MEDICAL SCIENCE MONITOR

MEDICAL TECHNOLOGY

e-ISSN 1643-3750
© Med Sci Monit, 2015; 21: 937-944
DOI: 10.12659/MSM.892741

Received: 2014.10.12
Accepted: 2014.12.02
Published: 2015.03.31

Comparison of Apical Microleakage of Dual-Curing Resin Cements with Fluid-Filtration and Dye Extraction Techniques

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Statistical Analysis C
Data Interpretation D
Manuscript Preparation E
Literature Search F
Funds Collection G

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Source of support: This work was supported by Research Fund of the Dicle University. Project Number: 12-DH-116

Background: Endodontically treated teeth with excessive loss of tooth structure are frequently restored using fiber posts. In this *in vitro* study, the apical leakage of self- and dual-activated curing modes for dual-curing resins cementing a translucent fiber post was evaluated using computerized fluid filtration meter and dye extraction method.

Material/Methods: One hundred and four extracted human maxillary incisors with single root and canal were used. Experimental samples embedded in a closed system were divided into 4 groups (n=20) according to 2 dual-curing luting systems, with 2 different curing modes (either with self- or light-activation): (1) Panavia F 2.0 with self-cure, (2) Panavia F 2.0 with light-activation, (3) Clearfill SA with self-cure, and (4) Clearfill SA with light activation. Twenty-four teeth served as negative and positive controls. Translucent fiber posts were luted in the roots except in the control groups.

Results: Statistical analysis indicated no significant difference in leakage among groups ($p>0.05$) with 4.12×10^{-4} (Panavia self-cure), 4.55×10^{-4} (Clearfill SA self-cure), 5.17×10^{-4} (Panavia dual-cure), and 5.59×10^{-4} (Clearfill SA dual-cure) in fluid-filtration method. Absorbance values for dye-extraction method were 266 nanometer (nm) (Panavia self-cure), 268 nm (Clearfill SA self-cure), 270 nm (Panavia dual-cure), and 271 nm (Clearfill SA dual-cure), in which difference among the groups were not statistically significant ($p>0.05$). When comparing the leakage, assessment methods results showed no statistically significant difference between the tested evaluation techniques ($p>0.05$).

Conclusions: Light- and self-activation curing modes of Panavia F 2.0 and Clearfill SA perform similar to each other in a closed system.

MeSH Keywords: **Curing Lights, Dental • Dental Leakage • Endodontics • Numerical Analysis, Computer-Assisted • Self-Curing of Dental Resins • Tooth, Nonvital**

Full-text PDF: http://www.medscimonit.com/abstract/index/idArt/892741

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Indexed in: [Current Contents/Clinical Medicine] [SCI Expanded] [ISI Alerting System] [ISI Journals Master List] [Index Medicus/MEDLINE] [EMBASE/Excerpta Medica] [Chemical Abstracts/CAS] [Index Copernicus]

Background

Endodontically treated teeth with excessive loss of tooth structure are frequently restored using fiber posts. The elastic modulus of these posts is similar to dentin, which allows favorable stress distribution [1] and reduces the risk of root fractures. The use of translucent fiber posts has recently increased because their light-transmission properties [2] enhance the aesthetic outcomes of final restorations. Composite resin in the post spaces is photoactivated by light transmission through the posts [3]. The use of these systems to bond both dentin and coronal cores has also been found to more evenly distribute forces along the root canal, thereby contributing to root reinforcement [3].

Resin-based bonding composites are commonly used during the cementation of fiber posts. Selection of the appropriate adhesive and composite to bond the post to the root canal dentin is a challenge that affects the success of the restoration [4,5]. Different types of bonding systems with different polymerization modes can be used with luting agents. The dentin bond may use either total- or self-etch adhesives [6], consisting primarily of an acidic conditioner, a primer, and an adhesive resin. Recent developments in adhesive dentistry have simplified these systems, which may be categorized as: a) 1-step etch and rinse systems, b) 2-step self-etching systems, and c) 1-step self-etching systems. Some studies have found these simplified systems to be less effective than conventional 3-step versions [7] or to be incompatible with chemically cured composites [5].

