Original Article

Thermal behavior and viscoelastic properties of gutta-percha used for back-filling the root canal

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Abstract *Background/purpose:* In clinical operations, qualitative differences in the texture and operational feeling of the regular type and soft type back-filled gutta-percha are readily discernible. This study aimed to investigate and compare the thermal behavior and physical properties of the two gutta-percha materials.
Materials and methods: The chemical compositions of regular and soft type Gutta-Percha Obturator® pellets were examined via energy dispersive X-ray spectroscopy. The thermal behaviors of the pellets during heating and cooling were evaluated using a differential scanning calorimeter. Finally, the viscoelastic properties of the two materials during cooling were assessed using a modular compact rheometer.
Results: The soft type gutta-percha contained a greater atomic percentage of zinc than the regular type material. In addition, the soft type gutta-percha exhibited exothermic peaks during cooling, whereas the regular type gutta-percha did not. The two materials exhibited different viscoelastic behaviors under cooling. In particular, the rate of change of the loss factor for the soft type gutta-percha was more than that of the regular type gutta-percha at temperature lower than 80°C.
Conclusion: The soft type gutta-percha underwent significant crystallization during cooling, and therefore exhibited pronounced volume shrinkage. Furthermore, the soft type gutta-percha underwent a greater rate of change in viscoelasticity under cooling than the regular type gutta-percha, and exhibited poorer physical stability. Consequently, in the back-packing procedure, soft type gutta-percha must be compacted more often over time than regular type gutta-percha to ensure the same quality of root canal obturation.

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Introduction

Obturation of the shaped, cleaned and disinfected root canal space is designed to provide an impermeable fluid-tight seal to that precludes microleakage and the potential for reinfection from the oral and apical regions.[1] Amongst the various filling materials available for this purpose, gutta-percha is the most commonly used core material. Historically, the dimensional stability of gutta-percha during the obturation process was generally achieved by applying a lateral compaction technique under isothermal conditions.[2] However, lateral compaction cannot guarantee a homogeneous, three-dimensional distribution of the filling material in the root canal system; often just consisting of a grouping of gutta-percha cones in a sea of sealer/cement.[3] Accordingly, Schilder[4] proposed a vertical compaction technique using warm gutta-percha under non-isothermal conditions. In the proposed method, the transformation of the gutta-percha from the heated state to a homogenous mass was achieved using a series of pluggers, resulting in a more thorough obturation of the shaped canal system under the combined effects of the heat-softened gutta-percha and sealer. However, the dental gutta-percha product is mixture. The composition of commercially available dental gutta-percha consists of 18%–22% gutta-percha, 59%–76% zinc oxide, 1%–4% waxes and resins, and 1%–18% metal sulfates.[5–8] The phase transformation temperatures of dental gutta-percha are affected by molecular weight, purity, compounding, thermal and mechanical history, and degree of crystallinity.[9] Therefore, the glass transition temperature (T_g) of commercial dental gutta-percha is differed from that of pure poly (trans-1,4-polyisoprene). The recent study by Chen et al.[10] indicated that the glass transition temperature of dental gutta-percha is around 40 to 44°C.

The vertical compaction process involves a down-packing operation followed by a reverse filling or back-packing procedures. Chen et al.[10] used an infrared thermography technique to evaluate the continuous change in the gutta-percha temperature during the down-packing process. The results showed that a moldable temperature of apical gutta-percha (44°C) could be achieved by applying two heating and compaction cycles at a distance of 3 mm from the apical foramen. Schilder[3] performed the back-packing process by inserting precut segments of gutta-percha into the root canal. Presently, back-packing is generally performed using a thermoplasticized, gutta-percha injection system. This system is traditionally performed using regular-type gutta-percha. However, the use of soft-type gutta-percha has become common in recent years.[11,12] In clinical operations, qualitative differences in the texture and operational feeling of the two materials are readily discernible. However, the published literature thus far lacks sufficient scientific evidence to qualify and explain these differences.

Therefore, this study aimed to evaluate and compare the composition, thermal behavior and viscoelastic behavior of regular- and soft-type gutta-percha that are used in the back pack procedure under controlled temperature conditions.

