

**ORIGINAL ARTICLE**

Year : 2015 | Volume : 18 | Issue : 3 | Page : 227--231

Physical-mechanical properties of Bis-EMA based root canal sealer with different fillers addition

Marcela Oliveira de Souza, Vicente Castelo Branco Leitune, Priscila Veit Bohn, Susana Maria Werner Samuel, Fabricio Mezzomo Collares

Department of Conservative Dentistry, School of Dentistry, Universidade Federal do Rio Grande do Sul, Porto Alegre - RS, Brazil

Correspondence Address:

Fabricio Mezzomo Collares

Ramiro Barcelosst, 2492 - 4th floor, Dental Materials Laboratory, School of Dentistry, Universidade Federal do Rio Grande do Sul, Porto Alegre, Rio Grande do Sul, Brasil-90035-003 Brazil

Abstract

Aim: To evaluate influence of three different filler particles on an experimental Bisphenol A ethoxylated dimethacrylate (Bis-EMA) based root filling material. **Materials and Methods:** Resin-based endodontic sealers were produced using Bis-EMA, camphorquinone, ethyl 4-dimethylaminobenzoate (EDAB), N, N-dihydroxyethyl-p-toluidine (DHEPT), butylated hydroxytoluene (BHT), and benzoyl peroxide. The experimental groups were formulated adding 10, 20, 30, 40, and 50% of calcium tungstate (CaWO_4), ytterbium trifluoride (YbF_3), and tantalum oxide (Ta_2O_5). Flow, thickness, and radiopacity tests were conducted in accordance with ISO 6876. Sorption and solubility (SL) tests were conducted in accordance with ISO 4049, pH was measured with a pH meter, and degree of conversion (DC) was evaluated with Fourier transform infrared spectroscopy (FTIR). For radiopacity, two-way analysis of variance (ANOVA) and Tukey «SQ»s multiple comparison test was performed. For DC analysis, one-way ANOVA and Tukey «SQ»s multiple comparison test was performed. All statistical analyses were performed with a significance level of 5%. **Results:** All groups showed lower flow with increased filler concentration. All groups showed film thickness values lower than 50 μm , as ISO recommends, except CaWO_4 50% group (76.7 μm). pH values varied from 5.95 (± 0.07) in YbF_3 40% group to 6.90 (± 0.07) in Ta_2O_5 40% group. In the radiopacity test, YbF_3 30%, Ta_2O_5 40%, and Ta_2O_5 50% groups showed no statistical significant difference to 3mmAl. Ta_2O_5 and YbF_3 groups in 10, 20, and 30% concentrations presented sorption and SL values as ISO recommendation. Addition of Ta_2O_5 and CaWO_4 decreased DC after 14 days. YbF_3 addition showed no difference in DC from control group. **Conclusion:** YbF_3 filler addition promoted higher properties compared to CaWO_4 and Ta_2O_5 on Bis-EMA based root canal sealer.

How to cite this article:

de Souza MO, Branco Leitune VC, Bohn PV, Werner Samuel SM, Collares FM. Physical-mechanical properties of Bis-EMA based root canal sealer with different fillers addition. J Conserv Dent 2015;18:227-231

How to cite this URL:de Souza MO, Branco Leitune VC, Bohn PV, Werner Samuel SM, Collares FM. Physical-mechanical properties of Bis-EMA based root canal sealer with different fillers addition. J Conserv Dent [serial online] 2015 [cited 2016 Mar 17];18:227-231
Available from: <http://www.jcd.org.in/text.asp?2015/18/3/227/157259>**Full Text****INTRODUCTION**

The filling material choice is of paramount importance to longevity of endodontic treatment, complementing the canal preparation. [1] Methacrylate-based materials are a reliable matrix for filler incorporation and recently have been used for root canal sealers. [2],[3]

Dimethacrylates like bisphenol Aethoxylated (Bis-EMA) appears to be a further trend in formulation of methacrylate-based dental materials, with a stiff phenyl ring core; however without the two pendant hydroxyl groups which are responsible for the water affinity and high viscosity like Bis-GMA based methacrylate matrix. [4],[5]

The aim of this study to evaluate influence of three different filler particles on an experimental Bis-EMA based root filling materials.

