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Effect of compression load and temperature on thermomechanical tests for gutta-percha and Resilon®

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Abstract

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Aim To analyse a method used to evaluate the thermomechanical properties of gutta-percha and Resilon® at different temperatures and compression loads.

Methodology Two hundred and seventy specimens measuring 10 mm in diameter and 1.5 mm in height were made from the following materials: conventional gutta-percha (GCO), thermoplastic gutta-percha (GTP) and Resilon® cones (RE). After 24 h, the specimens were placed in water at 50 °C, 60 °C or 70 °C for 60 s. After that, specimens were placed between two glass slabs, and loads weighing 1.0, 3.0 or 5.0 kg were applied. Images of the specimens were digitized before and after the test and analysed using imaging software to determine their initial and final areas. The thermomechanical property of each material was determined by the difference between the initial and final areas of the specimens. Data were subjected to ANOVA and SNK tests at 5% significance. To verify a possible correlation between the results of the materials, linear regression coefficients (r) were calculated.

Results Data showed higher flow area values for RE under all compression loads at 70 °C and under the 5.0 kg load at 60 °C ($P < 0.05$). Regarding gutta-percha, GTP showed higher flow under loads weighing 3.0 and 5.0 kg, at 60 and 70 °C ($P < 0.05$). GCO presented higher flow at 70 °C with a load of 5.0 kg. Regression analyses showed a poor linear correlation amongst the results of the materials under the different experimental conditions.

Conclusion Gutta-percha and Resilon® cones require different compression loads and temperatures for evaluation of their thermomechanical properties. For all materials, the greatest flow occurred at 70 °C under a load of 5.0 kg; therefore, these parameters may be adopted when evaluating endodontic filling materials.

Keywords: dental materials, endodontic filling materials, gutta-percha, Resilon®.

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Introduction

The main objective of root canal filling is to completely seal the root canal system (Schilder *et al.* 1974, Wu *et al.* 2001). This procedure is normally carried out using gutta-percha and an endodontic sealer. Whilst some filling techniques use cold gutta-percha cones, others involve thermomechanical compaction of the material (Tagger *et al.* 1984, Wu *et al.* 2001, De-Deus *et al.* 2008). Several filling techniques using thermoplasticized gutta-percha have been highlighted for their effectiveness when sealing root canal systems with irregular anatomy, or in the presence of lateral canals (Dulac *et al.* 1999, Bowman & Baumgartner 2002, Ordinola-Zapata *et al.* 2009).

Resilon® (Resilon Research LLC, Madison, CT, USA) is a synthetic thermoplastic polymer-based root canal

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filling material. Several studies have confirmed the thermoplastic ability of the polymers in Resilon® (Shipper *et al.* 2004, Tay *et al.* 2005, Carvalho-Junior *et al.* 2007, Maniglia-Ferreira *et al.* 2008, Sant'Anna-Junior *et al.* 2009), justifying its use in filling techniques that require the material to be plasticized.

Evaluation of thermomechanical properties of different types of gutta-percha and Resilon® is important to determine which material to use in different filling techniques. To date, no specific methodology has been described for testing the thermomechanical property of gutta-percha. One method for analysing filling materials was proposed by Tanomaru-Filho *et al.* (2007a), where the thermomechanical property of gutta-percha and Resilon® cones was evaluated. In that study, specimens were tested at a temperature of 70 °C and under a 5.0 kg compression load. The authors described the method as an adaptation of the American Dental Association (ADA) specification no. 57 for endodontic sealing materials. However, in the same study, they mentioned that further guidelines should be established regarding the ideal temperatures and compression loads to be applied when analysing the thermoplasticity of gutta-percha or Resilon® cones. Thus, the influence of temperature and compression suitable for evaluation of thermomechanical properties of gutta-percha and Resilon is unknown.

The present study aims to analyse the thermomechanical properties of gutta-percha and Resilon® cones at different temperatures and under several compression loads, with the purpose of establishing guidelines to be adopted in future studies of these materials.

Materials and methods

Samples

Two hundred and seventy specimens were fabricated, 90 for each material: conventional gutta-percha (GPC, Endopoints Indústria e Comércio Ltda. Paraíba do Sul, RJ, Brazil), thermoplastic gutta-percha (GTP, Endopoints Indústria e Comércio Ltda. Paraíba do Sul, RJ, Brazil) and Resilon® (RE, Pentron Clinical Technologies, Wallingford, CT, USA). Standardized specimens measuring 10 mm in diameter and 1.5 mm in height were divided into three groups ($n = 30$): A, B and C, according to the testing temperature (50, 60 or 70 °C). Then, each group was divided into three subgroups ($n = 10$): according to the compression load to be applied (1.0, 3.0 or 5.0 kg, respectively).

