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Sealing ability, water sorption, solubility and toothbrushing abrasion resistance of temporary filling materials

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Abstract

Pieper CM, Zanchi CH, Rodrigues-Junior SA, Moraes RR, Pontes LS, Bueno M. Sealing ability, water sorption, solubility and toothbrushing abrasion resistance of temporary filling materials. *International Endodontic Journal*, **42**, 893–899, 2009.

Aim To evaluate marginal seal, water sorption, solubility and loss of mass after brushing of several temporary filling materials.

Methodology For marginal seal, Class I cavities, including endodontic access preparations, were made in human molar teeth and restored using one or other of several temporary filling materials ($n = 10$): zinc oxide/calcium sulphate-based cement (Cavit, 3M,ESPE, St. Paul, MN, USA), zinc oxide/eugenol cement (IRM, Dentsply Caulk, Milford, DE, USA), glass ionomer cement (Vidrion R, SSWhite, Rio de Janeiro, RJ, Brazil) or a dimethacrylate-based filling (Bioplic, Biodinâmica, Londrina, PR, Brazil). Dye penetration was assessed after thermocycling and immersion in 0.5% basic fuchsine solution. For water sorption, solubility and loss of mass analyses, disc-shaped specimens were made. Water sorption and solubility were evaluated by mass alteration after storage in distilled water for 7 days ($n = 7$). Loss of mass was calculated based on the difference of mass after abrasion with a toothbrush ($n = 5$), and surfaces were analysed by SEM. Data of water sorption, solubility and loss of mass were submitted to ANOVA and Tukey's test, and marginal sealing data to Kruskal–Wallis test ($P < 0.05$).

Results Statistically significant differences were observed for marginal sealing ($P < 0.0001$), water sorption ($P < 0.01$), solubility ($P < 0.01$) and loss of mass ($P < 0.05$). Bioplic had the best marginal seal. Cavit had the greatest water sorption and solubility. Vidrion R and Bioplic had the lowest solubility. Loss of mass after brushing was higher for Cavit, followed by Bioplic, IRM and Vidrion R. Cavit and Vidrion R were worn aggressively by brushing.

Conclusions The resin-based temporary filling Bioplic produced the best marginal seal, and was associated with the lowest water sorption, solubility and loss of mass.

Keywords: loss of mass, marginal sealing, temporary filling, toothbrushing abrasion, water sorption/solubility.

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Introduction

The outcome of root canal treatment depends, amongst other factors, upon the sealing capacity of temporary restorations that prevents bacterial infiltration and recontamination of the root canal system (Torabinejad *et al.* 1990, Ray & Trope 1995, Hommez *et al.* 2002). Besides avoiding bacterial percolation, temporary fillings may help to protect weakened coronal tooth tissue from fractures when they have adhesive properties (Soares & Goldberg 2002). Conversely, fillings that expand during or after setting, due to hygroscopic expansion, may cause cusp deflection or fractures (Laustsen *et al.* 2005).

Characteristically, restorative materials undergo degradation in contact with water, such as leaching of

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components that may weaken their structure (Ferracane 2006). In addition, the oral environment is inhospitable for restorative materials, with extremes of thermal and mechanical challenges. The mechanical action of toothbrushing might also abrade the materials (Moraes *et al.* 2008).

Several temporary filling materials with different microstructures, compositions and setting mechanisms are available commercially. Cavit (3M; ESPE, St. Paul, MN, USA) is a premanipulated eugenol-free material that sets in contact with moisture, but has given conflicting marginal sealing results (Naoum & Chandler 2002). Bioplic (Biodinâmica, Londrina, PR, Brazil) is a resin-based material that sets upon light-curing, characteristically presenting volumetric shrinkage during polymerization. This contraction, however, is usually followed by expansion due to water sorption (Deveaux *et al.* 1992), although whether this hygroscopic expansion is sufficient to adequately seal the cavity is still unknown. Conventional glass–ionomer cements (GIC) are considered suitable materials for restorations for several reasons: they form a hard material upon setting, present relatively little or no exothermic reaction or shrinkage during setting, have no free monomer in the set matrix, and adhere to tooth structure (Culbertson 2001). Based on its adhesion potential, it could be expected that the marginal sealing produced by GICs is good. Naoum & Chandler (2002) have concluded that GIC is a satisfactory endodontic temporary filling, even in the long-term. IRM (Dentsply Caulk, Milford, DE, USA), a zinc oxide-eugenol (ZOE) based cement, has been associated with antibacterial activity (Naoum & Chandler 2002). Together with Cavit, IRM has been the most used temporary filling in endodontics (Koagel *et al.* 2008), even though its sealing capability has generated conflicting results (Mayer & Eickholz 1997, Naoum & Chandler 2002, Zmener *et al.* 2004, Koagel *et al.* 2008).

