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Polymerization stress, flow and dentine bond strength of two resin-based root canal sealers

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Abstract

Souza SFC, Bombana AC, Francci C, Gonçalves F, Castellan C, Braga RR. Polymerization stress, flow and dentine bond strength of two resin-based root canal sealers. *International Endodontic Journal*, **42**, 867–873, 2009.

Aim To compare two resin-based root canal sealers (AH Plus and dual cure Epiphany) in terms of flow, polymerization stress and bond strength to dentine.

Methodology Flow was evaluated by measuring the diameter of uncured discs of sealer (0.5 mL) after 7 min compression (20N) between two glass plates ($n = 5$). Polymerization stress was monitored for 60 min in 1-mm thick discs bonded to two glass rods (Ø = 5 mm) attached to a universal testing machine ($n = 3$). Bond strength was analyzed through micropush-out test ($n = 10$) and failure mode was examined with scanning electron microscope (100× and 2500×). Data were statistically analyzed using the Student's *t*-test ($\alpha = 0.05$).

Results Polymerization stress was 0.32 ± 0.07 MPa for Epiphany self-cure, 0.65 ± 0.08 MPa for Epiphany light-cure and zero for AH Plus ($P < 0.05$). Flow data and bond strength values were 30.9 ± 1.1, 28.6 ± 0.7 mm and 6.3 ± 5.3, 17.8 ± 7.5 MPa for Epiphany and AH Plus, respectively ($P < 0.001$). Failure mode was predominantly cohesive in the sealer for both materials.

Conclusions Epiphany had higher flow and polymerization stress and lower bond strength values to dentine than AH Plus. In view of these findings it can be implied that AH Plus would provide a better seal.

Keywords: apical gap, flow, micropush-out, polymerization stress, root canal sealer.

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Introduction

Complete filling of the root canal system with biocompatible and dimensionally stable filling materials is an important factor in achieving endodontic success (Sjögren *et al.* 1990). Gutta-percha in combination with sealers of different chemical compositions has been widely used in clinical practice. However, filling completely the root canals system remains a challenge despite the large number of techniques and materials available (Schwartz 2006). Adhesive bonding and resin cements developed for endodontic use have emerged as a possibility to improve root canal filling (Weis *et al.* 2004). In 2004, a new adhesive root filling material, Epiphany™ Root Filling System, was patented by Pentron Clinical Technologies (Wallingford, CT, USA). This system contains a polyester-based thermoplastic root canal core material (Resilon; Resilon Research LLC, Madison, CT, USA), a dual-cure methacrylate-based sealer and a self-etching primer. This material can promote hybridization with the dentine substrate and a chemical bond with Resilon, improving resistance to bacterial leakage (Shipper *et al.* 2004, 2005) and root fracture (Teixeira *et al.* 2004a) due to a potential resin monoblock formation (Teixeira *et al.* 2004b). Nevertheless, an ultrastructural evaluation revealed a weak link between Resilon and dentine (Tay *et al.* 2005a).

Methacrylate-based sealers shrink during polymerization (Ferracane 2005), generating stress within the material and at the tooth-restoration interface that can

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lead to gap formation (Carvalho *et al.* 1996, Braga *et al.* 2002, De Munck *et al.* 2005). The magnitude of stress is influenced by several factors, such as composition and volume of the material and cavity configuration factor (factor-C) (Davidson & de Gee 1984, Davidson *et al.* 1984, Davidson & Feilzer 1997). In composite restorations, the use of low viscosity materials has been associated with a reduced incidence of marginal gaps at the tooth/restoration interface (Uno & Asmussen 1991, Peutzfeldt & Asmussen 2004) and better adaptation to cavity walls (Ferdianakis 1998, Fruits *et al.* 2002). On the other hand, viscosity is directly related to degree of conversion (Lovell *et al.* 1999, Sideridou *et al.* 2002) which, in turn, is a determinant factor on polymerization stress development (Braga & Ferracane 2002, Stansbury *et al.* 2005). The high C-factor situation represented by the filling of root canals may originate high polymerization stresses (Goracci *et al.* 2004), exceeding bond strength to root dentine and causing debonding of the interface for stress relief (Tay *et al.* 2005b). Furthermore, resin sealer photoactivation for immediate coronal sealing hinders the resin viscous flow and increases stress build-up (Ferracane 2005), resulting in inappropriate bond strength or gap formation between sealer and root dentine (Nagas *et al.* 2007).

