Measurement of thermal expansion characteristic of root canal filling materials : Gutta-percha and Resilon

Kyung-A Jeon[1], In-Bog Lee[2], Kwang-Shik Bae[2], Woo-Cheol Lee[2], Seung-Ho Baek[2]*

[1]*Department of Conservative Dentistry, Anam Hospital, Korea University Medical Center*

[2]*Department of Conservative Dentistry, College of Dentistry, Seoul National University*

ABSTRACT

The purpose of this study was to evaluate the thermal expansion characteristics of injectable thermoplasticized gutta-perchas and a Resilon. The materials investigated are Obtura gutta-percha, Diadent gutta-percha, E&Q Gutta-percha Bar and Epiphany (Resilon).

The temperature at the heating chamber orifice of an Obtura II syringe and the extruded gutta-percha from the tip of both 23- and 20-gauge needle was determined using a Digital thermometer. A cylindrical ceramic mold was fabricated for thermal expansion test, which was 27 mm long, with an internal bore diameter of 3 mm and an outer diameter of 10 mm. The mold was filled with each experimental material and barrel ends were closed with two ceramic plunger. The samples in ceramic molds were heated in a dilatometer over the temperature range from 25℃ to 75℃. From the change of specimen length as a function of temperature, the coefficients of thermal expansion were determined.

There was no statistical difference between four materials in the thermal expansion in the range from 35℃ to 55℃ ($p > 0.05$). However, Obtura Gutta-percha showed smaller thermal expansion than Diadent and Metadent ones from 35℃ to 75℃ ($p < 0.05$). The thermal expansion of Epiphany was similar to those of the other gutta-percha groups. [J Kor Acad Cons Dent 31(5):344-351, 2006]

Key words: Thermal expansion, Gutta-percha, Resilon, Dilatometer, Temperature

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Ⅰ. INTRODUCTION

One of the most important objectives of nonsurgical root canal therapy for success is to completely obturate the prepared canal[1]. Several studies have revealed that an incomplete sealing of the canal is one of the most important causes of failure[2,3]. In order to obtain a three-dimensional obturation of root canal systems, various materials have been used. Above all, gutta-percha is still the material most favored by endodontists for the obturation of root canals due to its thermoplastic behavior and its biocompatibility[3-5]. For a long time this material has been used successfully with a variety of nonadhesive root canal sealers for obturation of root canal.

Improving the quality of root canal obturations should logically be accomplished by an alternative to gutta-percha. Resilon (Pentron Clinical Techn-

* *Corresponding Author: **Seung-Ho Baek***

Department of Conservative Dentistry,
College of dentistry, Seoul National university
28-2 Yeongeon-dong Chongno-gu, Seoul, 110-749, Korea
Tel: 82-2-2072-2005 Fax: 82-2-2072-3859
E-mail: shbaek@snu.ac.kr

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ologies, Wallingford, CT, USA), has recently been introduced as an another root canal filling material that has the potential to challenge the use of gutta-percha[6,7]. It is a polyester based thermoplastic polymer which contains a bioactive glass and some radiopaque fillers. It performs like gutta-percha, and has the same handling properties, and for retreatment purposes may be softened with heat or dissolved with solvent like chloroform. Furthermore, it is able to bond to resin-based systems, such as dentin bonding agents and resin-based luting cements, so a monoblock is formed without the gaps typical in gutta-percha filling. Shipper *et al.*[6] evaluated the resistance to bacterial penetration in roots filled with this new material and found that Resilon was superior to gutta-percha groups. They also reported that the Resilon "Monoblock" System was associated with less apical periodontitis, which may be due to its superior resistance to coronal microleakage[8]. Teixiera *et al.*[9] reported that filling the root canal with Resilon increased the *in vitro* resistance to fracture of single-canal extracted teeth. However, Tay *et al.*[10-12] doubted the bonding of Resilon with root canal wall dentin, and reported that Resilon is susceptible to enzymatic hydrolysis[13,14]. In any case there have been a few studies about the properties of this material because it is a relative new one.

Various techniques have been introduced for hermetic root canal sealing and all of these have been clinically successful[3,15,16]. The lateral condensation technique which has been used for a long time successfully takes advantage of the dimensional stability of gutta-percha. However, it cannot fill lateral canals and makes the obturation mass held together only by friction and the sealer. Homogeneity occurs only at the point where the coronal excess is removed with a hot instrument. The warm vertical condensation technique provides a homogenous filling and greater density obtained in the apical portion of the canal using gutta-percha in a plastic state without solvent[3,17]. Above all, continuous wave of condensation technique using system B and Obtura II makes warm vertical condensation more easier and faster, so it has been used increasingly by many clinicians today. However, every root canal filling materials show dimensional change by temperature change[18].

