Polímeros: Ciência e Tecnologia
ISSN: 0104-1428
abpol@abpol.org.br
Associação Brasileira de Polímeros
Brasil

Schwartzer, Eduardo; Genari, Bruna; Castelo Branco Leitune, Vicente; Mezzomo Collares, Fabrício; Werner Samuel, Susana Maria
Bismuth Subcarbonate as Filler Particle for an Epoxy-based Root Canal Sealer
Polímeros: Ciência e Tecnologia, vol. 23, núm. 6, 2013, pp. 743-747
Associação Brasileira de Polímeros
São Paulo, Brasil

http://dx.doi.org/10.4322/polimeros.2014.010

Bismuth Subcarbonate as Filler Particle for an Epoxy-based Root Canal Sealer

Eduardo Schwartzer, Bruna Genari
Dental Materials Laboratory, Department of Conservative Dentistry, School of Dentistry, Federal University of Rio Grande do Sul, Brazil

Vicente Castelo Branco Leitune, Fabrício Mezzomo Collares, Susana Maria Werner Samuel
Dental Materials Laboratory, Department of Conservative Dentistry, School of Dentistry, Federal University of Rio Grande do Sul, Brazil

Abstract: Introduction: The aim of this study was to evaluate the addition of bismuth subcarbonate with different concentrations regarding the rheological properties of an experimental epoxy-based root canal sealer. Materials and Methods: Endodontic sealers were prepared with epoxy resin-based sealer with bismuth subcarbonate additions of 20%, 40%, 60%, 80%, 100%, and 120%. Flow, film thickness, working time, setting time, dimensional change, sorption, solubility, and cytotoxicity were studied according to the ISO standards. Data were statistically analyzed by one-way ANOVA, and Tukey multiple comparisons were used, with a significance level of 5%. Results: The flow, working time, water sorption, and solubility significantly decreased and the film thickness and dimensional change increased with higher filler particle addition. There were no statistically significant differences for setting time and cytotoxicity between the filler particle proportions. Conclusion: Experimental resin-based sealer with bismuth subcarbonate addition up to 40% can be an alternative for root canal sealer.

Keywords: *Bismuth subcarbonate, epoxy-based endodontic sealer, filler particle.*

Introduction

The endodontic obturation fill the root canal space maintaining satisfactory biological and physicochemical properties of decontamination and sealing[1]. An appropriate sealer with dimensional stability avoid fluid percolation between the canal compartment and outside the canal[1,2]. The most frequently used endodontic sealers are resin-based sealers, zinc oxide-eugenol sealers, calcium hydroxide-containing sealers, glass ionomer-based sealers, and mineral trioxide aggregate (MTA)-based sealers[2]. These materials present in their compositions filler particles that provide better mechanical properties, rheological adjustment, and radiopacity[1,3-6]. Bismuth compounds are often used in root canal sealers to provide radiopacity[7,8] and have been used in Grossman's sealer[9]. However, resin-based endodontic sealers have been presented better properties than water-based sealers[10]. Therefore, experimental resin-based sealers with bismuth subcarbonate should be tested.

The aim of this study was to evaluate the addition of bismuth subcarbonate at different concentrations to an epoxy-based root canal sealer at selected properties. The null hypothesis tested in this study is that there is no difference between different concentrations of bismuth subcarbonate added in the resin-based endodontic sealer in terms of mechanical properties.

Materials and Methods

Experimental sealer formulation

Experimental endodontic sealers were prepared with epoxy resin-based sealer, bisphenol-A and epichlohydrin, (Fiberglass, Porto Alegre, Brazil) at 2:1 (base:catalyst) with bismuth subcarbonate additions of 20%, 40%, 60%, 80%, 100%, and 120% in weight. The filler particle size of the bismuth salt was obtained by laser diffraction with equipment CILAS 1800. The mean diameter particle was 18.72 μm and particle size distribution is shown in Figure 1. Colloidal silica (particle diameter of 7nm) was added at 0.05% to adapt the viscosity of these sealers according to ISO 6876[11].