The aim of this study was to compare an epoxy- and a methacrylate-based root canal sealer in terms of several characteristics involved in apical gap formation. The null hypothesis was that AH Plus™ (Maillefer, Dentsply Ind. e Com. Ltda., Petrópolis, RJ, Brazil) or Epiphany™ (Pentron Clinical Technologies, Wallingford, CT, USA) would show no difference terms of flow, polymerization stress and dentine bond strength.

Materials and methods

Flow

According to ADA 57 Specification (American National Standard/American Dental Association, 2000), 0.5 mL of sealers was mixed and placed using a graduated syringe, on a glass plate (40 × 40 × 5 mm). After 180 ± 5 s another glass plate was placed on top of the sealer, followed by load application of 20 N. Then, 10 min after mixing, the load was removed and maximum and minimum diameters of compressed discs were measured with a digital caliper with a 0.01 mm resolution (Mitutoyo MTI Corporation, Tokyo, Japan). Results were recorded only if both diameters were uniform and were within 1.0 mm. Flow was calculated by averaging five specimens.

Polymerization stress

Polymerization stress was determined using an established method (Condon & Ferracane 2000, Witzel *et al.* 2007, Gonçalves *et al.* 2008). One end of two glass rods (∅ 5 mm × 13 or 28 mm height) was sand-blasted with alumina (150–250 µm), silanated (RelyX Ceramic primer S; 3M ESPE, St Paul, MN, USA) and coated with a layer of unfilled resin (Adper™ Scotchbond Multi-purpose, bottle 3; 3M ESPE), which was exposed to the light source with 300 mW cm^{-2} for 40 s. The non-treated ends were attached to the opposite fixtures of a universal testing machine (Model 5565; Instron, Canton, MA, USA), and the distance between the treated surfaces was adjusted to 1.0 mm. The 28-mm rod was connected to a crosshead/load cell, whilst the 13-mm rod was connected to a stainless steel fixture containing a slot that allowed, when necessary the distal end of the light-curing guide to contact the rod opposite to the treated surface which was highly polished. Resin sealer (19.6 mm^3) was inserted between the treated glass surfaces and formed into a cylinder and excess was removed. An extensometer (Model 2630–101; Instron) was attached to the rods in order to monitor specimen height. The approximation of the glass rods due to composite shrinkage was registered by the extensometer and caused the crosshead to move in the opposite direction to restore the initial distance, with 0.01 µm accuracy. Therefore, the values registered by the load cell corresponded to the force necessary to maintain the initial height of the specimen in opposition to the contraction force exerted by the resin sealer (Fig. 1).

Three specimens were tested in each experimental condition at 37 °C, and force development was monitored for 60 min, starting 3 min after mixing. Experimental conditions were AH Plus, Epiphany self-cure (SC) and Epiphany light-cure (LC). Epiphany-LC was photoactivated (VIP Júnior; BISCO, Schaumburg, IL, USA) 17 min after mixing with 475 mW cm^{-2} for 51 s (24 J cm^{-2}), following manufacturer's instructions. Maximum nominal stress (σ, in MPa) was calculated by dividing the maximum contraction force [F (N)] by the cross-sectional area of the rods (A) as follows:

$$\sigma = \frac{F(\mathrm{N})}{A(mm^2)}$$

Micropush-out bond strengths

Twenty mandibular single-rooted human premolar teeth with straight root canals, anatomically similar

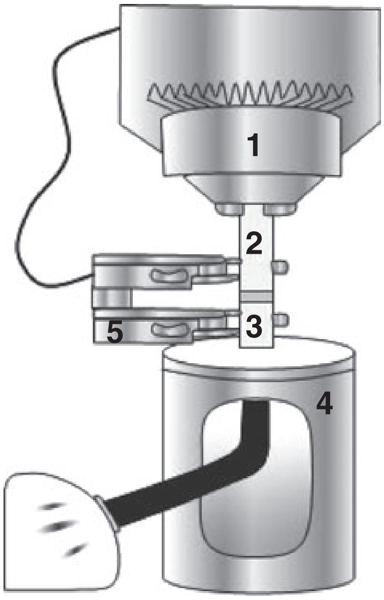

Figure 1 Schematic representation of the experimental set-up used for polymerization stress determination: (1) fixture conectect to the load cell; (2) long glass rod; (3) short glass rod; (4) stainless steel fixture with a slot to allow for the positioning of the light guide in contact with the glass rod; (5) extensometer.

dimensions, fully developed apices and patency foramen were collected after patient's informed consent had been obtained under a protocol reviewed and approved by the Ethical Research Committee of São Paulo University (protocol number, 177/05). Teeth were cleaned and the working length of each root was established with a size 15 K file (Dentsply Maillefer Ballaigues, Switzerland) 1.0 mm short of the apical foramen. Canals were prepared with a crown-down technique up to size 50 and irrigated with 0.5% NaOCl after every change of instrument. Five millilitres of 17% EDTA was used as final rinse to remove canal wall smear layer. EDTA solution was neutralized with 0.5% NaOCl and then the canal was rinsed with saline solution (15 mL) and dried with paper points.