These resin-based bonding materials may use self-cured (chemical), light-cured, or dual-cured (light and chemical) polymerization modes [6]. Dual-cured resin cements combine the favorable characteristics of self- and light-cured materials. Because they can polymerize with or without light, these cements may provide a more reliable bond to post-space dentin, including that in the deep apical regions of the root [7]. However, studies have produced conflicting results for the performance of dual-cured resin cements; they have exhibited better mechanical properties with exposure to light [6,8,9], but have shown higher bond strength when the material was chemically initiated [10] without light curing.

Shrinkage and contraction stresses induced during polymerization are important problems associated with the use of composite resins [11]. Material components and curing modes are the primary factors influencing the amount of shrinkage [12]. Non-uniform adaptation or incomplete polymerization of the resin-based material may negatively affect the dentin bonding by decreasing the longevity of the adhesive interface [4,10], often resulting in microleakage. Leakage around the root canal system is a primary cause of root canal therapy failure. Fogel [13] conducted a fluid filtration microleakage test of several post-core systems and found that none of the systems achieved a fluid-tight seal. The analysis of sealing ability is important in endodontics. This ability has been measured using several methods, such as dye, isotope, and bacterial penetration, as well as electromechanical means [14,15]. These methods produce only qualitative results [16], but microleakage may be quantitatively measured using the computerized fluid filtration [16] and dye extraction [17] methods. Active pressure leakage assays are superior to passive assays because they overcome the problem of air entrapment, which may mask true leakage patterns [16].

This study therefore evaluated the effects of self- and dual-activated curing modes for dual-curing resin cements by using both a computerized fluid-filtration technique and dye extraction method to quantitatively measure apical microleakage in a closed system. The null hypothesis posited that different curing modes would have no effect on the quantity of microleakage among dual-cure resin cements.

Material and Methods

Specimen preparation

One hundred and four extracted, single-rooted, maxillary incisors were stored in distilled water with 0.1% thymol solution until collecting process of teeth has been completed for 1 week. Because type of storage solution effects dentine permeability over time [18], samples were rinsed under running water, cleaned of calculus, soft tissue, and other debris, then stored in distilled water (4°C) for 2 weeks until use under protocol ISO/TS 11405: 2003 [19].

The roots were standardized by decoronation to 14 mm from the cementoenamel junction to allow the placement of posts that were equal in length (Figure 1). The posts were shortened to 10 mm using a slow-speed diamond saw with copious water cooling. Working lengths were established at 1 mm short of the root apex. Cleaning and shaping were performed with nickel-titanium rotary instruments (ProTaper, Dentsply Maillefer, Ballaigues, Switzerland) to size F3 (9% taper with a diameter of 0.30 mm) with saline irrigation between files.

Following preparation, teeth were embedded into plastic cylinders containing silicone impression material (Optosil P Plus, Heraeus Kulzer GmbH, Hanau, Germany) in an upwards position to generate a closed system that mimics the oral environment as suggested previously [20]. Then they were randomly divided into 4 groups of 20 teeth each. To maintain minimum parameters and ensure that only the effectiveness of the dual-cure resin cements was evaluated [21], no roots were obturated with root canal sealer and gutta-percha.

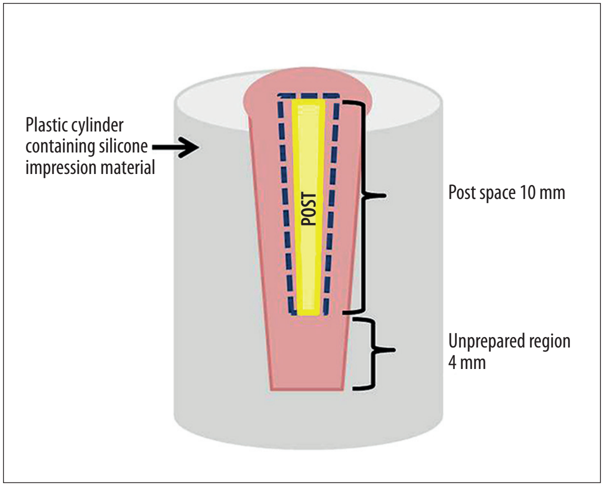

Figure 1. Illustration demonstrates the closed system that simulates the oral environment. Roots were embedded in silicone impression material until polymerization was completed.