Flow

The flow test was conducted in accordance with ISO 6876[11]. A total of 0.05mL of each experimental sealer was placed on a glass plate (40×40×5mm) with a graduated 1.5mL syringe. Another plate with a mass of 20±2g and a load of 100g was applied on top of the material. Ten minutes after the start of mixing, the load was removed, and the major and minor diameters of the compressed material were measured using a digital caliper (Digimess, Sao Paulo, Brazil). The test was done in triplicate and the mean value was recorded.

Film thickness

This evaluation was made according to ISO 6876[11]. Two glass plates (5×10mm) were placed together and their combined thickness was measured. A mount of 0.5mL of experimental sealer was placed at the center of one of the plates, and a second plate was placed on top of the material. At 180±5s after the start of mixing, a

***Corresponding author: Fabrício Mezzomo Collares**, Dental Materials Laboratory, School of Dentistry, Federal University of Rio Grande do Sul – UFRGS, Rua Ramiro Barcelos, 2492, Rio Branco, CEP 90035-003, Porto Alegre, RS, Brazil, e-mail: fabricio.collares@ufrgs.br*

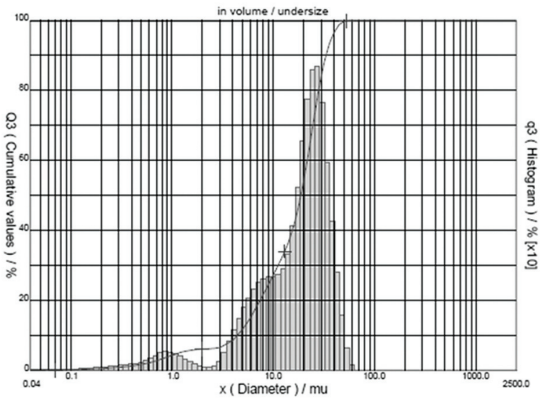

Figure 1. The particle size distribution of bismuth subcarbonate.

load of 150±3N was applied vertically onto the top glass plate. Ten minutes after the start of mixing, the thickness of the two glass plates and the interposed sealer film was measured using a digital caliper. The film thickness was recorded by the difference between the thickness of the two glass plates with and without sealer. The mean value of three measurements was recorded as the film thickness of the material.

Working time

The test to measure the necessary time to mix the components to obtain the sealer with appropriate properties was based on ISO 6876[11]. This test had the same sequence as the flow test, but it was repeated in major intervals of time between manipulation and setting time. The working time was recorded when the diameter of the specimen were 10% less than the diameter of the immediate manipulated sealer. The test was repeated three times and the mean values were recorded.

Setting time

The setting time was recorded according to ISO 6876[11]. Rings measuring 10mm in diameter and 1mm in height were filled with the material. These specimens were maintained under controlled temperature and humidity conditions, 37±1°C and 95%, respectively. Measurements were conducted using Gilmore needles, weighing 100±0.5g and having a flat end of 2.0±0.1mm diameter. The needle was lowered vertically onto the horizontal surface of each sample such that it touched every 5 minutes. The setting time was recorded one time for each specimen when the needle did not produce any visible indent on the sealer surface.

Dimensional change following setting

The dimensional change was measured based on ISO 6876[11]. Three cylindrical matrices were filling with the sealer. These specimens were positioned between two glass plates. Five minutes after the start of mixing, the specimens were placed in desiccators at 37°C and 95% relative humidity and held for a period three times the setting time. The specimens were removed from the matrices and the thickness was measured. A micrometer (Aus-JENA, Jena, German) with a capability of measuring 0.001mm was used for measurement purposes. The difference between before and after storage values was calculated. Measurements were made three times and the mean value of these measurements was recorded as the dimensional change of the material.