Prepared root canals were randomly (http://www.random.org) divided into two experimental groups ($n = 10$): AH Plus (Dentsply Ind. e Com. Ltda.) and Epiphany-SC (Pentron Clinical Technologies). Three disc slices of one-millimetre thick (±0.1 mm) were obtained after transverse sectioning (Isomet 1000 Precision Saw; Buehler Ltd., Lake Bluff, IL, USA) the apical 5.0 mm of each root under water cooling. The thickness of each root slice was measured by means of a digital caliper (Mitutoyo MTI Corporation, Tokyo, Japan). The diameters of each apical and cervical slice were photographed by a digital camera (Q-Color 5; Olympus America Inc., Center Valley, PA, USA) attached to a stereomicroscope (SZ61; Olympus America Inc., Miami, FL, USA) and was measured using Image J software (http://rsb.info.nih.gov/ij/; National Institute of Health) under 25× magnification. Specimens with noncircular shape were discarded to avoid nonuniform stress distributions during testing, resulting in approximately 25 slices per group. Endodontic sealers were mixed according to manufacturer's instructions and used to fill the entire root canal space. Prior to filling with Epiphany sealer, root canal dentine was etched for 30 s with Epiphany primer. Specimens were stored for 72 h at 37 °C and 100% relative humidity.

For the micropush-out test, a compressive load was applied to the specimen via a cylindrical stainless steel punch attached to a universal testing machine (Kratos Dinamômetros, Embu, SP, Brazil). For each specimen, a punch tip 0.2 mm smaller than its apical diameter was selected and positioned such that it touched only the sealer and did not stress the surrounding root canal walls. The apical aspect of the each specimen was positioned facing the punch tip. Loading was performed at a crosshead speed of 0.5 mm min^{-1} until the sealer was dislodged from the root slice. Tensile bond strength of each slice was calculated as the force (N) of failure divided by the bonded cross-sectional surface area and expressed in MPa (Patierno *et al.* 1996).

Failure mode analysis

For scanning electron microscope (SEM) observation (100× and 2500×, LEO Stereoscan 440, Electron Microscopy Ltd., Cambridge, UK) micropush-out specimens were cut longitudinally and root segments were covered with platinum (Coating System MED 020; BAL-TEC AG, Balzers, Liechtenstein). To estimate the percentage of free substrate the interface area was divided into eight segments. This approach, suggested by Fowler et al. (1992), was used to classify failure mode as: (≥75%); cohesive within sealer (≤25%) adhesive-cohesive (>25% to <75%).

Statistical analysis

Data from bond strength to dentine, flow and polymerization stress were analyzed using the Student's t-test. For the bond strength test each tooth derived one single value. The level of significance was fixed at 5%.

Results

Table 1 summarizes average and SD of the micropush-out test and flow of both sealers. Epiphany presented significantly high flow than AH Plus ($P < 0.001$). A significant difference was detected between polymerization stress for Epiphany-SC (0.32 ± 0.07 MPa) and Epiphany-LC (0.65 ± 0.08 MPa) as shown in Fig. 2 ($P < 0.05$). Epiphany-SC started to generate stress 20 min after mixing. Epiphany-LC was photoactivated after 17 min from the beginning of the test, when an abrupt increase on polymerization stress curve occurred. AH Plus revealed zero polymerization stress values during 60 min, and for this reason was excluded from statistical analysis.

For the micropush-out test Epiphany-SC had lower values when compared with AH Plus ($P < 0.001$). Failure mode distribution is shown in Fig. 3: 79.2% cohesive within sealer and 20.8% adhesive for AH Plus, 78.3% cohesive within sealer and 21.7% adhesive-cohesive for Epiphany-SC.

Discussion

Apical gap formation is influenced by local factors such as substrate morphology (Wu *et al.* 1998, Ferrari *et al.* 2000, Mjör *et al.* 2001), C-factor (Goracci *et al.* 2004, Tay *et al.* 2005b), and also material-related factors such as physical properties of sealers (i.e. flow, polymerization contraction) (Bergmans *et al.* 2005, Braga *et al.* 2005) and bond strength to dentine (Tagger *et al.* 2002, Bouillaguet *et al.* 2003). This study assessed the possible relationship between flow, polymerization stress and bond strength of AH Plus and Epiphany sealers with apical gap formation.