Warm vertical condensation technique must have needs of some heat, it means that there would be some dimensional change of canal filling material during canal obturation procedure. Especially it is expected that thermoplasicized injectable instruments like Obtura II show more dimensional change because they extrude very hot filling material. As gutta-percha doesn't have the ability to bond to dentin wall of a root canal, it is possible to doubt that canal leakage is started if some dimensional shrinkage would happen in the canal space after canal obturation was completed[19]. Although several researchers reported thermal expansion properties of gutta-percha[18-20], there is still insufficient information available on the effect of heat on the thermal expansion properties of injectable thermoplasticized canal filling materials including Resilon, newly introduced material. The comparison of thermal expansion properties of these materials are valuable.

The purpose of this study was to evaluate the thermal expansion characteristics of injectable thermoplasticized gutta-perchas and Resilon.

Ⅱ. MATERIALS AND METHODS

Materials

The materials investigated are listed in Table 1. Epiphany is Resilon, and the others are injectable thermoplasticized gutta-perchas.

Measurement of the temperature of extruded filling material

Each filling materials were loaded in an Obtura II injection gun (Spartan, Fenton, MO, USA) and 23- or 20-gauge application needle was connected.

The temperature of the extruded gutta-percha through either 23-G or 20-G was determined using a thermocouple connected to a Digital mul-

Table 1. Root canal filling materials used in this study

Product Name	Code	Manufacturer
Gutta-percha pellets- box 100	Obtu	Obtura Spartan, Fenton, MO, USA
Gutta-percha Obturator	Dia	Diadent group international Inc., Chong-Ju, Korea
E&Q Gutta-percha Bar	Meta	Meta Biomed Co., Ltd, Chong-Ju, Korea
Epiphany pellet	Epi	Pentron Clinical Technologies LLC, CT, USA

timeter (Fluke 179, Fluke Corp., WA, USA). The materials were extruded onto the tip of the thermocouple at room temperature (22 to 23℃). The thermocouple was thoroughly cleaned with alcohol cotton after each measurement. The extrusion needle and hub were removed and the thermocouple was placed into the orifice of the heating chamber of the Obtura II. The temperature of the heating chamber of the Obtura II was also measured. The whole procedure mentioned above was repeated for the setting temperature of Obtura II at 100℃, 150℃, 180℃ and 200℃. The maximum temperature when the materials were extruded was recorded. Each measurement was repeated three times.

Measurement of the thermal dimensional change

A cylindrical ceramic mold was made for thermal expansion test, which was 27 mm long, with an internal bore diameter of 3 mm and an outer diameter of 10 mm. And the ceramic plunger with a diameter of 3 mm was also made. The mold was filled with a material of each groups, and both barrel ends were sealed with two ceramic plungers.

The samples in the ceramic molds were loaded in a dilatometer (Thermal Dilatometer, DIL 402C, TASC 414/3A Controller, Netzche, Germany). Thermal expansion curves were obtained at a temperature range from 25℃ to 75℃ with a heating rate of 1℃/min under a load of 30N. From dimensional change of samples as a function of temperature, the coefficients of thermal expansion were determined at the range 35 - 45℃, 45 - 55 ℃, and 55 - 75℃. The measurement was repeated six times for each material. In addition, the percentages of volume change at the temperature range of 35 - 55℃ and 35 - 75℃ were calculated by the following equation.

(λ : the percentage of volume change, L_o : sample initial length, dL_A, dL_B : the length change of sample at temperature A, B)

$$\lambda = \frac{dL_B - dL_A}{L_o} \times 100\ (\%)$$

The data were analyzed by the One-way ANOVA and the Tukey's post-hoc test at 95% significant level using a statistical software (SPSS Release 12.0 for Windows, SPSS, Chicago, USA).

Ⅲ. RESULTS

Measurement of the temperature of extruded filling material

The mean temperatures of the extruded materials and heating chamber at different setting temperature are shown in Table 2. The measured temperatures increased in the order of 23-G, 20-G needle, and the heating chamber for all setting temperature of Obtura II, and were similar among the materials (Table 2).

The extruded temperature of the Obtura with a 23-G at the setting temperature of 200℃, which is the temperature recommended by the manufacturer, was about 76℃.

Measurement of the thermal dimensional change

There was almost no dimensional change of materials below 45 ℃. And at the temperature range of 45℃ - 55℃ the volume of materials increased rapidly, then slightly increased at over

55℃ (Figure 1). The coefficients of thermal expansion were calculated in the range of 35 - 45 ℃, 45 - 55℃, and 55 - 75℃ (Table 3). The coefficients at the range of 45 - 55℃ were larger than those of other temperature range, and Epiphany showed some larger coefficient than other materials at the range of 45 - 55℃ ($603 \pm 113.8 \times 10^{-6}$). However, there was no statistically differences between the materials.

The percentages of dimensional change between materials at the temperature range of 25℃ - 75℃ were shown in Figure 1 and 2. There was no statistical difference between four materials at the range from 35℃ to 55℃. However, Obtura gutta-percha showed smaller thermal expansion than Diadent and Metadent ones from 35℃ to 75℃ ($p < 0.05$, Table 4).