Discrepancies between studies still raise concerns about the capacity of temporary filling materials with different compositions to avoid bacterial percolation that could lead to post-treatment disease. As these materials have different setting mechanisms, different reactions with moisture and variable dimensional stability, there is a potential for them to produce different marginal sealing abilities. In addition, few studies have evaluated the *in vitro* performance of temporary fillings. Therefore, the aim of this study was to evaluate the marginal sealing ability, water sorption, solubility and toothbrushing abrasion resistance of different filling materials used as temporary restoration in root filled teeth.

Material and method

Temporary filling materials

Four temporary filling materials with different constituents and setting mechanisms were evaluated: a ZOE-based cement (IRM; Dentsply Caulk, Milford, DE, USA), a eugenol-free ZO cement (Cavit; 3M ESPE, St. Paul, MN, USA), a GIC (Vidrion R; SS White, Rio de Janeiro, RJ, Brazil), and a resin-based cement (Bioplic; Biodinamica, Londrina, PR, Brazil). Table 1 presents the composition of all materials.

Marginal sealing

Forty unrestored, caries-free human first and second molar teeth were selected under approval of the institutional Ethics Committee of School of Dentistry/Federal University of Pelotas (UFPel), Brazil (protocol no. 16/04). All teeth were examined at 10× magnification, and those with microcracks were excluded. The teeth were stored in 0.2% thymol solution for 7 days, after which the periodontal ligament was removed with a razor blade

Table 1 Temporary filling materials

Material	Composition	Manufacturer	Batch no.
Vidrion R	*Powder*: aluminium silicate glass *Liquid*: copolymers of polyacrylic, itaconic and tartaric acids	SS White	6040306
Cavit	Zinc oxide, calcium sulphate, zinc sulphate	3M ESPE	215000
Bioplic	Silicium dioxide, dimethacrylates, inorganic filler	Biodinâmica	632/05
IRM	*Powder*: Zinc oxide, polymethyl methacrylate *Liquid*: Eugenol	Dentsply Caulk	679307

and the teeth cleaned at low-speed with a water-pumice slurry. They were then stored in saline at 5 °C.

Class I endodontic access cavities with standardized outline were prepared using a handpiece under water-cooling. The coronal access to the pulp chamber started with a cylindrical diamond bur no. 1014 (KG Sorensen, Barueri, SP, Brazil) in enamel, and carbide burs no. 245 (SS White) in dentine. The burs were changed after 10 preparations. The pulp cavity and the root canals were rinsed with 1% NaOCl solution in order to remove debris. Root canals were dried through aspiration and using cotton pellets, and their entrance was filled with gutta-percha. To standardize the cavity depth, a periodontal probe was used to assure the existence of at least 4 mm between the cavity outline and the entrance of the root canals (Cruz *et al.* 2002). Since unrestored, caries-free molar teeth were used, the dentine surfaces after cavity preparation were sound.

The teeth were randomly assigned into four groups, defined by the temporary restorative fillings (Table 1). All materials were manipulated according to the manufacturers' specifications. IRM was prepared in a 6-g mL^{-1} powder/liquid ratio, and inserted and adapted to the cavity walls with a dental spatula. Vidrion R was manipulated and inserted with a Centrix syringe. For Cavit, the cavity was left slightly moist, the material inserted with a dental spatula and allowed to set in contact with a moist cotton pellet. Bioplic was inserted into the cavity, carved and light-cured for 40 s with a quartz–tungsten–halogen light-curing unit (Ultralux; Dabi Atlante, Ribeirão Preto, SP, Brazil – irradiance >400 mW cm^{-2}). The root apices were sealed with self-cured epoxy resin (Durepox; Alba Química Ind. e Com. Ltda., São Paulo, SP, Brazil) and teeth were covered with two coats of nail polish, except the restorations and a 1-mm area surrounding them.

