

# Setting Times of Resilon and Other Sealers in Aerobic and Anaerobic Environments

Benjamin A. Nielsen, DMD, William J. Beeler, DMD, MS, Christina Vy, DMD, and J. Craig Baumgartner, DDS, PhD

## Abstract

Eleven sealers, including Resilon sealer, were mixed according to manufacturer's instructions. Setting times were determined in both aerobic and anaerobic environments. Two samples of each sealer were mixed and placed in the following conditions: (a) uncovered in an aerobic incubator; (b) covered with a glass cover-slip in the aerobic incubator; (c) covered with phosphate buffered saline in an aerobic incubator; (d) uncovered in an anaerobic incubator; (e) light cured and then placed in the aerobic incubator (Resilon only). All samples were tested for setting times with a Gillmore needle at 15, 30, and 60 minutes, then hourly up to 8 hours, then at 24, 48, and 72 hours, and then weekly up to 3 weeks. Ketac Endo and Kerr Tubliseal, were the fastest sealers to set in aerobic environments. Ketac Endo and Resilon were the fastest sealers to set in anaerobic environments. Roth 801 and Roth 811 were the slowest sealers to set, taking over 3 weeks to set in either anaerobic or aerobic environments. Resilon sealer set in 30 minutes in both anaerobic environments. However, in the presence of air, Resilon took a week to set and when placed in PBS, an uncured layer remained on the surface. (*J Endod* 2006;32:130–132)

From the Oregon Health & Science University, Portland, Oregon.

Address requests for reprint to Dr. Craig Baumgartner, Department of Endodontology, 611 Campus Dr., Portland, OR 97239-3097. E-mail address: baumgarc@ohsu.edu. 0099-2399/\$0 - see front matter

Copyright © 2006 by the American Association of Endodontists.

doi:10.1016/j.joen.2005.10.024

The use of a sealer during root canal obturation with gutta-percha is considered essential to prevent microleakage. Numerous sealers have been used for this purpose. Resilon sealer is a recently marketed dual-cure, resin-based sealer. The Resilon system consists of a primer, a sealer, and synthetic polymer cones or pellets intended to form a "monoblock" to seal the root canal system (1). Resilon has been shown to create a satisfactory seal and possibly strengthen the root (1, 2). The manufacturer's instructions for Resilon state that light curing for 40 seconds can create an immediate coronal seal at the orifice with the rest of the sealer setting in 25 minutes (3). Commonly used resin cements have been shown to require anaerobic environments (4–6). The manufacturer's instructions for Resilon do not describe the need for an anaerobic environment for setting. However, in a pilot study, when root canals of extracted human teeth were filled with Resilon, the Resilon sealer at the orifice did not completely set after two days. The Resilon sealer left on the glass slab after mixing also did not set completely after the same amount of time. Most studies evaluating the setting times for sealers have not used both aerobic and anaerobic environments to simulate the root canal system and surrounding tissues (7–13).

The purpose of this study was to compare the setting times of the Resilon sealer with other commonly used sealers in both aerobic and anaerobic environments. In addition, light curing of the Resilon sealer was evaluated.

## Materials and Methods

Eleven sealers, including Resilon sealer were tested (Table 1). The sealers were mixed following manufacturer's instructions on a glass slab using a spatula. Ketac Endo Aplicaps (3M ESPE, St. Paul, MN) were mixed according to manufacturer's instructions using an amalgamator. Resilon sealer (Real Seal, Sybron Endo, Glendora, CA) was mixed using the syringe and mixing tips provided in the kit.

To study setting times of endodontic sealers, the American National Standard/American Dental Association Specification No. 57, 1999, recommends an incubator at a temperature of  $\pm 37^{\circ}\text{C}$  with relative humidity of not less than 95%. A Gillmore type needle is recommended with a mass of  $100 \pm 0.5$  g having a flat end  $2.0 \pm 0.1$  mm in diameter, with the needle cylindrical for a distance of approximately 5 mm from its end. The end of the needle should be at right angles to the axis of the rod (14).

