Original research article

Physicochemical properties of two commercially available bioceramic sealers: An in vitro study

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Abstract

A great number of bioceramic sealers commercially available nowadays show a great diversity in their biological, chemical, and physical properties. Such differences can also significantly affect clinical performance, and consequently, new commercially available products must be carefully investigated to assess quality and check differences with existing products. The current in vitro study investigated five physicochemical properties (setting time, solubility, pH analysis, radiopacity, and film thickness) of two commercially available bioceramic sealers. The null hypothesis was that the composition of the two products was the same, the manufacturing site was the same and consequently, they should exhibit the same properties when subjected to in vitro tests. Tests were conducted based on the International Standard Organization (ISO) 6876/2012 recommendations. For each test 10 samples for each product were evaluated. All data were recorded and analyzed statistically by ANOVA and Tukey's test at the 5% significance level. For all tests, no statistically significant differences were found between the two groups. Based on the findings of this study, we may conclude that there is a great similarity in chemical composition and manufacturing or, more likely, the two products, which are made by the same manufacturers, are identical.

Keywords: Root canal, sealer, obturation, bioceramic

Introduction

Successful endodontic therapy is accomplished by proper shaping and cleaning procedures followed by three-dimensional, hermetic sealing of the root canals with appropriate root canal filling materials (1). Endodontic filling materials are placed permanently in the root canal to ensure long-term endodontic success, aiming at avoiding leakage of bacterial invasion by blocking the pathways of communication between the root canal system and its surrounding tissues (2). For many decades gutta-percha has been the golden standard and the ideal core material of the filled root canals, being inert, biocompatible, non-resorbable and thermoplastic (1). However, due to the inability of gutta-percha to adhere to dentine, a root canal sealer was also needed to improve sealing, even if sealer was considered the weak part of the obturation, being more toxic and resorbable. Ideally, a sealer should also exhibit alkalinity and bioactive properties to improve tissue healing and remineralization. For many years several root canal sealers have been commercialized with a great diversity in their biological, chemical, and physical properties, but none of them possessed all the ideal properties described by Grossman (3).

In recent times endodontics has made huge progress, especially with advancements in nickel-titanium file design and metallurgy (4-6). New operative techniques have been developed, focusing on making chemomechanical preparation more rapid and efficient, and ideally increasing quality by providing more predictable cleaning and shaping results (7-10). As a consequence, there has been a similar interest in improving the simplicity and quality of root canal obturation, with a continuous request for alternative sealers that are more biocompatible, more bioactive, and more capable of bonding to the root canal wall when compared to traditional resin-based or eugenol-based sealers (11-16). Therefore, the criteria for the ideal material for use in endodontic obturations must be comprehensive

and include the following characteristics: non-toxic, insoluble in tissue fluids, dimensionally stable, antibacterial, hard tissue conductive, biocompatible, radiopaque and easy to handle (3). New bioceramic sealers were developed to fulfill these requests, and in the last decade, these products have become more widely investigated and clinically used worldwide, even if they are generally more expensive than traditional sealers (15). However, the various bioceramic sealers commercially available nowadays show a great diversity in their biological, chemical, and physical properties: most of them are mainly related to their chemical composition, but also to the manufacturing process. Such differences can also significantly affect clinical performance, and consequently, new commercially available products must be carefully investigated to assess quality and check differences with existing products (11-13). It has been shown that the safety data sheets and manufacturer details of currently available bioceramic sealers were imprecise. Moreover, little detail on composition was provided by manufacturers (11).

The current in vitro study investigated five physicochemical properties (setting time, solubility, pH analysis, radiopacity and film thickness) of two commercially available bioceramic sealers: a new one and the one which has been most extensively evaluated in the last decade. The null hypothesis was that they should exhibit the same properties when subjected to in vitro tests, because manufacturing site and process are the same.

Materials and methods

For each group and for each test 2 samples were taken from a different package. Overall 5 packages of Endosequence BC sealer (BUSA/Brasseler USA. Savannah, GA) and 5 packages of Edge Bioceramic (EdgeEndo, Albuquerque, New Mexico, USA) were used. Sample size was determined by Power Analysis and for each test it was calculated based on a power of 80% and a 0.05 alpha type error (G*Power, Heinrich-Heine-Universität Düsseldorf, Düsseldorf, Germany). For each group, the indicated sample size was 10. All samples were analyzed before the expiration dates established by the manufacturers.

Setting time.

Setting time was evaluated based on the International Standard Organization (ISO) 6876/2012 recommendations, with a slight change. Gypsum molds to measure the setting time of the bioceramic sealers instead of the recommended stainless steel ones were selected. This choice was performed to avoid any risk of incomplete setting related to the mold because gypsum contains water and bioceramic sealers require moisture for setting, whilst stainless steel does not contain water. For each group, each of the five packages was used to fill two molds. Overall, ten specimens for each group were tested and stored in an incubator (ICT-120 Permax, Treviglio Italy) with 95% humidity at 37 °C. The setting time was measured using a Gilmore needle, with a total weight of 100 g and a flat end of diameter 2.0 mm, starting 1 h before the setting time specified by the manufacturer, and was repeated every 5 minutes. The needle was carefully placed vertically against the sealer and if an indentation was visible it was raised to clean, and this process was repeated until the needle no longer indented the sealer surface**.** The final setting time (min) was recorded and all data were analyzed statistically by ANOVA and Tukey's test at the 5% significance level.