Materials and Methods**Formulation**

Experimental dual cured sealers were obtained from a base resin of Bisphenol A ethoxylated dimethacrylate (Bis-EMA). Camphorquinone, ethyl 4-dimethylaminobenzoate (EDAB), N, N-dihydroxyethyl-p-toluidine (DHEPT), and benzoyl peroxide (Esstech Inc, Essington, Pennsylvania, USA) were used for polymerization system. Butylated hydroxytoluene (BHT) was used to inhibit autopolymerization in 0.01 wt%. Calcium tungstate (CaWO_4), ytterbium trifluoride (YbF_3), and tantalum oxide (Ta_2O_5) at 10, 20, 30, 40, and 50% in weight, were added as filler to the base resin. The particle size distribution was assessed using a laser diffraction particle size analyzer (CILAS 1180, Orleans, France). The mean size of filler particles used was 17.79, 14.37, and 6.26 μm , respectively.

Fifteen endodontic experimental resin paste/paste sealers with different concentrations and one with no filler addition were developed. To perform sealer photoactivation, a light-emitting diode activation unit (Radii Cal, SDI, Bayswater, Victoria, Australia) with irradiation value of 1,200 mW/cm² was used for all tests.

Flow

The flow test was conducted according to ISO 6876:2001. A total of 0.05 (± 0.005) mL of each experimental sealer was placed on a glass plate (40 \times 40 \times 5 mm). At 180 \pm 5s after mixing was started, another plate with a mass of 20 \pm 2 g and a load of 100g was placed on top of the material. Ten minutes after mixing had been started, the load was removed, and the major and minor diameters of the compressed material were measured using a digital caliper. If both measurements were within 1 mm of each other, the test was conducted three times for each group (n = 3), and the mean value was recorded.

Film Thickness

The film thickness was evaluated according to ISO 6876:2001. Two glass plates that measured 5 mm in thickness and 40 mm in length were placed together, and their combined thickness was measured. The center of one of the plates was covered with 0.05 (± 0.005) mL of experimental sealer, and a second plate was placed on top of the material. At 180 \pm 5 s after the start of mixing, a load of 150 N was applied on top of the glass plate. Ten minutes after the start of mixing, the thickness of the two glass plates and the interposed sealer film

was measured. The mean value of three measurements for each sealer was recorded as the film thickness of the material.

Water Sorption (WS) and Solubility (SL)

The sorption and SL tests were based on ISO 4049:2009, with the exception of the dimensions of the samples. Five samples with 6.0 mm (± 0.5 mm) diameter and 1.0 mm (± 0.2 mm) of thickness for each group ($n = 5$) were made using a Teflon matrix. The amounts of WS and SL for each sample were calculated. [6]

pH

The pH determination was realized with a digital pH meter (PH21, Hanna Instruments, São Paulo, SP, Brazil). The solution employed to measure the pH was the resulting water from the sorption and SL test where each sample was stored for 7 days at 37 °C. The electrode was immersed 4 cm into the solution and a temperature probe was used so that the pH was automatically compensated with temperature variations.