O rings (Midwest rubber) were used as molds with an internal diameter of 10 mm and a thickness of 3 mm. The O rings were placed on white glazed tiles and filled with sealer. Two samples of each sealer were tested under the following conditions: (a) aerobic environment at  $37^{\circ}\text{C}$  and 100% humidity (incubator); (b) covered in phosphate buffered saline (PBS) in the incubator; (c) covered with glass slide cover slips in the incubator; (d) placed in an anaerobic chamber (Fig. 1). To test under an anaerobic condition, sealers were tested at  $37^{\circ}\text{C}$  in an anaerobic chamber (Bactron II Anaerobic Chamber, Sheldon Manufacturing Inc., Cornelius, OR) with an atmosphere of 85%  $\text{N}_2$ , 5%  $\text{CO}_2$ , and 10%  $\text{H}_2$ . Two samples for each group were used to assure consistent setting times within the same group.

The setting of each sample was tested using a Gillmore needle (both aerobic and anaerobic) that was carefully lowered vertically onto the flat surface of the sealer. This was repeated until needle indentations ceased to be visible (Fig. 1). The time, from start of mixing, until the indentations ceased was recorded. The samples were tested at 15, 30, 60 minutes then every 60 minutes up to 8 hours. If necessary, they were checked daily at 24, 48, and 72 hours and then weekly up to 3 weeks.

**TABLE 1.** Setting times in aerobic and anaerobic environments incubated at 37°C

Sealers	Manufacturer	Aerobic Environment		Anaerobic Environment	
		UC <sup>1</sup>	PBS <sup>2</sup>	UC <sup>3</sup>	CS <sup>4</sup>
Resilon	Sybron Dental, USA	1 Week	3+ Wks*	30 Min	30 Min
Sultan U/P Root Canal Sealer	Sultan Chemists, USA	4 Hrs	2 Hrs	24 Hrs	2 Hrs
Kerr Regular Set	Kerr Sybron, USA	24 Hrs	2 Hrs	24 Hrs	5 Hrs
Kerr Extended Working Time	Kerr Sybron, USA	24 Hrs	3 Hrs	24 Hrs	24 Hrs
Kerr Tubiseal Regular Set	Kerr Sybron, USA	2 Hrs	1 Hr	4 Hrs	30 Min
Kerr Tubiseal Extended Working Time	Kerr Sybron, USA	2 Hrs	2 Hrs	3 Hrs	1 Hr
Ketac Endo	ESPE-Premier, USA	30 Min	2 Hrs	30 Min	30 Min
AH-Plus	Dentsply, USA	24 Hrs	24 Hrs	24 Hrs	24 Hrs
Roth 801, Elite	Roth International, USA	3+ Wks*	3+ Wks*	3+ Wks*	3+ Wks*
Roth 811, Elite	Roth International, USA	3+ Wks*	3+ Wks*	3 Wks	3+ Wks*
Pulpdent Root Canal Sealer	Pulpdent Corporation, USA	6 Hrs	3 Hrs	3 Hrs	6 Hrs

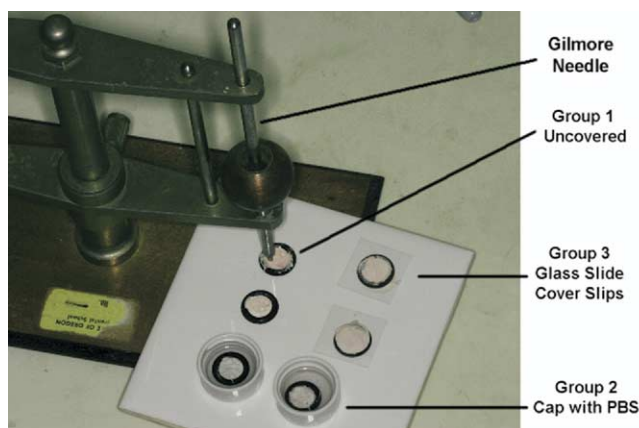
1. Uncovered in air. 2. Covered with phosphate buffered saline. 3. Uncovered in anaerobic environment. 4. Surface sealed with glass slide cover slip.