Table 1 Mean values and standard deviations of bond strength to dentine and flow for AH Plus™ and Epiphany™ sealers

Groups	Micropush-out (MPa)	Flow (mm)
AH Plus	17.8 (7.5)[a]	28.6 (0.7)[b]
Epiphany	6.3 (5.3)[b]	30.9 (1.1)[a]

Different letters on the same column show statistically significant differences ($P < 0.001$).

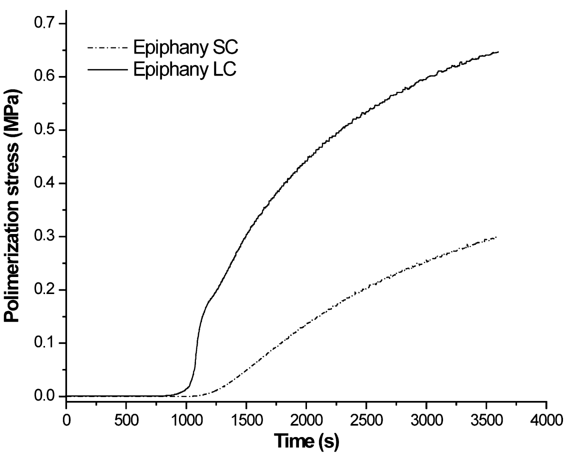

Figure 2 Polymerization stress (MPa) as a function of time (s) of Epiphany self-cure (SC) and light-cure (LC).

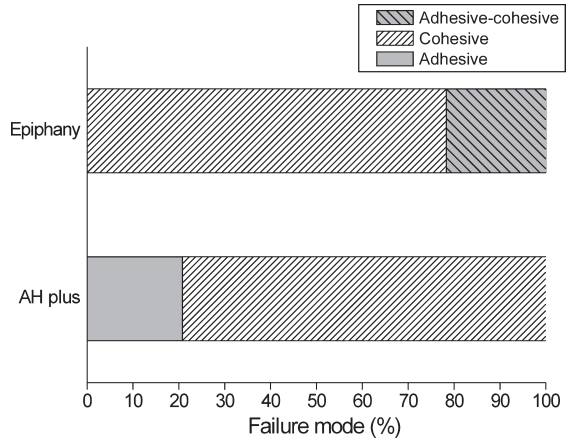

Figure 3 Failure mode distribution for experimental groups (%).

The fact that no stress development was observed for AH Plus up to 60 min after mixing agrees with the manufacturer information that states a setting time of 8 h at 37 °C. However, running the polymerization stress test for such long periods is impractical. Notwithstanding, this information is interesting for comparative purposes with the other sealer evaluated. For Epiphany, polymerization stress tests were performed for both curing modes: self-cured, relying only on the peroxide-amine reaction and dual-cured. Epiphany was tested in SC mode because clinically the light from photoactivation does not reach the middle or apical root regions (Hiraishi *et al.* 2005). The increased polymerization time in SC mode allows materials to flow in a pre-gel state, which could provide stress relief at the dentine/resin interface (Braga *et al.* 2002, Braga & Ferracane 2004), and be advantageous for this material. However, polymerization stress when light-curing was used (Epiphany-LC) doubled when compared with Epiphany-SC (Fig. 2; $P < 0.05$). This finding is related to an increase in polymerization rate caused by light activation. Nagas *et al.* (2007) suggested that a decreased polymerization time can adversely affect Epiphany bond strength to dentine. In fact, one could speculate that an