After storage in saline for 7 days, at 37 °C, the teeth were submitted to 500 thermal cycles between 5 ± 5 and 55 ± 5 °C, with 30 s dwell time and 3 s interval time. The teeth were then immersed in 0.5% basic fuchsine solution for 24 h, at room temperature, and washed for 24 h in running tap water. Sectioning was performed bucco-lingually to the long axis of the tooth using a diamond disc. Two previously calibrated examiners analysed both sections using a stereomicroscope, at 40× magnification, recording the highest penetration score. Dye penetration was determined based on the following scores: 0 – no visible dye penetration at the tooth/filling interface; 1 – dye penetration limited to the dentine–enamel junction; 2 – dye penetration up to half of the pulp chamber; 3 – dye penetration over half of the pulp chamber. Data were submitted to nonparametric Kruskal–Wallis test ($P < 0.05$).

Water sorption and solubility

Disc-shaped specimens ($n = 7$), 6 mm in diameter (D) and 1 mm in height (h) were prepared for each material. The GIC specimens were prepared and allowed to set in the mould with polyester strips for 2 days, in order to avoid dehydration of the material. All specimens were stored in a desiccator at 37 °C with silica gel, and were weighed daily to verify mass stabilization (dry mass, m_1), which was represented by mass variations lower than 0.1 mg in any 24 h interval. Thereafter, the specimens were stored in distilled water at 37 °C for 7 days to obtain the mass after saturation with water (m_2).

The specimens were then placed in the desiccator again, at 37 °C, and reweighed again until a constant dry mass (m_3) was obtained. Weighing was performed using an analytical balance with 0.1 mg accuracy (AG 200; Gehaka, São Paulo, SP, Brazil). The volume (V) of each specimen was calculated based on the following equation: $V = \pi R^2 h$, where R is the specimen radius. Water sorption and solubility, given in μg mm^{-3}, were calculated as follows: $WS = m_2 - m_3/V$; $SL = m_1 - m_3/V$. Data were submitted to One-Way Analysis of Variance and Tukey's test ($P < 0.05$).

Toothbrushing abrasion and loss of mass

Five disc-shaped specimens were prepared for each material following the same procedures previously described. The specimens were ultrasonically cleaned (MaxiClean 750; Unique, Indaiatuba, SP, Brazil) in distilled water for 10 min and dry-stored at 37 °C for stabilization of specimen mass. The pre-brushing mass (m_1) was obtained by weighing the specimens every 24 h until a constant mass was achieved. The abrasion test was carried out in a multi-station brushing device. Each sample was brushed in a different station, using a soft nylon-bristled toothbrush with a brush-head load of 200 g. During the brushing cycle, the specimens were completely immersed in slurry of dentifrice (Colgate Total, São Bernardo do Campo, SP, Brazil) and distilled water (1 : 2 wt ratio). In total, 5000 strokes (forward and reverse movement) were performed with a frequency of 4 Hz at 37 °C.

After testing, the specimens were cleaned with a air/water spray for 1 min and in a ultrasonic bath for

10 min. They were then dry-stored at 37 °C to constant mass (m_2). Mass loss, expressed in mg, was calculated by the difference between m_2 and m_1. Data were submitted to One-Way Analysis of Variance and Tukey's test ($P < 0.05$). Representative specimens for each material before and after brushing were gold-sputter coated (Denton Vacuum Desk II; Denton Vacuum, Moorestown, NJ, USA) for observation with scanning electron microscopy (SEM). Imaging of the surfaces was performed in secondary electron mode (JSM-5600LV; Jeol Inc., Peabody, MA, USA) at accelerating voltage of 15 kV.