Water sorption and solubility

Water sorption and solubility were determined based on the ISO 4049[12]. Five sealers disks were produced in a silicone matrix (10.0mm diameter, 1mm thick). Specimens were placed in a desiccator at 37°C for 22 hours and in a desiccator at 23°C for 2 hours. The disks were repeatedly weighed in an analytical balance (Shimadzu Corp., Tokyo, Japan) until a constant mass (m_1) was obtained (i.e., until the mass loss of each specimen wasn't more than 0.1mg in any 24h period). Time, in days, to achieve a constant mass ranged from 27 to 28 days. Diameter and thickness of each specimen were measured with a digital caliper to calculate the volume (V) of each disk (in mm). Thereafter, the specimens were stored in sealed glasses vials with 10mL of distilled water at 37°C for 7 days. After seven days, the disks were weighed after being washed under running water and gently wiped with an absorbent paper to obtain a mass (m_2) and then returned to the desiccator. Next, the specimens were weighed until a constant mass (m_3) was obtained (as described above). The test was realized one time for each specimen. Water sorption (WS) and solubility (SL) in micrograms per cubic millimeter were calculated using the following formulae:

$$WS = m_2 - m_3 / V \quad (1)$$

$$SL = m_1 - m_3 / V. \quad (2)$$

Cytotoxicity

According to ISO 10993-5[13], the cell viability was analyzed using mononuclear cells obtained from human peripheral blood. These cells were routinely maintained in Dulbecco's modified Eagle's medium (DMEM) with HEPES - HDMEM, with 10% fetal calf serum. The cells were maintained with endodontic sealers incubated for 48 hours at 37°C and 5% CO_2. The controls consisted of cells incubated without endodontic sealer. The rate of viable cells was quantified by testing (3-4,5-dimethylthiazol-2-yl)-2,5-diphenol tetrazolium bromide) MTT assay after 24 and 48 hours in contact with the endodontic sealer. The test was realized one time for each specimen.

Statistical analysis

Data normality distribution was analyzed by Kolmogorof-Smirnov and passed. The results for all tests presented normality distribution. Therefore, the test used was one-way ANOVA, and Tukey multiple comparisons were used with a significance level of 5% for all tests.

Results

The results of rheological properties of experimental sealer are shown in Tables 1 and 2.

Flow

The flow of the sealer significantly decreased with the increase of filler particle concentration ($p<0.05$), ranging from 23.5 to 16.77mm for 20% to 120%, respectively. The results of flow measurements are shown in Table 1.

Film thickness

The film thickness results are shown in Table 1. The film thickness values significantly increased with higher filler particle addition, ranging from 43.3 to 50mm for 20% to 120%, respectively. The values obtained with 100% and 120%, 93.3 and 113.3, respectively, were significantly higher than other groups ($p<0.05$).

Working time

The working time significantly decreased as filler particle concentration increased ($p<0.05$). These vales varied from 64.33 to 44.67 for 20% to 120%, respectively. The means and standard deviations of the working time are shown in Table 1.

Setting time

The setting time measurements are shown in Table 1. There was no statistically significant difference ($p>0.05$) among groups.

Dimensional change following setting

The results of dimensional change are shown in Table 2. The dimensional change obtained with 100% filler particle proportion was significantly higher than other groups, ranging from –0.2 to –0.57 for 20% to 100%, respectively. Specimens with higher bismuth subcarbonate concentrations presented higher dimensions ($p<0.05$).

Water sorption and solubility

Water sorption and solubility results are shown in Table 2. Sorption significantly increased with higher filler particle addition. These values varied from 34.85 to 109.85μg/mm^3 for 20% to 120%, respectively. Solubility with 120% filler particle proportion presented significant differences compared to 40%, 60%, and 80% ($p<0.05$).

Cytotoxicity

There was no statistically significant difference ($p>0.05$) between the filler particle proportions regarding cytotoxicity. The results are shown in Figure 2.