Solubility test

The test was carried out in accordance with the ISO 6876/2012 recommendations using 10 samples for each group. Similarly, to the setting time tests gypsum molds with an internal diameter of 10 mm and height of 2 mm were fabricated, then filled with the endodontic sealers and stored in an incubator (ICT-120 Permax, Treviglio Italy) with 95% humidity at 37 °C for 24 hours for setting. The disc-shaped specimens were then removed from the mould, and weighed (initial mass = W1) by using an analytical balance (Mettler-Toledo, model AE1633, Novate Milanese, Italy)) with an accuracy of 0.001 g. Then, each specimen was hung using a nylon thread in a closed plastic fbootle containing 10 mL of saline solution and stored again in the incubator for two weeks with the same humidity and temperature described above. The specimens were then removed from the incubator, dried with absorbent paper, and placed in a dehumidifying chamber for 24 hours. After completing this procedure the specimens were weighed again (final mass = W2) and the material loss was calculated as a percentage of the difference in weight by the following formula: (w1-w2) /w1 x100. All data were analyzed statistically by ANOVA and Tukey's test at the 5% significance level.

PH analysis

The test was carried out using 10 specimens for each group. Each sample of root canal sealer was inserted into polytetrafluoroethylene tubes to obtain discs with a 5 mm diameter and 2 mm thickness. After the sealer setting in an incubator (ICT-120 Permax, Treviglio Italy) with 95% humidity at 37 °C. for 24 hours, each specimen was immersed into a closed flask containing 10 mL of distilled water at an initial pH of 7 and a temperature of 25 °C. Then the specimens were stored again in an incubator at 37 °C and 95% relative humidity for 7 (?). The calibration of the pH meter (Jen-way 3510 bench pH meter, UK) was performed with a standard solution at pH 4.0 and 7.0 at a constant temperature of 25 °C. The pH of the solution was measured after 24 hours. All data were recorded and analyzed statistically by ANOVA and Tukey's test at the 5% significance level.

Radiopacity

The test was performed following the recommendation described in ISO 6876/2012. For each group, each of the five packages was used to make two disc-shaped specimens (10 x 1 mm). Overall, ten specimens for each group were tested. After setting the materials in an incubator (ICT-120 Permax, Treviglio Italy) with 95% humidity at 37 °C. for 24 hours, radiographs were taken using periapical digital film (Digora, Dexis) including a graduated aluminum step wedge varying from 2 to 16 mm in thickness for comparison. The dental X-ray unit (Kodak) was set at 70 kVp, 10 mA, and a distance of 50 cm. The radiopacity of sealers was compared to that of the aluminum step wedge using VIXWIN-2000 software. All data (mmAl) were recorded and analyzed statistically

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by ANOVA and Tukey's test at the 5% significance level.
Film thickness
Tests were performed according to the ISO 6876/2012 recommendations. Initially, the total thickness of two pieces of flat glass plates (5 mm in thickness, 200×10 mm surface area) placed over one another was measured (TH1) using an electronic digital caliper (ELE Digital Caliper, Atessa, Italy). Each glass was also weighted with an analytical balance (Adam Equipment 4-digit precision weighing balance, UK) with an accuracy of 0.001 g. For each group, 10 samples of sealer were collected from the syringe transferred immediately onto the lower glass plate and covered by the upper glass plate. The weight glasses with sealer were measured again to ensure a similar amount of sealer (0,05 ml) in all specimens, and specimens were put into an incubator with a 150 N load weight vertically applied for 3 minutes on the upper glass plate. After 10 minutes the total thickness (TH2)of the plates, including the sealer, was measured again using the same digital caliper, and the amount of the film thickness (µm)was obtained by subtracting the initial measurement of the two glasses from final total thickness of the glass plates (TH2-TH1) All data were recorded and analyzed statistically by ANOVA and Tukey's test at the 5% significance level.

Results

Results are shown in Table 1. Mean values, standard deviation, and significance are shown for each of the following five in vitro tests: setting time, solubility, pH analysis, radiopacity, and film thickness. Mean values for the setting time (min) were 90,25 and 86,77, respectively for BC Endosequence and Edge Bioceramic. Mean values for the solubility test (%) were 3,4 and 3,6, respectively for BC Endosequence and Edge Bioceramic. Mean values for PH were 10,8 and 10,9 respectively for BC Endosequence and Edge Bioceramic. Mean values for the radiopacity (mmAl) were 3,25 and 3,6, respectively for BC Endosequence and Edge Bioceramic. Mean values for the film thickness (µm) were 48 and 45, respectively for BC Endosequence and Edge Bioceramic For all tests, no statistically significant differences were found between the two groups.