\*Setting time exceeded three weeks when last tested.

To evaluate the light cure setting times of Resilon sealer, an Optilux curing unit (Demetron Research Corp., Danbury, CT) was used with an intensity reading of 480 mw/cm<sup>2</sup> measured from a curing radiometer (Model 100, Demetron Research Corp.). Resilon sealer was placed in four different O-rings and the light source was placed within 1 mm of the surface for the first ring, at 3 mm for the second ring, 6 mm for the third ring, and 9 mm for the fourth ring. Samples were tested with the Gillmore needle at 20-s increments of exposure to the light at each distance.

## Results

The setting times for sealers in both aerobic and anaerobic environments ranged from 30 minutes to over 3 weeks (Table 1). All pairs of samples in each group had the same setting time and are recorded in Table 1. Resilon sealer set in 30 minutes in both the anaerobic environments. However, in the presence of air, Resilon took a week to set and when placed in PBS, an uncured layer remained on the surface that indented with the Gillmore needle. The Resilon under this uncured surface layer set in 30 minutes. Similarly an uncured layer over a set layer was observed on the uncovered samples stored in the aerobic incubator. Unlike the PBS samples, after 1 week, the top layer did set completely. For the light cured Resilon sealer samples, no indentations were noted with the Gillmore needle after 20 seconds at all distances evaluated with the light source. However, as the light source to sealer distance increased, the cured layer was thinner and more fragile.



**Figure 1.** Samples of groups 1 to 3 for Resilon. Samples are shown on the tile while measuring “set” with the Gillmore needle.

## Discussion

This study used both aerobic and anaerobic environments to study setting times of eleven endodontic sealers. The anaerobic environments simulated a closed root canal system and the aerobic environments simulated the canal orifice and periradicular tissues. Of most interest was the Resilon sealer. The surface of the Resilon sealer, when left exposed to an aerobic environment, set differently than in anaerobic environments where it set completely. The Gillmore needle continued to indent the surface for up to 1 week in samples left open to air, but the material under the soft, top layer was hard after 30 minutes. The same was true for the Resilon sealer covered with PBS except that there was a top surface layer that remained unset over 3 weeks. PBS was used to simulate aerobic periapical tissue fluid in the event that sealer was extruded from a root-end. Oxygen tension in periradicular tissues may inhibit the setting of extruded Resilon sealer producing a possible cytotoxicity (15–21). The partial pressure of oxygen (pO<sub>2</sub>) in air is about 150 mm Hg (22). Oxygen diffuses down a gradient as it passes through the lungs and blood to tissues where the pO<sub>2</sub> ranges from 30 to 40 mm Hg (22). Free radical polymerization of resins is inhibited by oxygen (23–26). Potential sources of oxygen are air, water, and body tissues or fluids. Oxygen creates an inhibited layer and results in a chemically active surface. The degree of conversion in resins represents the percentage of double bonds of molecules in the resin, such as Bis-GMA, that react (27). The degree of conversion is generally higher for photo-initiated or dual cure polymerization (27). The molecules that do not react are a potential source of toxicity. This toxicity, and how it affects dental pulps, has been studied but it has not been studied in human periradicular tissues (15–21). In a pilot study, it was noticed that the glass cover slip had to be in intimate contact with the sealer for it to set completely. The surface of Resilon sealer not in contact with glass, which was exposed to an aerobic environment, did not set immediately. Likewise, in a pilot study, the layer of Resilon sealer in contact with PBS did not set unless the PBS was prereduced (oxygen removed from solution).