Results

Marginal sealing

Results are shown in Fig. 1. The Kruskal–Wallis test revealed statistically significant differences between groups ($P < 0.0001$). Bioplic produced the best marginal seal (all specimens with score 0), followed by Cavit. Vidrion R presented intermediate results, whilst IRM resulted in the poorest marginal seal (9 out of 10 specimens presenting score 3).

Water sorption and solubility

Results are shown in Fig. 2. Significant differences occurred between materials for water sorption ($P < 0.01$) and solubility ($P < 0.01$). Both parameters were significantly higher for Cavit. IRM and Vidrion R presented similar intermediate values for water sorption, whilst Bioplic had the lowest values. Significantly lower solubility was observed for Vidrion R and Bioplic compared with the other materials ($P < 0.05$).

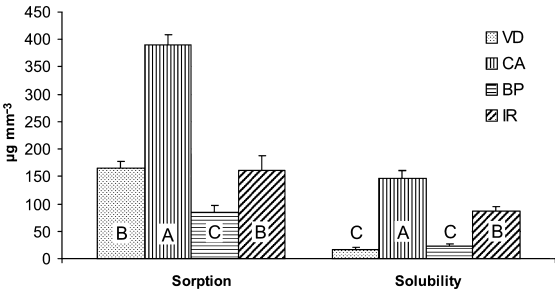

Figure 2 Results for water sorption and solubility. Distinct letters indicate statistical differences amongst materials ($P < 0.05$).

Toothbrushing abrasion and loss of mass

Results of loss of mass after toothbrushing are shown in Fig. 3.One specimen of Vidrion R fractured during the brushing cycling and was replaced. Significant differences were observed between materials ($P < 0.05$). Loss of mass after brushing was significantly higher for Cavit ($P < 0.05$). Bioplic had intermediate loss of mass values, similar to IRM and to Vidrion R, which had the lowest loss of mass of all groups. SEM micrographs of the control and brushed surfaces are shown in Fig 4. Before abrasion, a relatively smooth surface was observed for all groups, especially for Bioplic and IRM. After toothbrushing, all materials had characteristic worn surfaces, with Cavit and Vidrion R showing an aggressive wear pattern characterized by extensive loss of substance for Cavit, and deep grooved scratches

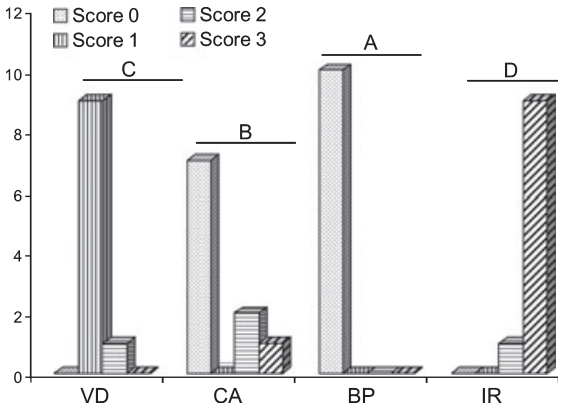

Figure 1 Marginal leakage observed for the different temporary filling materials. Distinct letters indicate statistical differences amongst materials ($P < 0.05$).

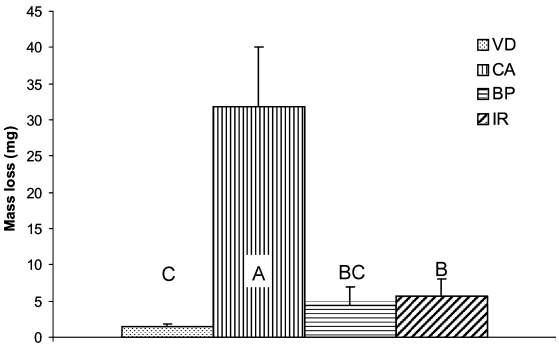

Figure 3 Mass loss (mg) of the temporary filling materials after toothbrushing abrasion. Distinct letters indicate statistical differences amongst materials ($P < 0.05$).