Post spaces 10 mm in length were prepared using the drills supplied with the fiber post system. The apical 4 mm were not penetrated. Translucent fiber posts (Luscent Anchors; Dentatus, NY, USA) were cemented using dual-cured cement (Panavia F 2.0; Kuraray Medical, Osaka, Japan) and resin-based luting cement (Clearfill SA; Kuraray Medical). The cements were self- or dual-activated. Panavia F 2.0 consists of 2 pastes and a self-etching primer and uses a 2-step bonding procedure. Clearfill SA uses a 1-step bonding procedure that requires the cement to be loaded into an auto-mix delivery system. Post cementation was performed according to the manufacturer's instructions. For the light-activated groups, the power density of the light source was maintained between 1000 and 1100 mW/cm^2 with a digital radiometer (Jetlite light tester; J. Morita, Irvine, CA, USA).

Panavia dual-cured (PD) group

Equal amounts of PD Primer liquids A and B (Kuraray Medical, Osaka, Japan) were combined on a mixing dish, applied to the inside of the canal with a microbrush, and allowed to stand for 60 s. Excess liquid was eliminated with a paper point before completely drying the primer with a gentle air flow. Equal amounts of Panavia F pastes A and B (white shade; Kuraray Medical) were then combined for 20 s on the mixing plate and applied to the post with a brush. The cement-covered post was inserted into the root canal and polymerized for 20 s with a light-curing unit (Elipar Free Light 2; 3M ESPE, St. Paul, MN, USA) operating from the cervical orifice at a power density of 1000 mW/cm^2.

Panavia self-cured (PS) group

The teeth in this group were subjected to the same protocol as used for the PD, until the curing phase. No irradiation unit was used for this group, and the cements were allowed to cure without light.

Clearfill dual-cured (CD) group

Clearfill SA cement (white shade) with an auto-mixing delivery system was introduced into the post space using an Endo tip (Clearfill SA; Kuraray Medical) until excess resin was visible. The post was then immediately inserted into the root canal, excess resin was removed, and the post was polymerized for 20 s with a light-curing unit (Elipar Free Light 2) operating from the cervical orifice at a power density of 1000 mW/cm^2.

Clearfill self-cured (CS) group

The teeth in this group were subjected to the same protocol as used for the CD group, until the curing phase. No irradiation unit was used for this group, and the cements were allowed to cure without light.

After curing for 10 min, all specimens were removed from impression material and thermocycled for 500 cycles at 37°C using an automated thermocycling machine, prior to fluid-filtration test and dye penetration. Each root was then removed from the water and allowed to reach room temperature for 1 h before testing.

Generating positive and negative control groups

Twenty-four additional teeth served as negative (n=12) and positive (n=12) controls for fluid-filtration and dye extraction methods. Teeth in these groups were only enlarged to size F3 and were not restored by posts. Using the computerized fluid-filtration method, the negative control teeth (n=6) were completely coated with 3 layers of nail polish, including the apical foramina. Teeth for positive controls (n=6) were only enlarged to size F3 and they were not covered by nail polish. Using the dye extraction method, the negative control teeth (n=6) were completely coated with 3 layers of nail polish and positive control teeth (n=6) were not covered by nail polish.

Sample analyzing

Computerized fluid-filtration method

For the computerized fluid-filtration microleakage study, 40 root sections were inserted apex-first into a plastic tube and connected to an 18-gauge stainless steel tube. Cyanoacrylate adhesive (Scotch gel universal; 3M, Leiden, The Netherlands) was applied circumferentially between the root and the plastic tube. The sealing abilities of the specimens were measured using the computerized fluid filtration method described by Oruçoğlu et al. [16] (Figure 2). All operations were controlled with PC-compatible