Materials and methods

Regular-type and soft-type Gutta-Percha Obturator® materials were purchased from Sure Dent Corporation (Seongnam-si, Korea). All the following analyses were performed three times for each tested material.

Composition analysis

Energy dispersive X-ray spectroscopy was used to determine the qualitative and semi-quantitative analyses of the chemical compositions of the two gutta-percha materials. The analyses were conducted on both the sample surface and the sample cross-section. In both cases, the samples were mounted on aluminum stubs and examined using an SU3500 Scanning Electron Microscope (Hitachi, Tokyo, Japan) fitted with an energy dispersive X-ray spectrometer.

Thermal behavior analysis

The phase transition temperatures and thermal behaviors of the two gutta-percha materials were evaluated using a differential scanning calorimeter (DSC 4000, PerkinElmer, Waltham, MA). Two heating and cooling cycles were implemented as follows:

a) Heating followed by slow cooling
   1. Temperature increased from 25°C to 70°C with heating rate of 1°C/minute.
   2. Temperature increased from 70°C to 130°C with heating rate of 5°C/minute.
   3. Temperature maintained at 130°C for 10 min.
   4. Temperature decreased from 130°C to 25°C with slow cooling rate of 5°C/minute.
b) Heating followed by rapid cooling
   1. Temperature increased from 25°C to 70°C with heating rate of 1°C/minute.
   2. Temperature increased from 70°C to 130°C with heating rate of 5°C/minute.
   3. Temperature maintained at 130°C for 10 min.
   4. Temperature decreased from 130°C to 25°C with rapid cooling rate of 20°C/minute.

Viscoelastic behavior analysis

The viscoelastic behavior of the two gutta-percha materials was analyzed during cooling using a modular compact

rheometer (MCR 102, Anton Paar GmbH, Graz, Austria). For both materials, the viscoelastic behavior was examined using oscillatory tests performed with a strain amplitude of 1% and an oscillation frequency of 2 Hz as the temperature was reduced from 150°C to 37°C at a rate of 1°C/minute. The viscoelastic behavior of the samples was quantified in terms of the following four properties:[13]

1. **Storage modulus G':** a measure of the stored deformation energy of the test sample. G' represents the elastic behavior of the material, and is given by

$$G' = \left(\frac{\tau_A}{\gamma_A}\right)\cos\delta$$

where τ_A is the shear stress amplitude, γ_A is the shear strain amplitude, and δ is the phase shift angle.

2. **Loss modulus G″:** a measure of the lost deformation energy of the test sample. G″ represents the viscous behavior of the material, and is given by

$$G'' = \left(\frac{\tau_A}{\gamma_A}\right)\sin\delta$$

where τ_A is the shear stress amplitude, γ_A is the shear strain amplitude, and δ is the phase shift angle.

3. **Loss factor tanδ:** the quotient of the lost deformation energy to the stored deformation energy. $\tan\delta$ represents the ratio of the viscous portion of the viscoelastic behavior of the material to the elastic portion, and is given by

$$\tan\delta = \frac{G''}{G'}$$

where G'' is the loss modulus and G' is the storage modulus.

4. **Complex viscosity η*:** the viscoelastic flow resistance of the material for practical applications. η^* is formulated as

$$\eta^* = \frac{\tau_A}{\dot{\gamma}_A} \text{ and } |\eta^*| = \frac{|G^*|}{\omega} = \frac{\sqrt{(G')^2 + (G'')^2}}{\omega}$$

where τ_A is the shear stress amplitude, $\dot{\gamma}_A$ is the rate of change of the shear strain amplitude, G^* is the complex shear modulus and ω is the angular frequency.

Results

Composition analysis

Energy dispersive X-ray spectroscopy results indicated that there was a notable difference in the chemical compositions of the cross-sectional, gutta-percha samples and surface area samples, respectively (Tables 1 and 2). In particular, for both materials, the cross-sectional samples consisted mainly of C, O and Zn, but also contained smaller amounts of F, Mg, Si and Pt. By contrast, the surface samples contained only C, O and Zn, i.e., no other elements were detected; even in trace amounts. Furthermore, the atomic percentage of Zn in the soft-type gutta-percha

(cross-section: 18.50; surface: 1.55%) was higher than that in the regular-type gutta-percha (cross-section: 13.21; surface: 0.90%).