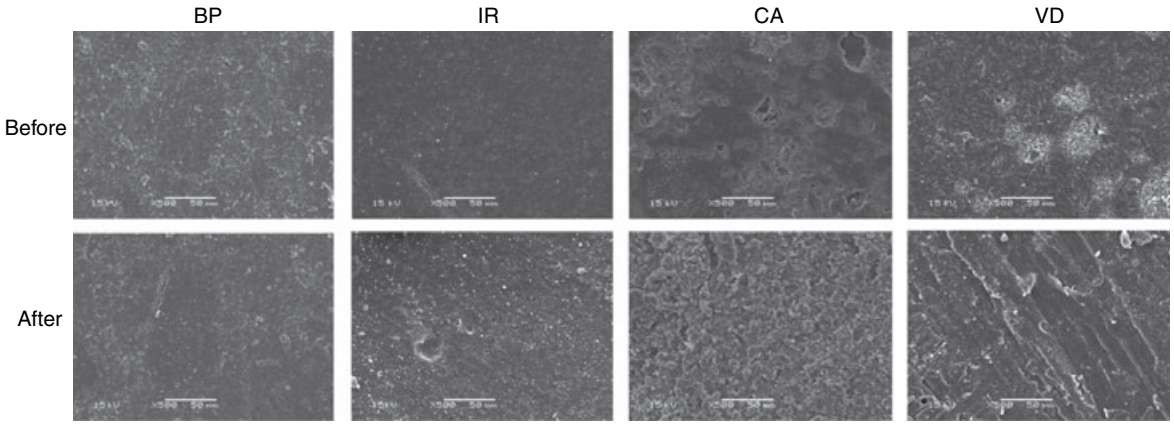

Figure 4 Representative SEM micrographs of the temporary filling materials before and after toothbrushing abrasion. A relatively smooth surface was observed for all groups before abrasion, especially for BP and IR. After toothbrushing, all materials presented characteristics of worn surfaces, with CA and VD showing an aggressive pattern of wear, characterized by extensive loss of substance for CA, and deep grooved scratches for VD. BP showed the least altered surface after abrasion.

for Vidrion R. Bioplic had the least altered surface after abrasion.

Discussion

The sealing ability of temporary fillings can be evaluated in several ways (Cruz *et al.* 2002, Naoum & Chandler 2002, Balto *et al.* 2005, Sauáia *et al.* 2006). According to Raskin *et al.* (2001), lack of standardization of the test methods compromises comparisons and, therefore, the reliability of marginal sealing results. Methodological aspects of the test used in the study, namely basic fuchsine as leakage tracer, the thermocycling protocol and the assessment of dye penetration through sections of the specimen, have been reported as the most frequent choices in marginal sealing evaluations (Raskin *et al.* 2001). In this sense, the test protocol employed allows comparisons with similar studies, besides being a rapid way to determine the sealing ability of the materials used.

Conventional GICs adhere to tooth tissue as a result of a chelation reaction with calcium (Culbertson 2001). Therefore, one could expect dye penetration in enamel to be lower than in dentine, since the former has more calcium available. However, 9 out of 10 specimens of Vidrion R group had dye penetration up to the enamel–dentine junction, which might reflect the effect of the thermocycling on the interaction between GIC and enamel and their different coefficients of thermal expansion. The bond strength of conventional GIC to tooth tissue is difficult to evaluate, due to the extremely brittle nature of the cement, which leads to cohesive failure within the material (Mount 1991). Thus, one could hypothesize that the tracer percolated through fracture lines within the cement, close to the tooth/restoration interface.

Bioplic, a dimethacrylate-based temporary filling, prevented dye penetration in all the specimens. This material has the advantage of not requiring etching of the dental surface or application of an intermediate bonding material, thus eliminating additional clinical steps. According to the manufacturer's information, Bioplic tends to expand in contact with moisture, improving its adaptation to the cavity walls. The light-curing characteristic of Bioplic seems to be an important factor on its sealing ability, as the contact with the wet environment occurs after polymerization. In a previous study, Jenkins *et al.* (2006) observed considerably higher marginal sealing ability for a resin-based light-cured material in comparison with conventional self-curing cements and other temporary fillings. Moreover, the translucency of Bioplic allows the passage of the curing light through the material, requiring a single light activation step, even with layers thicker than 2 mm.