In a recent study, Resilon was used *in vivo* in dogs (28). The amount of apical periodontitis, as determined by inflammation, was assessed. All inflammation seen was mild, but there was less inflammation seen in the teeth obturated with the Resilon system compared to the group using the resin sealer AH 26 and gutta-percha (28). No mention was made in this study if any extrusion of sealer was seen upon examination of the sections (28). If sealer was not extruded, the orifice light cured, and there was no contact with oxygen, then the Resilon sealer was probably set in 30 min with an effective seal. A future study should

examine the periradicular response to extruded Resilon. The present study suggests that Resilon will not set completely in periradicular tissues.

Measurements to determine the setting time of the sealers in aerobic environments were done by taking the sample out of the incubator and measuring the samples on a countertop at room temperature. This process took less than 1 minute. Samples stored in the anaerobic chamber were mixed, stored and measured in an anaerobic environment because of the time necessary to move samples in and out of the chamber.

Light cured Resilon sealer was tested at four distances from the light source to simulate an endodontically treated tooth where the sealer and obturation material are often several millimeters from the occlusal surface and light source. In curing other resins, distance from the light source has been shown to affect setting of the resin (29–30). A set surface of Resilon sealer was produced even when the curing light was 9 mm from the surface after 20 seconds. However, even though no indentations were noticed, the weight of the needle depressed the cured layer and it was observed that the sealer under the cured layer was not set. The weight of the needle cracked the thin cured layer covering the uncured resin. Curing with the light for 40 and 60 seconds increased the thickness of cured resin. When the Resilon sealer was light cured and then allowed to set for an additional 30 minutes in the aerobic incubator, no uncured top layer was observed like with the aerobic and PBS samples. Under conditions of this study, a curing time of more than 40 seconds is recommended if the curing light cannot be placed immediately over the sealer, as is often the case. The curing time may be affected by the type of curing light used (31). It appears that light curing the top layer of this sealer is the most predictable way to get the most thorough setting of material at the orifice.

With the other sealers, differences in setting times were often observed with the same sealer under different environments (Table 1). Ketac Endo was the fastest to set in an aerobic environment (30 min). Ketac Endo, Kerr Tubliseal regular set, and Resilon were the fastest to set in anaerobic environments (30 min). Roth 801 and Roth 811 were the slowest sealers to set. It took more than 3 weeks for the Roth sealers to set in either anaerobic or aerobic environments. The other sealers had intermediate setting times.

