealer for clinical use is a decision that contributes to the long-term success of root canal treatment. The main functions of root canal sealers are (i) sealing off the voids, patent accessory canals, and multiple foramina, (ii) forming a bond between the core of the filling material and the root canal wall, and (iii) acting as a lubricant while facilitating the placement of the filling core and entombing any remaining bacteria. The zinc oxide- eugenol-based root canal sealers have been long employed for root canal filling and quite satisfactory chemical outcomes have been observed but the biocompatibility of these materials is impaired by the presence of eugenol which has cytotoxic properties. The calcium hydroxide-based root canal sealers were therefore introduced to meet the ideal requirements of the biological and physiochemical properties.

Nowadays, bioceramic sealer have been introduced which contain calcium silicate and zirconium oxide so the properties of the sealer improved. The bioceramic sealer hastwo main effects. Firstly, their biocompatibility prevents rejection by the surrounding tissues andsecondly, bioceramic materials contain calcium phosphate which enhances the setting properties of bioceramics and results in chemical composition and crystalline structure similar to tooth and bone apatite. The main advantage of a premixed calcium-silicate root canal sealer consists of having a homogeneous mixture, irrespective of the alteration of powder/liquid ratio[21,22]. Physiochemical properties of root canal sealer have to provide an apical seal avoiding the leakage of irritants and pathogens from the root canal system into the peri-radicular tissues(visa-versa). Indeed, any alteration of the powder/liquid ratio during the mixing procedure may cause alteration of the compressive strength, the solubility [23]and the bond strength of endodontic cement to dentin [24]. This is eliminated with premixedthat is user-friendly and less time-consuming in nature[25].

According to ADA specification No. 57, which explains the physiological properties of sealers.The current study has assessed the pH changes, Ca^{+2} ion released and solubility (%) of two Bioceramic sealers(Ceraseal) and MTA sealers[26].

MTA Fillapex (group 1) is an endodontic sealer based on MTA. It is more stable than calcium hydroxide, provides constant release of calcium ions and maintains a pH that elicits an antibacterial effect. The tissue recovery and the reduction inthe inflammatory response are optimized by the use of MTA and salicylate resin[27].

Ceraseal (group 2)is formed of calcium silicate which absorbs the moisture present in the root canal resulting in the formation of calcium aluminate hydrate gel (CAH) and calcium silicate hydrate gel (CSH). Moreover, Cerasealexhibits an excellent sealing ability resulting in a perfectly and fully sealed hermetic root canal thus, preventing any bacterial progression[28].

A high pH value is important for root canal sealers because the release of calcium ions not only stimulates hard tissue formation but also triggers antibacterial activity. Anantimicrobial activity is promoted by high pHresults from the release of hydroxyl ions because, the alkaline pH induces the loss of the integrity of the cytoplasmic membrane of the cells, promoting the inactivation of the enzymes involved in the cellular metabolism, and damaging bacterial DNA[29]. In this study the pH of both sealers increases on the first day of immersion; group 1 and group 2 show mean value 11.17 and 10.13 respectively whereas on seventh-day group 1, 8.10 and group2 shows 11.55. On the 14thday and 28thday, pH mean values remain constant.

In 2013, Silva et al.[30] suggested that due to the high alkalinity of MTAFillapex, it had a strong capacity to release hydroxyl ions, thereby causing a high Ca^{+2} ion release[31]. The alkaline media could activate the alkaline phosphatase, neutralize the acid, inactivate the osteoclasts, prevent further bone destruction and allow tissue repair with concomitant apatite formation. The first clinical use of calcium hydroxide as a root canal–filling material was probably by Rhoner in 1940 (1.6). The two most important reasons for using calcium hydroxide as a root-filling material are stimulation of the periapical tissues in order to maintain health or promote healing and secondly for its antimicrobial effects.Calcium hydroxide denatures proteins found in the root canal and makes them less toxic. Calcium hydroxide activates the calcium-dependent adenosine triphosphatase reaction associated with hard tissue formation [32]. Calcium hydroxide diffuses through dentinal tubules and may communicate with the periodontal ligament space to arrest external root resorption and accelerate healing [33]. According to Parirock and Torabinejad, the presence of calcium may favor an alkaline pH, which leads to a biochemical effect that accelerates the healing process. Group 2 (109.3) showed higher Ca^{+2} release compared to group 1 (50.25) on the first day. As the number of immersion days increases the solubility of the sealer decreases, at the last 28th-day solubility of group 1was 13.66 andgroup 2 was 11.63.

The solubility of root canal sealers should not exceed 3% mass after 24 h in water according to ISO specification 6876:2012.In the present study, after the initial immersion day solubility of group 1 was 13.8 and group 2 was 16.9. Group 2 showed the highest level of solubility compared togroup 1. On the 28th day solubility of group 1 was30.4 and group 2 was 43.43.The endodontic sealers must have low solubility because the leaching of their components can generate undesirable biological effects on the surrounding tissues[34]. High solubility may be responsible for creating gaps between the sealer materials and the root canal dentin, causing loss of the sealing ability and a pathway for microorganisms. The endodontic filling materials are designed to be kept inside root canals to promote an impermeable sealing in the long term and to eliminate any communication route between the oral cavity and periapical tissues. This premise was reinforced by Faria-Júnior et al.[35] who concluded that the neutral pH and low solubility of the sealer would eliminate its antibacterial activity. Thus the null hypothesis of this study is rejected as Ceraseal (group 2) showed more solubility and the pH values decreased with passage of time, Ca^{+2} ion also decreases with time as compared with MTA fillapex. MTA fillapex (group 1) showing pH values, Ca^{+2} ions and solubility which remain constant. Furthermore, the clinical studies required to investigate the properties of the sealer clinically.

Conclusion :

The scientific evidence on the obturation of the root canal system with bioceramic root canal sealers is limited, therefore, its use in clinical practice must be considered with great caution, taking into consideration the physicochemical properties, biocompatibility, biomineralization as well as retractability of each material. In this current study, the Bioceramic sealer Ceraseal shows more solubility than MTA Fillapex. The MTA fillapex shows pH, Ca^{+2} ion and solubility in which values are maintained and showing better results compared to ceraseal. Since the clinical data of obturation with bioceramic root canal sealers is less, the selection of materials should be made based on the available scientific evidence, individual cases, material availability and operator's preference.

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