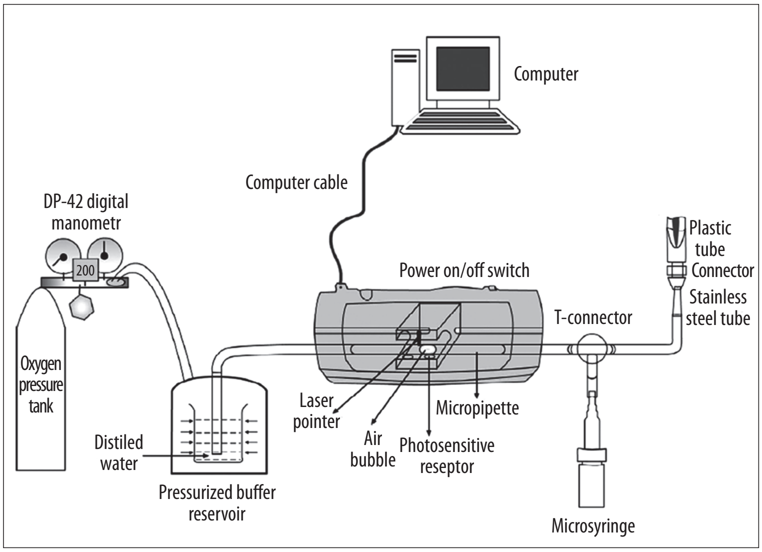

Figure 2. Computerized fluid-filtration technique uses a pressure tank (1.6 atm), digital air pressure regulator, and special software. The software automatically measures fluid movement in each sample every 2 min for an 8-min period. Leakage is expressed as μl/cmH_2O/min^{-1}.

Table 1. Mean microleakage and absorbance values are given for computerized fluid-filtration and dye extraction methods. The values are given in (μl/cmH_2O/min$^{-1}\cdot10^{-4}$) for fluid-filtration and in (nm) for dye extraction.

Experimental groups	Computerized fluid-filtration		Dye extraction
	n	Mean microleakage ±SD	Mean absorbance ±SD
Panavia self-cured (PS)	10	$4.12\times10^{-4}\pm0.000198$*	466.200±0.63*
Panavia dual-cured (PD)	10	$5.17\times10^{-4}\pm0.000188$*	467.600±1.14*
Clearfill self-cured (CS)	10	$4.55\times10^{-4}\pm0.000135$*	467.100±0.87*
Clearfill dual-cured (CD)	10	$5.79\times10^{-4}\pm0.000254$*	467.900±1.23*

* Groups with no significant difference were shown with the same supercripted letters.

software (Fluid Filtration'03, Konya, Turkey). A pressure tank (1.6 atm) was used to apply 160 kPa of O_2 to the apical side of the tooth through a digital air pressure regulator (DP-42 digital pressure and vacuum sensor; Sunx Sensors, Des Moines, IA, USA). The software automatically measured fluid movement in each sample every 2 min for an 8-min period. The quantity of leakage is expressed as μl/cmH_2O/min^{-1}, and means were determined.

Dye extraction method

For this method, 40 roots were covered with 2 layers of nail varnish (Vepa Kozmetik, Istanbul, Turkey), not including the last apical millimeter. The apices of the teeth were dipped in glass vials for 48 h at 37°C in a neutral-buffered 2% methylene-blue solution, under normal atmospheric pressure. After removal from the dye, the teeth were rinsed under tap water for 30 min and the varnish was removed using a sharp scalpel, and then stored individually in a glass vial containing 600 μl of concentrated (65%) nitric acid for 3 days. The vials were centrifuged at 14 000 rpm for 5 min, and 100 μl of the supernatant from each was then analyzed in a UV-Visible spectrophotometer (Shimadzu UV-160, Shimadzu Corp., Kyoto, Japan) after a kinetic assay at 550 nm wavelength using concentrated nitric acid as the blank.

Statistical analysis

The results were evaluated statistically using 1-way analysis of variance (Kruskall-Wallis H test) at 5% significance levels with Dunn multiple comparison post hoc tests. The analysis of correlation (Pearson test) was performed for the comparison of apical leakage methods.

Results

Computerized fluid-filtration method

Mean microleakage measurements and standard errors are shown in Table 1. The mean apical leakage values were 4.21, 4.53, 5.23, and 5.57 μl/cmH_2O/min^{-1}, and 10^{-4} for the PS, CS, PD, and CD groups, respectively. No significant difference in leakage was found among groups ($p>0.05$), except for both control groups ($p<0.001$).

The positive controls demonstrated extreme amounts of apical microleakage (mean: 89×10^{-4} μl/cmH$_2$O/min^{-1}; $p<0.001$), whereas no leakage was detected in the negative control group $p<0.001$).