### References

- Shipper G, Orstavik D, Teixeira FB, Trope M. An evaluation of microbial leakage in roots filled with a thermoplastic synthetic polymer-based root canal filling material (Resilon). *J Endod* 2004;30:342–7.
- Teixeira FB, Teixeira ECN, Thompson JY, Trope M. Fracture resistance of roots endodontically treated with a new resin filling material. *JADA* 2004;135:646–52.
- Real Seal soft resin endodontic obturation system instructions for use. SybronEndo. Glendora, CA. REF 077–0143 Rev. A.
- Reilly B, Davis EL, Joynt RB, Quevedo J. Shear strength of resin developed by four bonding agents used with cast metal restorations. *J Prosthet Dent* 1992;68:53–5.
- Mendoza DB, Eakle WS. Retention of posts cemented with various dentinal bonding cements. *J Prosthet Dent* 1994;72:591–4.
- Pegoraro LF, Barrack G. A comparison of bond strengths of adhesive cast restorations using different designs, bonding agents, and luting resins. *J Prosthet Dent* 1987;57:133–8.
- Allan NA, Walton RE, Schaffer M. Setting times for endodontic sealer under clinical usage and in vitro conditions. *J Endod* 2001;27:421–3.
- Weiner B, Schilder H. A comparative study of important physical properties of various root canal sealers I. Evaluation of setting times. *J Oral Surg* 1971;32:768–77.
- Grossman L. Physical properties of root canal cements. *J Endod* 1976;2:166–75.
- Higgenbotham T. A comparative study of the physical properties of five commonly used root canal sealers. *Oral Surg Oral Med Oral Pathol* 1967;24:89–101.
- McComb D, Smith D. Comparison of physical properties of polycarboxylate-based and conventional root canal sealers. *J Endod* 1976;2:228–34.
- Benatti O, Stolf W, Ruhnke L. Verification of the consistency, setting time, and dimensional changes of root canal filling materials. *Oral Surg Oral Med Oral Pathol* 1978;46:107–13.
- Caicedo R, von Fraunhofer J. The properties of endodontic sealer cements. *J Endod* 1988;14:527–33.
- Proposed Am Nation Standard/Am Dental Association Specification No. 57. Endodontic Sealing Materials: 5.6 setting time: Council of Scientific Affairs, Draft Revision; 1999:6.
- Hanks CT, Strawn SE, Wataha JC, Craig RG. Cytotoxic effects of resin components on cultured mammalian fibroblasts. *J Dent Res* 1991;70:1450–5.
- Ratanasathien S, Wataha JC, Hanks CT, Dennison JB. Cytotoxic interactive effects of dentin bonding components on mouse fibroblasts. *J Dent Res* 1995;74:1602–6.
- Wenneberg A, Mjör IA, Hensten-Petersen A. Biological evaluation of dental restorative materials: a comparison of different methods. *J Biomed Mater Res* 1983;17:23–36.
- Hanks CT, Wataha JC, Parsell RR, Strawn SE. Delineation of cytotoxic concentration of two dentin bonding agents in vitro. *J Endod* 1992;18:589–96.
- de Souza Costa CA, Lopes do Nascimento AB, Teixeira HM, Fontana UF. Response of human pulps capped with a self-etching adhesive system. *Dent Mater* 2001;17:230–40.
- Geurtsen W. Biocompatibility of resin-modified filling materials. *Crit Rev Oral Biol Med* 2000;11:333–55 (review).
- Geurtsen W, Lehmann F, Spahl W, Leyhausen G. Cytotoxicity of 35 dental resin composite monomers/additives in permanent 3T3 and three human primary fibroblast cultures. *J Biomed Mater Res* 1998;41:474–80.
- Ganong WF. Review of medical physiology, 21<sup>st</sup> ed. New York: Lange Medical Books/McGraw-Hill, 2003:669.
- Rueggeberg FA, Margeson DH. The effect of oxygen inhibition on an unfilled/filled, composite system. *J Dent Res* 1990;69:1652–8.
- Mohsen NM, Craig RG, Hanks CT. Cytotoxicity of urethane dimethacrylate composites before and after aging and leaching. *J Biomed Mater Res* 1998;39:252–60.
- Rathbun MA, Craig RG, Hanks CT, Filisko FE. Cytotoxicity of a BIS-GMA dental composite before and after leaching in organic solvents. *J Biomed Mater Res* 1991;25:443–57.
- Anusavice KJ. Phillips' science of dental materials, 11<sup>th</sup> ed. St. Louis, MO: Saunders, 2003:161.
- Craig RG, Powers JM. Restorative dental materials, 11<sup>th</sup> ed. St. Louis, MO: Mosby, 2002;Chapter 7.
- Shipper G, Teixeira FB, Arnold RR, Trope M. Periapical inflammation after coronal microbial inoculation of dog roots filled with gutta-percha or Resilon. *J Endod* 2005;31:91–6.
- Rueggeberg FA, Jordan DM. Effect of light-tip distance on polymerization of resin composite. *Int J Prosthodont* 1993;6:364–70.
- Sakaguchi RL, Douglas WH, Peters MC. Curing light performance and polymerization of composite restorative materials. *J Dent* 1992;20:183–8.
- Nomoto R, McCabe JF, Hirano S. Comparison of halogen, plasma, and LED curing units. *Oper Dent* 2004;29:287–94.