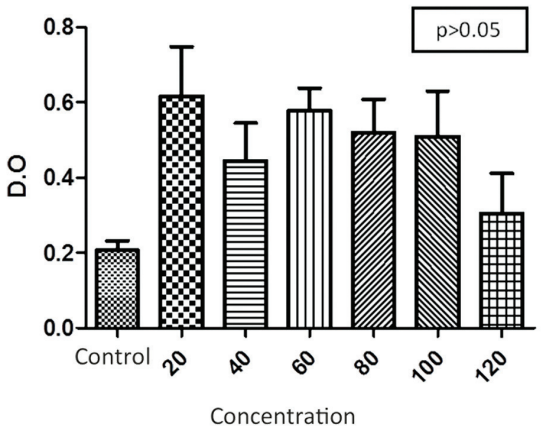

Figure 2. Cytotoxicity of the sealer with bismuth subcarbonate in different proportions.

Table 1. Flow, film thickness, working time and setting time of the sealers with bismuth subcarbonate in different proportions.

	Flow (mm)	Film thickness (mm)	Working time (min)	Setting time (h)
20%	23.25 (0.37)^a	43.3 (11.5)^a	64.33 (01.15)^a	07:05 (00:55)^a
40%	22.15 (1.07)^a	50 (10)^{a,b}	56.67 (02.08)^b	06:57 (00:47)^a
60%	18.52 (0.13)^b	63.3 (5.8)^{b,c}	51.33 (01.53)^{b,c}	06:45 (00:30)^a
80%	17,19 (0.37)^{b,c}	70 (10)^{b,c}	51.67 (3.21)^{b,c}	06:31 (00:23)^a
100%	17.08 (0.26)^c	93.3 (11.5)^c	48.33 (03.06)^{c,d}	06:12 (00:25)^a
120%	16.77 (0.13)^c	113.3 (15.3)^c	44.67 (02.08)^d	05:59 (00:18)^a

Different letters in same column represent statistically significant differences ($p < 0.05$).

Table 2. Dimensional change, sorption, solubility and radiopacity of the sealers with bismuth subcarbonate in different proportions.

	Dimensional change (%)	Sorption (μg/mm^3)	Solubility (μg/mm^3)
20%	–0.2 (0.02)^a	34.85 (3.48)^d	13.78 (2.15)^{a,b}
40%	–0.3 (0.08)^{a,b}	37.66 (4.29)^{c,d}	11.02 (1.8)^b
60%	–0.39 (0.06)^b	45.14 (11.62)^c	11.23 (4.87)^b
80%	–0.46 (0.04)^b	56.04 (3.12)^c	11.42 (2.58)^b
100%	–0.57 (0.06)^c	88.76 (4.7)^b	15.67 (5.24)^{a,b}
120%	*	109.85 (14.04)^a	21.53 (6.2)^a

*It was not possible to evaluate because of disintegration of the specimens. Different letters in same column represent statistically significant differences ($p<0.05$).

Discussion

Requirements as facility to manipulate, stability in the oral environment, radiopacity, biocompatibility, antimicrobial action, low shrink or expansion during polymerization and facility to remove for post placement or retreatment are desirable for an endodontic sealer[14]. The properties of the sealers are modified by the filler particle according to the present study. Therefore, the null hypothesis was rejected.

The experimental sealers with 60% to 120% presented lower flow than ISO requirements[11]. However, this standard is for water-based sealers. The flow of commercial available resin-based sealers also presented values lower than the standard[15]. In addition, the flow of the sealer cannot be so high due to a possible periapical extrusion that can have a negative impact on the healing of the periapical region[16].

Film thickness is an important characteristic of an endodontic sealer, because it is regarded as the ability of the material to fill the smallest voids and enter the dentinal tubules[17]. According to ISO 6876[11], the film thickness should be up to 50μm. The groups with 60% to 120% concentrations showed values higher than the standard. However, Sealapex and AHPlus, both resin-based sealers, also present values around 50μm[18].