For fabrication of the samples, materials were heated at 70 °C for 60 s, in a thermometer-controlled water bath (Righetto e Cia., Campinas, SP, Brazil), then placed in standardized moulds that consisted of a standard metal ring with an inner diameter of 10 mm and a thickness of 1.5 mm. The moulds containing the heated materials were placed between two glass slabs, which were then compressed under a constant and controlled force of 5.0 N for 1 min. After removal from the mould, excess material was excised, using a sharp blade at the outer edges of the disc, and the dimension of each specimen was checked using a digital micrometer with 0.01-mm precision (Mitutoyu, Suzano, SP, Brazil). The samples were kept at 25 °C ± 2 °C for 24 h. The initial image of each specimen was photographed using a digital camera (Nikon D80, Nikon Corporation, Tokyo, Japan). After that, the specimens were placed for 1 min in water bath (Righetto e Cia., Campinas, SP, Brazil) set at the different evaluation temperatures (50, 60 or 70 °C).

Each specimen was then placed between two glass slabs and subjected to a load of 1.0, 3.0 or 5.0 kg, according to each subgroup, for 2 min. The post-compression images were photographed, and image processing was undertaken to regularize the image allowing a clear view of the outer boundary of the specimen using the Adobe Photoshop 6.0 software (Adobe Systems Inc, San Jose, CA, USA) and imported into the Image Tool software (UTHSCSA Image Tool for Windows version 3.0, San Antonio, TX, USA) for comparison between the initial and final areas (in mm^2) of each sample. A millimetre ruler was photographed beside the specimen and served as a calibration parameter for the Image Tool program during the measurement. The resulting area was obtained by the demarcation of points, circumscribing the outer edge of the image of the specimen. Flow property was determined in mm^2 by subtracting the final (after compression – Fig. 1) area of each specimen from its original area (before compression – Fig. 2).

Analysis of the results

Data were tabulated and subjected to ANOVA ($\alpha = 0.05$) to evaluate the effect of the three variables (material, temperature and compression load), as well as their interaction, on the flow values.

After ANOVA, the Student-Newman-Keuls (SNK) test, also at 5.0% significance, was applied to complement the three-way analysis of variance. In addition, to test for a possible correlation amongst the results of the

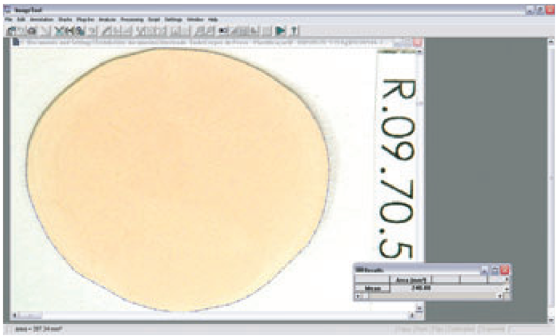

Figure 1 Image of the demarcation area of the specimen as seen in the Image Tool software after the compression test.

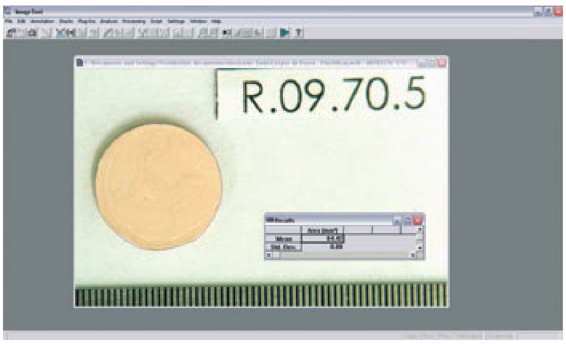

Figure 2 Image of the demarcation area of the specimen as seen in the Image Tool software before the compression test.

materials under different conditions of temperature × load, linear correlation coefficients (r) were calculated.

Results

The means and standard deviations of the differences between the final and initial areas for each material evaluated are shown in Table 1. Generally, higher flow values were detected when materials were subjected to a compression load of 5.0 kg under 70 °C, showing statistically significant difference from the other subgroups ($P < 0.05$). For thermoplastic gutta-percha and Resilon®, compression loads of 5.0 kg at 60 °C, as well as 3.0 kg at 70 °C, also resulted in significantly higher flow values than those observed in the other subgroups ($P < 0.05$).

Regression analysis between the results of the materials under the different experimental conditions showed a poor fit with the linear function. Therefore, poor linear correlations were observed amongst the results of the analysed materials. The mean linear regression coefficients (r) under the different temperature × load conditions (50 °C × 1.0, 3.0 or 5.0 kg; 60 °C × 1.0, 3.0 or 5.0 kg and 70 °C × 1.0, 3.0 or 5.0 kg) were, respectively, 0.68, 0.31, 0.24; 0.28, 0.26, 0.04 and 0.43, 0.15, 0.19.

Discussion

The thermomechanical properties of gutta-percha and Resilon® have been analysed in previous studies with similar methodology, using compression loads of 5.0 kg at 70 °C (Tanomaru-Filho *et al.* 2007a,b). The thermomechanical property of materials is indirectly evaluated by subtracting the final from the initial areas of samples subjected to compression at a certain temperature. The present study was designed to determine whether lower temperatures (50 and 60 °C) and compression loads (1.0 and 3.0 kg) would significantly alter the performance of root canal filling materials in terms of flow. The null hypothesis is that different temperatures and compression loads did not influence the thermomechanical properties of root filling materials.