Thermal behavior analysis

Figs. 1 and 2 show the experimental thermograms obtained for the regular-type and soft-type gutta-percha in the heating cycles with slow and rapid cooling procedures, respectively. For both heating cycles, the two materials underwent glass transition at temperatures of 40°C and 38°C, respectively, as the temperature slowly increased from 25°C to 70°C. Moreover, both materials exhibited two phase transition regions. For the regular-type gutta-percha material, the first phase transition region extended from 40—49°C, while the second region extended from 52—59°C. For the soft-type gutta-percha, the first phase transition region lay between 38°C and 49°C, whilst the second region lay between 50°C and 58°C. The peak temperature values in the phase transitions of the regular-type gutta-percha (i.e., 46°C and 56°C) were higher than those of the soft-type gutta-percha (i.e., 43°C and 55°C).

No obvious changes in the heat flow were observed for either material at temperatures higher than 120°C during the heating process. Furthermore, no exothermic peaks were observed in either cooling process (i.e., slow cooling (Fig. 1) or rapid cooling (Fig. 2)) for the regular-type gutta-percha. However, the soft-type gutta-percha, exhibited prominent exothermic peaks that were seen at temperatures of 52°C and 32°C, respectively, in the slow cooling process and 52°C in the rapid cooling process.

Viscoelastic behavior analysis

Fig. 3 shows the variation of the four viscoelastic parameters of the regular-type and soft-type gutta-percha during cooling from 150°C to 37°C. For both materials, the storage modulus G', loss modulus G″ and complex viscosity η* increased with decreasing temperature. The rate of change of G', G″ and η* with decreasing temperature was approximately the same for both materials as the temperature decreased from 150°C to 60°C. However, as the temperature was further decreased to 40°C, the three parameters increased relatively slowly for the regular-type gutta-percha, but more dramatically for the soft-type gutta-percha.

Table 1 Composition of regular-type and soft-type gutta-percha (GP) cross-section.

Chemical element	Regular type-GP		Soft type-GP	
	Wt %	At %	Wt %	At %
C	45.81	64.39	46.57	61.93
O	17.68	18.66	16.82	16.80
F	0.66	0.59	1.23	1.03
Zn	17.99	13.21	26.62	18.50
Mg	1.53	1.06	0.99	0.65
Si	1.32	0.79	0.93	0.53
Pt	15.01	1.30	6.84	0.56

Wt %: weight percentage, At %: atomic percentage.

Table 2 Composition of regular-type and soft-type gutta-percha (GP) surface area.

| Chemical element	Regular-type GP		Soft-type GP	
	Wt %	At %	Wt %	At %
C	79.22	85.88	78.05	86.51
O	16.24	13.21	14.35	11.94
Zn	4.54	0.9	7.60	1.55

Wt %: weight percentage, At %: atomic percentage.

For both materials, all three parameters increased extremely rapidly as the samples underwent final cooling from 40°C to 37°C. Comparing the viscoelastic parameters of the regular-type gutta-percha and the soft-type gutta-percha, respectively, the regular-type gutta-percha had higher values of G′, G″ η* at temperatures of 50°C to 150°C, but lower values at temperatures less than 50°C.

For the regular-type gutta-percha, the loss factor declined gradually and continuously as the temperature reduced from 150°C to 40°C. However, for the soft-type gutta-percha, the loss factor reduced with a decreasing temperature until around 110°C, but then increased to approximately 1.0 at a temperature of 80°C before reducing sharply as the temperature further decreased to 40°C. For both materials, the loss factor decreased extremely rapidly as the temperature reduced from 40°C to 37°C.