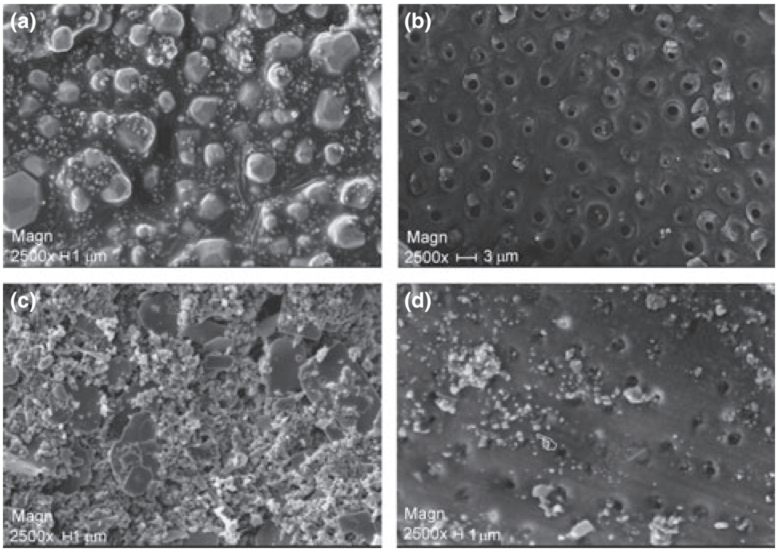

Figure 4 Representative scanning electron microscope (SEM) micrographs of failure mode for AH Plus™ (a and b) and Epiphany™ (c and d): (a) sealer cohesive failure showing dentine surface recovered by a thick organic matrix layer with different sizes fillers; (b) adhesive failure showing clean dentine surface only with small fillers and dentinal tubules with organic matrix tags; (c) sealer cohesive failure indicating dentine surface recovered by an organic matrix layer with granular small fillers, and major fillers with a thin plaque format, and also some empty spaces; (d) cohesive and adhesive failure demonstrating dentine surface covered by Epiphany primer and some sealers fragments with fillers closing total or partially dentinal tubules (pointer).

increased polymerization rate conferred by light activation can restrict the chances for polymerization stress release during the pre-gel state (Tay *et al.* 2005b).

In theory, total bond strength is the sum of the strengths of resin tags, hybrid layer and surface adhesion (Pashley *et al.* 1995). The low viscosity and hydrophilic nature of resin-based sealers in association with pressure caused by condensation technique allowed the sealer to infiltrate into dentinal tubules, forming long tags and secondary branchings (Bergmans *et al.* 2005, Tay *et al.* 2005a) In this study, both resin sealers differed in flow ($P < 0{,}001$; Table 1), and both of them exceeded specification 57 of American National Standard/American Dental Association (2000). Despite that, Tay *et al.* (2005a) showed in SEM and Transmission Electron Microscope (TEM) the loss of integrity at dentine/Epiphany sealer and gutta-percha/AH Plus sealer interfaces. These gaps, presumably created by polymerization contraction forces (Tay *et al.* 2005b), suggest that hybrid layer and long tags do not guarantee the absence of gaps (Bergmans *et al.* 2005).

Bond strength between endodontic cements and dentine may be an important property to provide a seal (Tagger *et al.* 2002). Micropush-out values for Epiphany were lower than for AH Plus ($P < 0.001$; Table 1). Epiphany polymerization stress may have contributed to its lower bond strength value. The amount of stress associated with shrinkage may result in separation of resin-based sealer and dentinal walls, and consequently, bond strength values of this interface would decrease (Hiraishi *et al.* 2005). In this study, bond strength results for Epiphany sealer are comparable with other experiments that showed values between 0.32 and 3.73 MPa (Gesi *et al.* 2005, Ungor *et al.* 2006, Fisher *et al.* 2007, Sly *et al.* 2007, Kaya *et al.* 2008, Lawson *et al.* 2008, Lee *et al.* 2008) though towards the high end range. Although filling the root canal only with the sealer does not accurately represent the clinical situation, this experimental model was chosen because it represents a worst case scenario, as polymerization stress development is directly related to the volume of shrinking material (Tay *et al.* 2005b). Moreover, by not using gutta-percha and resilon cones, it can be assured that the tested interface is comprised of sealer and dentine only.

Epiphany-LC was not included in the micropush-out test because the study was designed to simulate the clinical conditions found at the apical third of the root canal, where the effect of light-curing is likely to be zero. It is reasonable to speculate that, when used in SC mode, the sealer does not totally polymerize. The incomplete polymerization can impair cement mechanical properties and chemical stability (Braga *et al.* 2002). In fact, failure mode analysis revealed a high incidence of sealer cohesive failure for Epiphany (Figs 3 and 4).

The integrity loss on dentine/Epiphany interface can be explained by comparing its bond strength to dentine with stress generated during the polymerization contraction. Apparently, shrinkage stress was high enough to surpass bond strength (Bouillaguet *et al.* 2003, Tay *et al.* 2005a). The apparently negligible polymerization stress values determined in the mechanical test (Fig. 2) might be of a much higher magnitude in the root canal, where geometric shape and material confinement are obstacles for stress release. According to Tay *et al.* (2005b), C-factor of adhesive bonding root filling materials in root canals is highly unfavourable, challenging the concept of total bonding in root canals.

Conclusion

The null hypothesis was rejected for the three variables analyzed. Epiphany had higher flow, lower bond strength to dentine and also developed higher polymerization stress than AH Plus. Within the limitations of this laboratory study and in view of the results it can be speculated that, clinically, a better interfacial sealing could be expected with AH Plus. The higher bond strength to dentine obtained with AH Plus can be partially explained by its lower polymerization stress. Moreover, its higher viscosity compared with Epiphany did not seem to impair its bond strength.

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