Radiopacity

Radiographic images were obtained by a digital system with phosphorous plates (VistaScan, Dürr Dental GmbH & Co. KG, Bietigheim-Bissingen, Germany), the specimens were 6.0 mm (± 0.5 mm) in diameter and 1.0 mm (± 0.2 mm) in thickness ($n = 5$). The X-ray source (DabiAtlante model Spectro 70x) was operated with a tungsten anode at 70 kV and 8 mA, exposure time of 0.4s and a focus-film distance of 400 mm. For each film, one specimen from each group with the same concentration was used, resulting in five films per concentration. For all images, an aluminium step-wedge was exposed simultaneously with the specimens. The thickness of the aluminium step-wedge ranged from 0.5 to 9.0 mm. The images were analyzed using Photoshop software (Adobe Systems Incorporated, San Jose, CA, USA). The means and standard deviations of the grey levels (pixel density) of the aluminium step-wedge and the specimens were measured in a standardized area of 2.0 mm². [7]

Degree of Conversion (DC)

The DC of the sealers was evaluated using Fourier transform infrared spectroscopy (FTIR) with a Vetrex 70 (Bruker Optics, Ettlingen, Germany), equipped with an attenuated total reflectance device composed of a horizontal diamond crystal with a mirror angle of 45 degrees. A support was coupled to fix the light-curing unit and standardize the distance between the fiber tip and sample at 5 mm. Opus software (Bruker Optics, Ettlingen, Germany) was used with Blackman-Harris 3-Term apodization in a range of 4,000-400 cm⁻¹ and resolution of 4 cm⁻¹. With this setup, one spectrum was obtained prior to photocuring and one immediately after photocuring. The samples (3 μ l) were directly dispensed onto the diamond crystal and light-activated for 120s (polymer immediate) and after 14 days storage at 37 °C (polymer at 14 days). The test was repeated three times on each group ($n = 3$).

Statistical Analysis

Flow, thickness, and radiopacity tests were conducted in accordance with ISO 6876. Sorption and SL tests were conducted in accordance with ISO 4049, pH was measured with a pHmeter and DC was evaluated with FTIR. For radiopacity, two-way analysis of variance (ANOVA) and Tukey's multiple comparison test was performed. For DC analysis, one-way ANOVA and Tukey's multiple comparison test. All statistical analyses were performed with a significance level of 5%.

RESULTS

[Table 1] shows flow, film thickness, and pH results. Flow of experimental sealers ranged from 10 ± 0.1 to 30 ± 0.2 mm. As the filler concentration increased, the flow decreased for all groups ($P < 0.05$). The groups with addition of Ta 2 O 5 showed higher flow compared to groups with YbF 3 and CaWO 4 ($P < 0.05$). Film thickness ranged from 10 to 76 μ m. The groups showed a film thickness up to 50 μ m, except for the group CaWO 4 50% (76.7 μ m). The pH of experimental sealers ranged from 5.95 ± 0.07 (YbF 3 40%) to 6.90 ± 0.07 (Ta 2 O 5 40%) ($P < 0.05$).{Table 1}

The groups with CaWO 4 addition showed the highest values of WS and SL. The groups with YbF 3 and Ta 2 O 5 addition at 10, 20, and 30% showed values of WS and SL in accordance with ISO 4049 standards [Table 1].

The results of radiopacity are shown in [Figure 1]. The groups Ta 2 O 5 40 and 50% and YbF 3 30% showed no statistically significant difference to 3 mm Al, as recommended by ISO 6876 for root canal sealer. The DC of experimental sealers is shown in [Figure 2]. The groups with addition of Ta 2 O 5 and CaWO 4 presented lower values of DC than the control and YbF 3 groups ($P < 0.05$).{Figure 1}{Figure 2}

DISCUSSION

Root canal sealers with increased physical-mechanical properties are related to high quality root filling. [8] This study evaluated experimental root canal sealers with Bis-EMA as resinous matrix and showed reliable properties for root filling.