Discussion

The manufacturer's specification details provide very little information regarding the regular-type and soft-type gutta-percha materials used in the present study. It can be inferred that the two materials have different viscosities
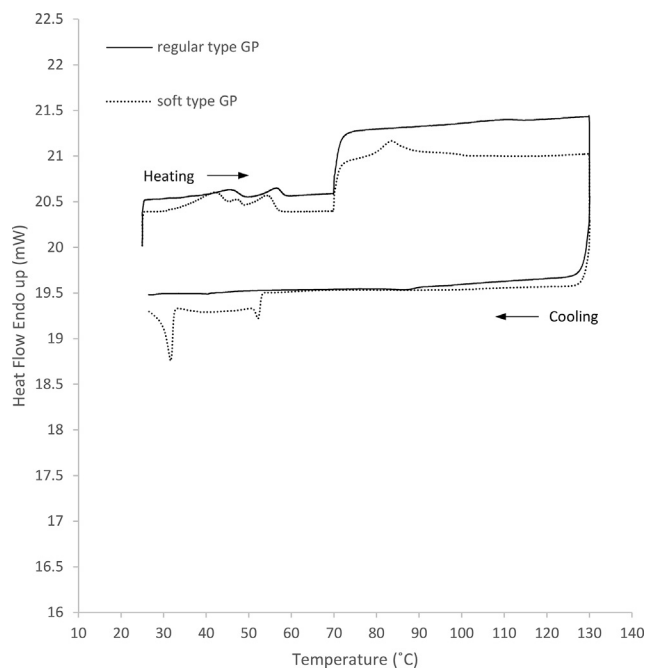
Figure 1 Thermograms of regular-type and soft-type gutta-percha with slow cooling procedure.
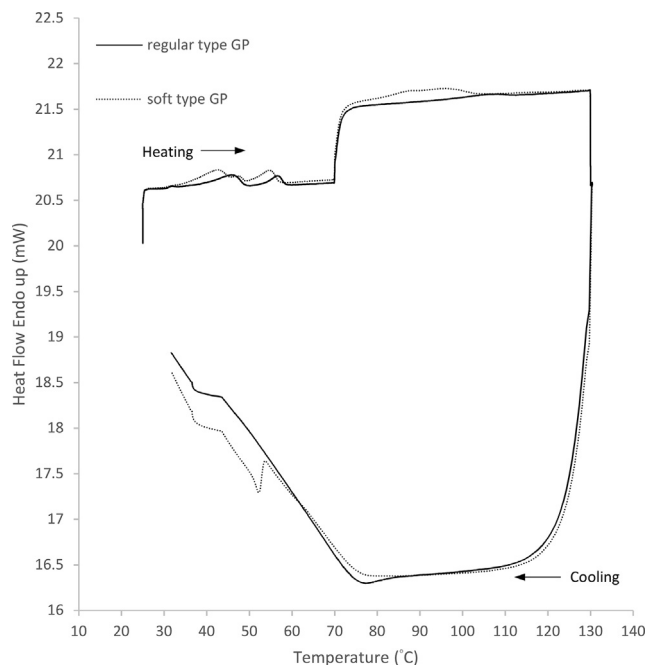
Figure 2 Thermograms of regular-type and soft-type gutta-percha with rapid cooling procedure.

since the regular-type gutta-percha is generally injected with a 23-gauge needle, while the soft-type material is injected with a 25-gauge needle. However, the literature lacks detailed scientific insights into the thermal behaviors and viscoelastic properties of the two materials. The present results have shown that there exist significant differences in the chemical compositions, thermal behaviors and viscoelastic properties of regular-type and soft-type gutta-percha material, and these differences should be taken into account in performing the back-packing process in root canal obturation.

For the surface area samples, the atomic percentage of each element in the regular-type gutta-percha was similar to that in the soft-type gutta-percha (Table 2). In other words, both samples had similar surface layers. By contrast, for the cross-sectional samples, the atomic percentage of zinc in the regular-type gutta-percha was lower than that in the soft-type gutta-percha (Table 1). Zinc oxide has high thermal conductivity.[14] Hence, it can be inferred that the heated temperature of soft-type gutta-percha may be higher than that of regular-type gutta-percha under the same heat energy input.