Table 1 Mean flow area after thermoplasticity values (mm^2) and standard deviations (±) under different experimental conditions

	Temperature	1 kg	3 kg	5 kg
Resilon®	50 °C	2.277 (0.52) A^{a}	2.121 (0.58) A^{a}	2.390 (1.00) A^{a}
	60 °C	5.201 (0.57) A^{a}	11.013 (1.89) A^{ab}	133.926 (9.39) B^{e}
	70 °C	155.166 (17.86) A^{c}	224.313 (27.83) B^{e}	321.821 (35.75) C^{f}
GCO	50 °C	2.546 (0.86) A^{a}	2.618 (0.61) A^{a}	2.469 (0.58) A^{a}
	60 °C	5.343 (1.42) A^{a}	8.475 (1.21) A^{ab}	14.526 (1.65) A^{a}
	70 °C	7.311 (1.42) A^{a}	18.663 (0.75) A^{bc}	76.843 (4.98) B^{c}
GTP	50 °C	5.209 (1.40) A^{a}	5.118 (0.91) A^{ab}	6.258 (1.22) A^{a}
	60 °C	15.824 (1.91) A^{a,b}	26.981 (2.32) A^{c}	55.074 (2.86) B^{b}
	70 °C	21.223 (1.76) A^{b}	45.575 (2.79) B^{d}	98.788 (11.69) C^{d}

GTP, thermoplastic gutta-percha.
Identical upper case letters in the same row and identical lower case letters in the same column indicate statistically significant values ($P \leq 0.05$).

Considering that the ADA does not currently have specific norms for the evaluation of root filling materials (aside from endodontic sealers), the aim of this study was to establish specific guidelines to be followed when evaluating the flow of different endodontic materials.

The results from the present study indicate that solid materials can be evaluated by the method used: at 70 °C and under a compression load of 5.0 kg, the different samples showed significant differences in area, allowing comparison between the materials. As the results under the different experimental conditions were poorly correlated, few differences were observed when the flow of the materials was compared. In addition, considering the lower regression coefficients observed, the results of the materials could not be predictable from one to the other. Therefore, it is suitable to select parameters of temperature and load in which the differences amongst the results of thermoplasticity of the materials could be more evident.

When testing the heated materials, the relationships between temperature (°C), load (kg) and differences in area before and after compression (mm^2) were observed. Although the results demonstrated that testing with a load of 5.0 kg at 60 °C promoted significant area changes in the materials analysed, when a 5.0 kg load was applied at 70 °C, greater changes were observed and allowed better comparisons between the subgroups. The evaluated materials were kept in water at 70 °C for 60 s according to previous studies (Tanomaru-Filho *et al.* 2007a,b).

The results demonstrated that all three materials evaluated (conventional gutta-percha, thermoplastic gutta-percha and Resilon®) have thermoplastic ability. Resilon® showed significant thermoplastic ability at 70 °C, even under lower compression loads (1.0 and 3.0 kg).

Tanomaru-Filho *et al.* (2007a), in a study on the thermoplasticity of different gutta-percha cones and Resilon®, observed that the latter had significantly higher thermoplasticity than both types (conventional and thermoplastic) of gutta-percha. Shipper *et al.* (2004) studied root fillings performed with the Obtura II delivery system and verified a lower incidence of bacterial leakage in canals filled with Resilon/Epiphany® than with gutta-percha using the same delivery system, confirming the good thermomechanical property of Resilon®.

The thermomechanical ability of gutta-percha is directly dependent on its composition; this phenomenon is more evident in its pure form than in industrialized versions (Kolokuris *et al.* 1992). Chemically, gutta-percha is not a single substance but a mixture of several constituents. As the proportions of these constituents in the material are not constant, the properties of gutta-percha are subject to variation. Other studies have also reported that the amount of inorganic substances added during the manufacturing process can affect the thermoplastic properties of gutta-percha cones (Marciano *et al.* 1992, Combe *et al.* 2001, Gurgel-Filho *et al.* 2003).

In the present study, thermoplastic gutta-percha demonstrated significantly higher thermoplastic ability than its conventional counterpart, which suggests the presence of a greater percentage of gutta-percha in its formulation. Results from a previously conducted chemical and radiographic analysis of five brands of gutta-percha showed wide variation in the percentages of zinc oxide (from 84.30 ± 0.50% to 66.50 ± 0.50%) and of gutta-percha (from 14.5 ± 0.70% to 20.4 ± 0.40%) in their formulations (Gurgel-Filho *et al.* 2003). In another analysis of the same commercial brands of gutta-percha cones, Gurgel-Filho *et al.* (2006) verified that those containing greater amounts of gutta-percha were more capable of filling simulated lateral canals by thermatic compaction.

Based on the results, gutta-percha and Resilon® cones require different compression loads and temperatures for evaluation of their thermomechanical properties. For all materials, the greatest flow occurred at 70 °C under a load of 5.0 kg; therefore, these parameters may be adopted when evaluating endodontic filling materials.

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