Discussion

Bioceramic sealers have shown more favorable properties when compared to traditional sealers, documented by many in vitro and in vivo studies (17-24). These studies have also shown significant differences amongst currently available bioceramic sealers (17-24). In a recent review, authors showed that in the last years, endodontists faced a rapid increase in materials identified as bioceramics on the market, but these materials have different chemistries, and some of the constituents are not declared (11). The authors conclude that this may affect the clinical performance of these materials and highlighted the importance of using reputable materials that have been adequately researched both in vitro and in clinical practice (11).
Results from the present study showed that in all the in vitro physical and chemical tests performed there was no statistically significant difference between the two groups. This could be explained by a great similarity in manufacturing or, more likely, the two products, which are made by the same manufacturers, are identical. EndoSequence BC sealer could also be considered as a control because of its reputation as a gold standard for studies on bioceramic sealers. It is one of the very first commercial products and has been subjected in recent years to many in vitro and vivo researches, which provided excellent results, and currently it is the bioceramic sealer most widely investigated in dental literature and most validated for clinical use (16-24). On the contrary no studies are available concerning Edge Bioceramic sealer.
Setting time was similar between the two products, which are both available in premixed syringes inside which inorganic components of the sealers (calcium phosphate, silicates) are premixed with water-free thickening vehicles. This formulation enables the sealer to be delivered in the form of a premixed paste, and water/moisture is required for the setting reaction. This is a very user-friendly way of applying the sealer into canals and also avoids poor performance related to incorrect mixing of the sealers. In a clinical environment, the setting reaction can be also catalyzed by the presence of moisture in dentinal tubules and be completed in a few hours, even if it could be longer in particularly dried canals. Therefore, the amount of moisture present in the dentinal tubules of the canal walls and, consequently, the setting time could also be affected by differences in the absorption with paper points. On the contrary, too much water left inside canals could decrease the microhardness of the material.
In the present study, the solubility of both products was slightly higher than the minimum ISO standard value, with no statistically significant differences between the two groups. Such high values could be attributed to sealers' hydrophilic nanosized particles, which have a positive effect on film thickness and flowability but could also increase their surface area and allow more liquid molecules to come into contact with the sealer and consequently increase its solubility. These results are in accordance with previous studies on bioceramic sealers, but they also conflict with other studies that demonstrated that the solubility of Endosequence BC sealer was consistent with ISO standards (23). Such different findings could be related to variations in the test methods (24); more precisely variations in the procedure used to dry samples may result in significant differences. Another important factor is the time between mixing the sealer and immersion in the storage solution. In some studies, mixed sealers were immersed after 150% or 300%

Table1	SettingT time (min)	Solubility test (%)	PH analysis	Radiopacity (mmAl)	Film thickness (µm)
BC Endosequence	90,25 +/- 12,5	3,4 +/-0,4	10,8 +/- 0,25	3,25. +/- 0,25	48 +/- 0,1
Edge Bioceramic	86,75 +/- 11,5	3,6 +/-0,5	10,9 +/- 0,25	3,6 +/- 0,5	45 +/- 0,2
	NS	NS	NS	NS	NS

of their setting time compared with 300% whilst others used a set duration of 24 h after mixing. It is noticeable that the reported solubility values were inversely related to the duration between mixing and immersion (24) Both sealers showed a strong alkaline pH after 24 hours, with no significant differences between the two groups. This is a positive finding because it can determine a prolonged setting time, thus allowing adequate time for obturation and a long-lasting antibacterial effect that eliminates the residual bacteria that survived after chemo-mechanical canal preparation. An alkaline pH may also contribute to enhanced osteogenic potential by activating alkaline phosphatase, neutralizing lactic acid from osteoclast, and allowing tissue repair with the formation of hydroxyapatite. An alkaline PH is also related to improved biocompatibility. PH data from the present study are similar to values obtained in other studies which analyzed EndoSequence BC sealer (25). Radiopacity of root canal obturation materials is a fundamental physical property that allows radiographic evaluation of the root canal filling. In clinical practice, the quality of the filling is checked by radiographs immediately after obturation. Single-cone hydraulic condensation with bioceramics allows to verify if overfilling or underfilling is present, and if needed clinicians can modify the obturation by adding more sealer of placing the gutta-percha cone more apically or coronally, due to an adequate setting time of the sealer. An ideal material should be clearly visible inside and outside the canal,also to detect overextension (a moderate one is usually tolerated by tissue due to the biocompatibility of the bioceramic sealer) and its possible resorption over time. In the present study, the radiopacity test was performed following ISO 6876/2001 recommendations, using aluminum as the control material. Both products showed higher values compared to the ISO minimal requirements (radiopacity equivalent to 3 mm thick), but there were no statistically significant differences between the two groups. For both products film thickness was in accordance with ISO minimal requirements and in accordance with other published studies for EndoSequence BC sealer (25)

Conclusions

The present in vitro study investigated five physicochemical properties (setting time, solubility, pH analysis, radiopacity, and film thickness) of two commercially available bioceramic sealers: BC EndoSequence and Edge Bioceramic. Based on the findings of this study, which showed no statistically significant differences in all tests between the two products we may conclude that there is a great similarity in manufacturing or, more likely, the two products, which are made by the same manufacturers, are identical.

Declaration of competing interest

The authors have no conflict of interest relevant to this article.

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