According to Combe et al.;[15] Ferrante et al.;[16] and Maniglia-Ferreira,[8] the heating procedure of this experiment was divided into two steps. The first step used a slow heating rate (1°C/minute) to detect all the peaks in phase transition of the tested material. Based on the study by Schilder et al.,[10] the transformation temperatures of dental gutta-percha are 42—49°C. for the beta to alpha transition and 53 to 59°C. for the alpha to amorphous transition. Therefore, in the first step, the focus was on the temperature range from room temperature (25°C) to 70°C to evaluate the tested commercial dental gutta-percha materials. The second heating step (heating rate: 5°C/minute), followed the previous study designs of Combe et al.;[15]
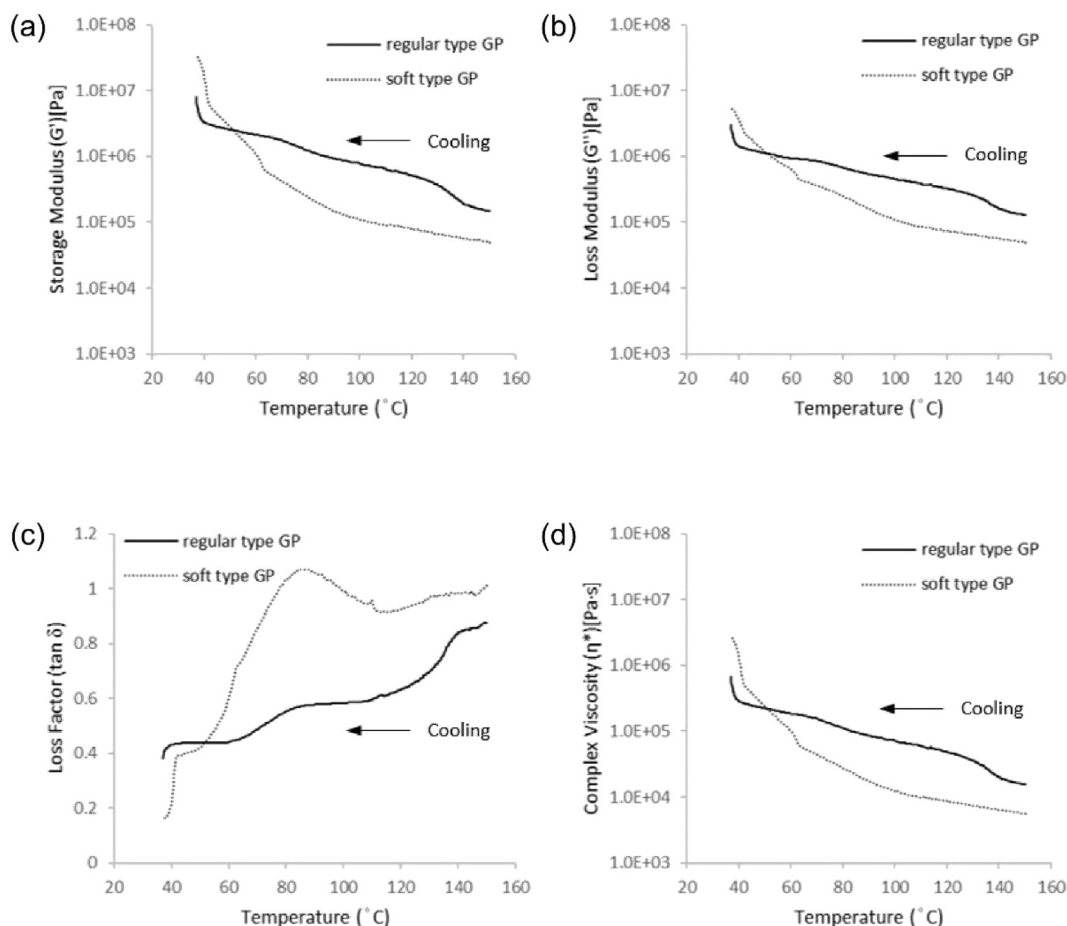
Figure 3 Viscoelastic parameters of regular-type and soft-type gutta-percha during slow cooling. (a) Storage modulus G'; (b) loss modulus G''; (c) loss factor tan δ; (d) complex viscosity η^*.