The incorporation of inorganic fillers is used to increased related properties of materials, such as radiopacity and viscosity. The presence of fillers allow the root canal sealers to be identified in radiographic images and increase the accuracy of diagnostics. [7],[9] Groups with 40 and 50% of Ta 2 O 5 and 30% of YbF 3 presented values in accordance to ISO 6876 for root canal sealers. On the other hand, groups with CaWO 4 obtained lower values than 3 mm aluminum even at high concentrations. The low radiopacity of CaWO 4 is one reason to this filler be used as adjuvant in commercial sealers. As the concentration of fillers increased, the viscosity of root canal sealers increased, decreasing the flow. [10] Groups with Ta 2 O 5 at 10, 20, and 30%; YbF 3 at 10%; and CaWO 4 at 10% were in accordance of the flow requirements of ISO. Despite ISO recommendations of flow; higher than 20 mm, groups presenting values higher than clinical demanded, since the root canal sealers used in the present study are resin-based material. [11] ISO 6876 standardization describes requirements for water-based sealers. All groups presented film thickness lower than 50 μ m as recommended by ISO 6876, except for group with CaWO 4. The determinant influence to radiopacity, flow, and film thickness was carried by inorganic fillers, and Bis-EMA acted as resin matrix that allowed the incorporation of high amount of particles.

Resin-based root canal materials present a radical polymerization and low DC. Formed polymer is more prone to degradation leading to leached compounds that could achieve periapical region. The presence of uncured material in the apex could promote tissue inflammatory reaction and could lead to clinical failure of endodontic treatment. In the present study, the groups with addition of YbF 3 showed a higher DC compared to the groups with CaWO 4 and Ta 2 O 5. The DC of photo activated resinous materials increases with the level of radiated energy. [12] The initial generation of free radicals of methacrylate monomers is directly related to the radiation absorbed by the material, such as photo activated sealers. The DC is proportional to the square root of the light intensity absorbed by photo initiator system and the concentration of photo initiator. [13] Therefore, amore opaque material promotes a physical barrier to light transmission, generating lower DC of sealer. In a visual analysis of formulated sealers, cements with YbF3 were more translucent than cements with CaWO 4 and Ta 2 O 5. The YbF 3 has refractive index (1.51) similar to the matrix resin used (1.7), which could not interfere significantly with the absorption of light. Refractive index value of filler should be ideally closer to resin matrix, producing an homogeneous material, benefiting the curing process.

Considering that a high pH could alter tissue inflammatory conditions in the apical region resulting in a bactericidal effect and a faster repair process, the alkalinity of the sealer is important. [14] The pH of experimental groups ranged from 5.95 to 6.9 favoring biocompatibility. WS and SL are related to degradation of materials. [15],[16] The infiltration of water into the resin matrix may cause plasticization of the formed polymer network, leading to the polymer degradation. [6],[15] The groups with YbF 3 and Ta 2 O 5 addition at 10, 20, and 30% showed values of WS and SL in accordance with ISO 4049 standards. CaWO 4 addition groups in all concentrations showed higher WS values, which could increase degradation and decrease mechanical properties. [17],[18] High SL of a sealer must be avoided in order to prevent release of monomers that could lead to tissue cytotoxicity. [19],[20] Furthermore, dissolution of sealer could lead to gaps between the filling material and the root canal walls. [20],[21] The properties of endodontic sealers are mainly determined by the type and proportions of the main components. In this study, Bis-EMA enabled the addition of filler particles maintaining acceptable properties of sealers.

CONCLUSION

YbF 3 filler addition promoted higher properties compared to CaWO 4 and Ta 2 O 5 on Bis-EMA based root canal sealer.