The eugenol-free ZO cement Cavit sets in contact with moisture, and has produced conflicting sealing results (Uranga *et al.* 1999, Jenkins *et al.* 2006, Sauáia *et al.* 2006). The hygroscopic properties result in expansion of the material, potentially sealing the tooth/filling interface (Cruz *et al.* 2002, Sauáia *et al.* 2006), and might explain the absence of interfacial dye penetration in 70% of the samples. The presence of dye, though, was observed in the material itself, confirming

previous findings (Cruz *et al.* 2002) and indicating the possibility of recontamination of the canal by bacterial infiltration through the material itself.

The sealing ability of the eugenol-based ZOE cement (IRM) was poor, confirming previous reports (Deveaux *et al.* 1999, Balto *et al.* 2005). Extensive degradation was observed, with the presence of dye within the body of the material (Zmener *et al.* 2004). Studies have pointed out that stress, such as the one imposed by thermocycling, promotes a significant degradation of IRM (Gilles *et al.* 1975), whilst others indicate that variations in volume resulting from contraction of the material and the inhomogeneous mixing process could partially explain the poor sealing results with this filling (Deveaux *et al.* 1999). In addition, it has been reported that ZOE-based cements may impair the polymerization of resin composites, and should be avoided when final restorations of such materials are to be made (Naoum & Chandler 2002). In contrast, temporary fillings such as Bioplic and Vidrion R are compatible with resin-based materials, and theoretically do not need to be completely removed to execute the final restoration.

Water sorption and solubility were calculated by weight differences of specimens, and were used as a measure of the degradation of the fillings (Carvalho Júnior *et al.* 2003, Ferracane 2006) (Fig. 2). Carvalho Júnior *et al.* (2003), also determined the sealing ability of temporary fillings, and recommend that water sorption and solubility should be minimal. Usually, the absorption of water precedes events such as volumetric changes, swelling and softening of the materials (Ferracane 2006), which may compromise their microstructure and, as a consequence, the seal produced by the restoration.

Water uptake is a key factor in the setting mechanism of Cavit. The expansion caused by the water diffusion is responsible for the sealing of the tooth/restoration interface, but also allows the swelling of components from the spaces occupied by water (Ferracane 2006), explaining the high solubility observed for this material (Fig. 2). The intermediate sorption results observed with IRM and Vidrion R reflect the cement nature of these materials, which characteristically absorb water. IRM had greater solubility than Vidrion R, confirming the previously reported disintegration this cement undergoes in contact with moisture. This process was explained by Wilson & Batchelor (1970) as eugenol loss of the cement matrix by aqueous leaching, resulting in microstructural degradation and reduction of mechanical strength. It is important to highlight that Vidrion R specimens were dehydrated in order to reach the first dry mass. Although this procedure does not mimic the *in vivo* situation, it is inherent to the test and might have caused appreciable structural modifications in the GIC that might have influenced the results.

Resin-based materials have different patterns of water uptake, depending upon the chemical structure of the resin (Sideridou *et al.* 2007), which involves the hydrophilic nature of the monomers and differences between the solubility parameter of the monomers and the solvent (Ferracane 2006). In addition, the cross-link density of the polymer network is also important, since it dictates the presence and the amount of pendant molecules that could be swelled following water uptake (Ferracane 2006). In this sense, light-cured materials, such as Bioplic, justify their low water sorption and solubility by being able to set prior to contact with moisture.

Brushing simulation was used in the present study to test the surface wear and degradation of the fillings under cyclic mechanical challenge (Moraes *et al.* 2008). The eugenol-free ZO cement underwent considerable disintegration after brushing, as shown by the substantial loss of mass and the rough surface pattern observed (Figs 3 and 4). SEM images of the GIC Vidrion (Fig. 4) also depicted aspects of aggressive wear in the surface of the cement. Nevertheless, Vidrion had the lowest mass loss, indicating that the brushing action might affect only the surface of the material. SEM images of Bioplic and IRM (Fig. 4) revealed smoother surfaces after brushing, which indicate a more homogeneous wear pattern.

Conclusions

The resin-based light-cured temporary filling material Bioplic produced the best marginal sealing and was associated with the lowest water sorption, solubility and loss of mass in comparison with all other materials.

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