Ferrante et al.;[16] and Maniglia-Ferreira.[8] According to a recent study by Hsu et al.,[17] the real temperature of heated dental gutta-percha extruded from commercial endodontic heat guns (B&L Beta® system, B&L BioTech, Fairfax, VA; Obtura III Max® system, Obtura Spartan, Algonquin, IL) is lower than 130°C. Therefore, 130°C as set as the final temperature in the end of the second heating step.

Previous studies on the phase transition temperature and weight loss characteristics of gutta-percha during the heating cycle simply regard the cooling procedure as a pre-set operation for the following cycle.[8,16,18,19] However, in the present study, the cooling process was recognized as a key stage in the back-packing process and was afforded more attention accordingly. The present study has considered both a slow cooling procedure (5°C/minute) and a rapid cooling procedure (20°C/minute). The former was intended to yield fine data undetectable during rapid cooling (Fig. 1), while the latter procedure was designed to approximate the cooling process under actual clinical conditions (Fig. 2). For the regular-type gutta-percha material, no obvious exothermic peaks were observed in the thermograms during either of the two cooling processes. This suggests that the material underwent no apparent reaction during cooling. In other words, it can be inferred that regular-type gutta-percha maintains a dominant amorphous

non-crystalline structure over the cooling temperature range of 150°C to 25°C. By contrast, the soft-type gutta-percha exhibited exothermic peaks in both cooling processes. The present findings for the heat flow behavior of soft-type gutta-percha during cooling are consistent with those reported in a previous study on the thermal properties of gutta-percha.[20] In general, endothermic and exothermic peaks represent the onset of phase transition. For example, a previous study on the crystal structure of gutta-percha reported that the exothermic peaks of gutta-percha correspond to a crystallization of the gutta-percha from an amorphous structure to a beta-form structure.[21] Therefore, the structure of soft-type gutta-percha has a dominant crystalline component. As mentioned previously, soft-type gutta-percha underwent significant crystallization during cooling. Hence, comparing the two materials, the soft-type gutta-percha may exhibit more obvious volume shrinkage during the cooling process. The careful manipulation and control of soft-type gutta-percha during the compaction stage of the clinical back-packing procedure is of great importance.

For both gutta-percha materials, the loss factor generally reduced with a reducing temperature (Fig. 3(c)). That is, the rate of increase of modulus G'' with decreasing temperature was less than that of the storage modulus G'.

In other words, both materials exhibited a dominant elastic behavior and a certain rigidity. This finding in consistent with that of Chang et al.[22]

Schilder[4] used a plugger to compact the gutta-percha during its cooling to overcome the problem of volumetric shrinkage. The present results suggest that deformed gutta-percha previously compacted may partially return to its original shape once the vertical force is removed due to its inherent elastic behavior. However, further investigation is required to ascertain the exact relationship between the amount of rebound and the physical shrinkage of gutta-percha during clinical manipulation. The results presented in Fig. 3(c) have additionally shown that the rate of change of the loss factor for soft-type gutta-percha is more than that for regular-type gutta-percha at cooling temperatures lower than 80°C. This finding suggests that the soft-type gutta-percha has relatively more unstable physical properties than regular-type gutta-percha at lower cooling temperatures. In other words, soft-type gutta-percha changes more quickly from a deformable state to a non-deformable state during clinical back-packing. Consequently, soft-type gutta-percha exhibits relatively lower physical stability and may therefore involve a more technique-sensitive back-packing process to ensure a similar quality of the root canal obturation as that obtained using regular-type gutta-percha.

In conclusions, this study identified the existence of distinct differences in the thermal and rheological behaviors of regular-type and soft-type gutta-percha during cooling. These properties affect the handling of the two different materials in clinical practice. In particular, the results suggest that soft-type gutta-percha should be compacted more often over time than regular-type gutta-percha in the back-packing procedure to ensure the quality of the obturation.

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Declaration of Competing Interest

The authors deny any conflict of interest related to this study.

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