Dye extraction method

The results of the dye extraction test are shown in Table 1. The mean absorbance values were 266, 268, 270, and 271 for the PS, CS, PD, and CD groups, respectively. The Kruskal-Wallis H-test was unable to detect significant differences among all experimental groups ($p>0.05$), except for both control groups.

The positive control showed the highest dye absorbance, with 2963 ($p<0.001$). Negative control samples had low absorbance (73) close to that of the blank (nitric acid), which had absorbance at 52 ($p<0.05$).

Computerized fluid-filtration method versus Dye extraction method

Evaluating the leakage methods, the Pearson correlation showed no statistically significant difference between the groups ($p>0.05$).

Discussion

Light curing of composite resins is impossible in many clinical situations involving inlay or onlay restorations, core materials, root canal posts, or opaque restorations [8,22]. Bonding is usually achieved through chemical curing in these situations. Polymerization may be light- or chemically activated (e.g., in the apical region) in dual-cure resins [8] during fiber post cementation.

The quality of adhesion at the post–cement–adhesive-dentin interfaces may be influenced by many factors, such as the action of sodium hypochlorite, hydrogen peroxide, ethylenediaminetetraacetic acid, or other irrigation solutions on dentin collagen, the type of bonding resin [23], unfavorable cavity configuration factors, the density and orientation of dentin tubules, the physical properties of the posts, and the remnants of root canal obturation materials [24]. To avoid affecting the results of the present study, saline was the only irrigant used, and the roots were not obturated prior to post cementation.

The configuration factor (C-factor) is an important consideration in dentin bonding procedures [11]. Where the C-factor is high, especially in prepared post spaces, slower-setting materials that allow material flow and relieve polymerization stress at the bonding interface are recommended [25,26]. Bouillaguet et al. [23] reported the fewest spontaneous failures with chemically cured cements, which set more slowly than dual-cured materials. Similarly, Goracci et al. [27] recommended slow-setting resin cements to prevent high polymerization shrinkage stresses along root canal walls. In addition, Tay et al. [26] suggested that when sealer thickness decreases, the unbounded flowable amount of sealer decreases, making the C-factor increase; thus thicker sealer reduces C-factors in root canals. Thicker sealers can partially compensate for the high theoretical C-factors by increasing the unbonded surface area and permitting some stress release by resin flow. In the present study, the auto-injection aspect of the Clearfill SA cement may have increased resin thickness between the post and root dentin, thereby performing similarly to the microleakage results with the Panavia F 2.0 as well (Table 1).

Our evaluation of apical microleakage with both fluid-filtration and dye-extraction techniques showed no significant difference among groups ($p>0.05$; Table 1). However, self-activated resins performed better than the light-activated resins. Our results might have been affected by using the closed system for study design. One can assume that, by embedding the samples into an impression material to simulate the oral environment, light-curing a dual-cement at the apex is probably impossible, leading to incomplete polymerization of the resin at this critical area. For this reason, probably all samples in the light-curing groups polymerized in a dual-cure manner and no difference among the groups were detected due to this 1 type of polymerization, as it was dual-curing of all samples, in the present study.

Material composition is an important factor that can influence the amount of shrinkage after polymerization [11]. To decrease viscosity and facilitate clinical handling, resin cements have lower filler content and therefore exhibit more volumetric shrinkage than do heavy filled composites [12]. The materials used in this study had high inorganic filler contents (Panavia F 2.0: 70.8%; Clearfill SA: 66%); lower filler contents may have resulted in greater polymerization shrinkage [28]. Better shrinkage results were obtained with Panavia F than with Clearfill SA, but this difference was not statistically significant. Our results are in accordance with those of Miyazaki et al. [29], who found that 10% filler content increased bond strength and improved the mechanical properties of bonding agents.

In contrast to Clearfill SA, Panavia F 2.0 has 2 components: a paste mixture and an ED primer containing co-initiators. Silva et al. [10] recommended the application of primer to the substrate before cementation to achieve proper dual-curing polymerization because the cement becomes light-independent when the primer is used. T-isopropylic benzenic sodium sulfinate is an important co-initiator in ED primer liquid B, as this salt may react with the acid resin monomers in primer liquid A and the resin cement to produce free radicals that would

Table 2. Chemical compositions and the application procedures of the materials tested in the study. The contents (*) are provided by the manufacturers.