References

- 1 Bodrumlu E, Tunga U. Coronal sealing ability of a new root canal filling material. J Can Dent Assoc 2007;73:623.
- 2 Marin-Bauza GA, Rached-Junior FJ, Souza-Gabriel AE, Sousa-Neto MD, Miranda CE, Silva-Sousa YT. Physicochemical properties of methacrylate resin-based root canal sealers. J Endod 2010;36:1531-6.
- 3 Leitune VC, Takimi A, Collares FM, Santos PD, Provenzi C, Bergmann CP, *et al.* Niobium pentoxide as a new filler for methacrylate-based root canal sealers. Int Endod J 2013;46:205-10.
- 4 Ogliari FA, Ely C, Zanchi CH, Fortes CB, Samuel SM, Demarco FF, *et al.* Influence of chain extender length of aromatic dimethacrylates on polymer network development. Dent Mater 2008;24:165-71.
- 5 Andreani L, Silva LL, Witt MA, Meier MM, Joussef AC, Soldi V. Development of dental resinous systems composed of bisphenol a ethoxylated dimethacrylate and three novel methacrylate monomers: Synthesis and characterization. J Appl Polym Sci 2013;128:725-34.
- 6 Collares FM, Ogliari FA, Zanchi CH, Petzhold CL, Piva E, Samuel SM. Influence of 2-hydroxyethyl methacrylate concentration on polymer network of adhesive resin. J Adhes Dent 2011;13:125-9.
- 7 Collares FM, Ogliari FA, Lima GS, Fontanella VR, Piva E, Samuel SM. Ytterbium trifluoride as a radiopaque agent for dental cements. Int Endod J 2010;43:792-7.
- 8 Orstavik D, Eriksen HM, Beyer-Olsen EM. Adhesive properties and leakage of root canal sealers *in vitro*. Int Endod J 1983;16:59-63.
- 9 Beyer-Olsen EM, Orstavik D. Radiopacity of root canal sealers. Oral Surg Oral Med Oral Pathol 1981;51:320-8.
- 10 Weisman MI. A study of the flow rate of ten root canal sealers. Oral Surg Oral Med Oral Pathol 1970;29:255-61.
- 11 Baldi JV, Bernardes RA, Duarte MA, Ordinola-Zapata R, Cavenago BC, Moraes JC, *et al.* Variability of physicochemical properties of an epoxy resin sealer taken from different parts of the same tube. Int Endod J 2012;45:915-20.
- 12 Calheiros FC, Daronch M, Rueggeberg FA, Braga RR. Influence of irradiant energy on degree of conversion, polymerization rate and shrinkage stress in an experimental resin composite system. Dent Mater 2008;24:1164-8.
- 13 Watts DC. Reaction kinetics and mechanics in photo-polymerised networks. Dent Mater 2005;21:27-35.
- 14 Seux D, Couble ML, Hartmann DJ, Gauthier JP, Magloire H. Odontoblast-like cytodifferentiation of human dental pulp cells *in vitro* in the presence of a calcium hydroxide-containing cement. Arch Oral Biol 1991;36: 117-28.
- 15 Mortier E, Gerdolle DA, Jacquot B, Panighi MM. Importance of water sorption and solubility studies for couple bonding agent-resin-based filling material. Oper Dent 2004;29:669-76.
- 16 Huang C, Kei LH, Wei SH, Cheung GS, Tay FR, Pashley DH. The influence of hygroscopic expansion of resin-based restorative materials on artificial gap reduction. J Adhes Dent 2002;4:61-71.
- 17 Ferracane JL. Hygroscopic and hydrolytic effects in dental polymer networks. Dent Mater 2006;22:211-22.
- 18 Kalachandra S, De Porter CD, Grubbs HJ, Mc Grath JE. Polymeric materials for composite matrices in biological environments. Polymer 1993;34:778-82.
- 19 Kaplan AE, Goldberg F, Artaza LP, de Silvio A, Macchi RL. Disintegration of endodontic cements in water. J Endod 1997;23:439-41.
- 20 McMichen FR, Pearson G, Rahbaran S, Gulabivala K. A comparative study of selected physical properties of five root-canal sealers. Int Endod J 2003;36:629-35.
- 21 Schafer E, Zandbigliari T. Solubility of root-canal sealers in water and artificial saliva. Int Endod J 2003;36:660-9.

Thursday, March 17, 2016

[Site Map](#) | [Home](#) | [Contact Us](#) | [Feedback](#) | [Copyright and Disclaimer](#)