The clinical utility demands that the time must be long enough to allow placement and adjustment for root filling[19]. Acording to ISO 6876[11], working time should be less than 30 min and setting time from 30 min to 72 h. In the present study, all groups presented setting time in accordance with ISO requirements, but higher working time than the standard. The values of the present study were similar to Apexit Plus[4].

ISO 6876[11] requires that a sealer should not shrink more than 1% nor swell more than 0.1%. In the present study, the experimental endodontic sealers presented dimensional expansion higher than 0.1%. Commercial sealers presented similar dimensional change[20]. In addition to dimensional change, water sorption could cause higher frictional forces and lower mechanical properties[21,22]. After separation of polymer chains, the unreacted monomers are released through porosities and voids, leading to degradation of the sealer[22-24]. ISO 4049[12] specifies that the water sorption of the resin-based materials should not be higher than 40μg/mm^3 and the water solubility should be up to 7.5μg/mm^3. In the present study, the sealer with bismuth subcarbonate in proportions of 20% and 40% presented water sorption in accordance with the requirement. Higher water sorption and solubility could result in leaching compounds of the resin-based materials, which could lead to cytotoxic effects on periapical tissue[20]. Materials in contact with human tissues must present low cytotocity[25]. The present study presented no difference between the groups regarding the cell viability.

In the search for an ideal endodontic sealer, the epoxy-based sealer with bismuth subcarbonate can be an alternative for clinical use. The microbiological, bond strength, and radiopacity of the experimental sealer could be tested in future studies.

Conclusion

Within the methodology and the results of this study, it is possible to conclude that the experimental resin-based sealer with bismuth subcarbonate addition up to 40% can be an alternative for root canal sealer

Acknowledgements

The authors are grateful for the scholarship from CAPES (Coordenação de Aperfeiçoamento de Pessoal de Nível Superior) for E.S and V.C.B.L.

References

1. Schilder, H. – J. Endod., **32**, p.281 (2006). PMid:16554195. http://dx.doi.org/10.1016/j.joen.2006.02.007
2. Bernardes, R. A.; De Amorim Campelo, A.; Silva Junior, D. S.; Pereira, L. O.; Duarte, M. A.; Moraes, I. G. & Bramante, C. M. - Oral Surg. Oral Med. Oral Pathol. Oral Radiol. Endod., **109**, p.e47 (2010). PMid:20123369. http://dx.doi.org/10.1016/j.tripleo.2009.08.038
3. Silva, E. J. N. L.; Rosa, T. P.; Herrera, D. R.; Jacinto, R. C.; Gomes, B. P. F. A. & Zaia, A. A. - J. Endod., **39**, p.247 (2013).
4. Marín-Bauza, G. A.; Silva-Sousa, Y. T. C.; Cunha, S. A.; Rached-Junior, F. J. A.; Bonetti-Filho, I.; Sousa-Neto, M. D. & Miranda, C. E. S. - J. Appl. Oral Sci., **20**, p.455 (2012). http://dx.doi.org/10.1590/S1678-77572012000400011
5. Da Conceição, B. M.; Visconte, L. L. Y. & Furtado, C. R. G. - Polímeros, **22**, p.352 (2012).
6. Collares, F. M.; Leitune, V. C.; Rostirolla, F. V.; Trommer, R. M.; Bergmann, C. P. & Samuel, S. M. - Int Endod J., **45**, p.63 (2012). PMid:21899568. http://dx.doi.org/10.1111/j.1365-2591.2011.01948.x
7. Ghatak, S. K.; Dey, D.; Sen, S. & Sen, K. - Analyst, **138**, p.2308 (2013). PMid:23446647. http://dx.doi.org/10.1039/c3an36842d
8. Collares, F. M.; Ogliari, F. A.; Lima, G. S.; Fontanella, V. R.; Piva, E. & Samuel, S. M. - Int Endod J., **43**, p.792 (2010). PMid:20579134. http://dx.doi.org/10.1111/j.1365-2591.2010.01746.x
9. Savioli, R. N.; Pecora, J. D.; Mian, H. & Ito, I. Y. - Braz. Oral Res., **20**, p.127 (2006). PMid:16878205. http://dx.doi.org/10.1590/S1806-83242006000200007
10. Lee, K. W.; Williams, M. C.; Camps, J. J. & Pashley, D. H. - J. Endod., **28**, p.684 (2002). PMid:12398164. http://dx.doi.org/10.1097/00004770-200210000-00002
11. International Organization for Standardization. - "ISO 6876: Dental Root Canal Sealing Materials", 2nd ed., ISO (2001).
12. International Organization for Standardization. - "ISO 4049: Dentistry – Polymer-based restorative materials", 4th ed., ISO (2009).
13. International Organization for Standardization. - "ISO 10993-5: Biological evaluation of medical devices Part 5: Tests for in vitro cytotoxicity", 3rd ed., ISO.
14. Schwartz, R. S. - J. Endod., **32**, p.1125 (2006). PMid:17174666. http://dx.doi.org/10.1016/j.joen.2006.08.003