Material	Batch#	Filler (%w)	Type	Chemical composition*	Application procedure
Panavia F 2.0	Paste A: 00245F Paste B: 00024B	70.8	Dual-cured resin cement	*Paste A:* – 10-methacryloyloxydecyl hydrogen phosphate (MDP) – Hydrophobic and hydrophilic dimethacrylate – Benzoyl peroxite – Camphoroquinone – Colloidal silica *Paste B:* – Sodium fluoride – Hydrophobic and hydrophilic dimethacrylate – Diethanol-p-toluidine – T-isopropylic benzenic sodium sulfinate – Barium glass – Titanium dioxide – Colloidal silica	• Mix Paste A and B in equal amounts for 20 s • Apply to root canal with lentulo drill • Remove resin excess with small brush • If light-cured, cure for 20 s
ED Primer	Primer A: 00207E Primer B: 00086E	0	One-step self-etch adhesive	*Primer A:* – 2-hydroxyethyl methacrylate (HEMA) – MDP – NM-aminosalicylic acid – Diethanol-p-toluidine – 5-NMSA sodium benzene sulfinate – Water *Primer B:* – NM-aminosalicylic acid – T-isopropylic benzenic sodium sulfinate – Diethanol-p-toluidine – Water	• Mix ED Primer A and B (1:1) • Apply the mixture for 60 s • Air-dry
Clearfill SA	0006AA	66	Dual-cured self-adhesive resin cement	*Paste A:* – 10-Methacryloyloxydecyl dihydrogen phosphate (MDP) – Bis-phenol A diglycidylmethacrylate (Bis-GMA) – Triethyleneglycol dimethacrylate (TEGDMA) – Hydrophobic aromatic – Dimethacrylate – dl-Camphorquinone – Benzoyl peroxide – Initiator – Silanated barium – Glass filler – Silanated colloidal silica *Paste B:* – Bis-phenol A diglycidylmethacrylate (Bis-GMA) – Hydrophobic aromatic dimethacrylate – Hydrophobic aliphatic dimethacrylate – Accelerators – Pigments – Surface treated sodium fluoride – Silanated barium glass filler – Silanated colloidal silica	• Place the auto-mix syringe tip • Dispense material until the working area is fulled • Remove resin excess with small brush • If light-cured, cure for 20 s

enhance the polymerization reaction [30]. In the present study, the minimal apical leakage observed in the PS group may be directly related to this process.

Sulfinates act as initiating compounds during polymerization in simplified adhesive systems [8]. The sulfinate salts are converted to acids when they contact the acidic monomers of the dentin surface and are capable of initiating polymerization without co-initiators [8]. In the present study, the Panavia F paste and ED primer (Table 2) contained sulfinic acid, but the Clearfill SA cement did not. The superior results of the self- and dual-cured Panavia F 2.0 groups in comparison with the Clearfill SA groups may be related to this component difference. Asmussen and Peutzfeldt [8] suggested that pretreatment with sulfinate solution increased the bond strength of the material.

Piwowarczyk et al. [25] reported higher bond strength after water storage and thermal cycles when Panavia F cement was self-cured without light activation. The ED primer component of Panavia F is a water-based adhesive, and thus is not sensitive to surface moisture content [24]. A water-based adhesive may be especially practical in cases where moisture cannot be eliminated completely, such as the deep regions of prepared root canals. These factors may also have positively affected the apical microleakage results of the Panavia F 2.0 cement.

Because the amount of light passing through the cement may also influence bond strength [7], the present study used white resin shades to minimize variation due to this factor. Although about 1000 mW/cm^2 of light was delivered, attenuation of this energy when passing through deep and narrow post spaces may have adversely affected the light curing of both cements. Such attenuation may have increased microleakage quantities in the light-activated groups, as previously observed in a clinical situation [8].

Conclusions

Within the limitations of this study performed in a closed system, in both leakage tests apical microleakage quantities were similar among light- and self-activated curing modes of dual-cured resins used with translucent fiber posts either with fluid-filtration or with dye-extraction methods. Our null hypothesis was thus confirmed. From the standpoint of simplicity, 1-step self-adhesive composite Clearfill SA cement showing slow polymerization may be safely used in root canal adhesion that may relieve shrinkage stress regarding the resin flow, because of the prolonged gelation time.

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