15. Nawal, R. R.; Parande, M.; Sehgal, R.; Naik, A. & Rao, N.R. - Int. Endod. J., **44**, p.307 (2011). PMid:21219358. http://dx.doi.org/10.1111/j.1365-2591.2010.01829.x
16. Sjogren, U.; Hagglund, B.; Sundqvist, G. & Wing, K. - J. Endod., **16**, p.498 (1990). http://dx.doi.org/10.1016/S0099-2399(07)80180-4
17. Gambarini, G.; Testarelli, L.; Pongione, G.; Gerosa, R. & Gagliani, M. - Aust. Endod. J., **32**, p.31 (2006). PMid:16603043. http://dx.doi.org/10.1111/j.1747-4477.2006.00005.x
18. Hosoya, M.; Kurayama, H.; Iino, F. & Arai, T. - Int. Endod. J., **37**, p.178 (2004). PMid:15009407. http://dx.doi.org/10.1111/j.0143-2885.2004.00781.x
19. McMichen, F. R.; Pearson, G.; Rahbaran, S.; & Gulabivala, K. - Int. Endod. J., **36**, p.629 (2003). PMid:12950578. http://dx.doi.org/10.1046/j.1365-2591.2003.00701.x
20. Ferracane, J. L. - Dent. Mater., **22**, p.211 (2006). PMid:16087225. http://dx.doi.org/10.1016/j.dental.2005.05.005
21. Versiani, M. A.; Carvalho-Junior, J. R.; Padilha, M. I. A. F.; Lacey, S.; Pascon, E. A. & Souza-Neto, M. D. - Int. Endod. J., **39**, p.464 (2006). PMid:16674741. http://dx.doi.org/10.1111/j.1365-2591.2006.01105.x
22. Garcia, F. G.; Leyva, M. E. & De Queiroz, A. A. A. - Polímeros, **21**, p.253 (2011).
23. Brazel, C. S. & Peppas, N. A. - Biomaterials, **20**, p.721 (1999). http://dx.doi.org/10.1016/S0142-9612(98)00215-4
24. Juchem, C. O.; Leitune, V. C. B; Collares, F. M.; Samuel, S. M. W. - Polímeros, **21**, p.103 (2011). http://dx.doi.org/10.1590/S0104-14282011005000031
25. Loushine, B. A.; Bryan, T. E.; Looney, S. W.; Gillen, B. M.; Loushine, R. J.; Weller, R. N.; Pashley, D. H. & Tay, F. R. - J. Endod., **37**, p.673 (2011). http://dx.doi.org/10.1016/j.joen.2011.01.003

Received: 04/15/13
Revised: 08/19/13
Accepted: 10/21/13