Table 2. Temperature of each materials, when extruded at the tip of application needles and existed in the heating chamber (℃)

	20G	23G	Body	20G	23G	Body
		100℃			150℃	
Obtu	48.27 (3.36)	43.77 (0.31)	86.57 (2.63)	67.27 (4.52)	54.90 (1.65)	126.87 (3.78)
Dia	46.67 (0.47)	43.33 (0.65)	86.10 (1.14)	60.43 (2.58)	56.30 (1.06)	124.20 (2.17)
Meta	48.77 (2.14)	46.33 (1.04)	88.73 (0.75)	69.33 (1.53)	59.67 (5.69)	125.10 (1.93)
Epi	44.17 (2.02)	43.67 (1.53)	83.40 (0.66)	57.73 (2.00)	51.60 (1.85)	121.33 (1.26)
		180℃			200℃	
Obtu	80.93 (7.49)	65.67 (3.22)	152.17 (2.75)	92.37 (4.12)	76.33 (3.79)	172.50 (2.29)
Dia	81.67 (4.16)	64.63 (3.51)	147.27 (2.55)	95.00 (3.00)	79.73 (4.05)	167.40 (0.53)
Meta	85.53 (1.29)	70.13 (6.86)	149.60 (4.29)	93.17 (1.26)	83.63 (7.16)	166.17 (2.02)
Epi	71.70 (3.34)	64.77 (1.57)	144.5 (4.44)	87.33 (2.52)	75.00 (2.17)	159.90 (0.85)

† The numbers in paranthesis are S.D.

Table 3. The linear thermal expansion coefficient (10^{-6}/℃) of each tested material at different temperature range

Temperature range (℃)	Obtu	Dia	Meta	Epi
35 - 45	16.1 (18.8)	8.9 (5.83)	30.6 (42)	21.1 (15.3)
45 - 55	494.3 (105.9)	464.0 (159.8)	414.0 (185.2)	603.0 (113.8)
55 - 75	156.0 (7.9)	252.7 (50.5)	281.7 (92.7)	139.0 (38.5)

† The numbers in parentheses are S.D.

Table 4. The percentage of thermal expansion of each material at different temperature range (unit: Vol %)

Temperature range (℃)	Obtu	Dia	Meta	Epi
35 - 55	1.53 (0.48)	1.42 (0.47)	1.33 (0.64)	1.87 (0.36)
35 - 75	2.47 (0.35)*	2.93 (0.18)**	3.02 (0.29)**	2.71 (0.31)

† The numbers in parentheses are S.D.

‡ There were statistical differences between * and **.

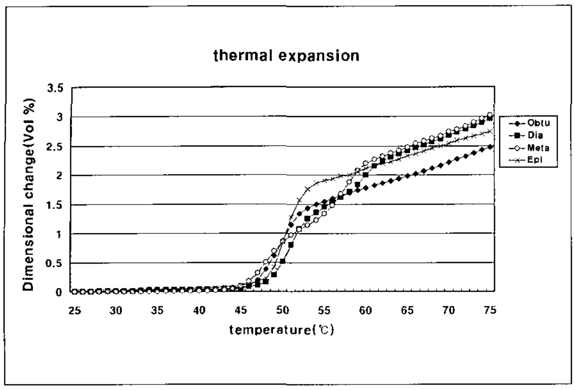

Figure 1. The mean dimensional volume change of four materials as a function of temperature at the range of 25℃ - 75℃.

Ⅳ. DISCUSSION

Resilon, a thermoplastic synthetic polymer-based root canal filling material, has recently been introduced. It has the similar properties of gutta-percha, so it is considered as the potential substitute for gutta-percha as a root filling material. Based on synthetic polymers of polyester, it contains bioactive glass, bismuth oxychloride, and barium sulfate. The overall filler contents is approximately 65% by weight. The thermoplasticity of Resilon is attributed to the incorporation of polycarprolactone, a synthetic, biodegradable, semi-crystalline aliphatic polyester[13]. And its bonding ability with resin cement and self-etching primers is derived from the inclusion into the resin of difunctional methacryloxy groups.

The composition of gutta-percha cones is approximately 19% to 22% gutta-percha, 59% to 75% zinc oxide, with the remaining small percentages of the components various waxes, coloring agents, anti-oxidants, and metallic salts. The particular percentage of the components vary by manufacturer, with resultant variations in the several physical properties of the individual cones primarily because of the gutta-percha and zinc oxide percentages[3,21-23].

In spite of different chemical composition between gutta-percha and Resilon, they showed similar thermal expansion in this study and there was no statistical significant difference between them. This result means that Resilon have a similar thermal expansion property to gutta-percha which has been widely used for a long time as a root canal filling material, and can substitute it successfully at the point of thermal expansion characteristic like other favorable properties (radiopacity, plasticity and ability to flow at elevated temperatures, solubility in certain organic solvents etc).

Previous measurement of thermal volumetric change of dental gutta-percha has been performed dilatometrically with mercury. This technique has its advantages and disadvantages. Mercury based dilatometer has potential health wizards, is very sensitive to thermal fluctuations and very labour intensive[24,25]. In this study we used a ceramic mold which has a relatively very low thermal expansion coefficient. Generally the thermal expansion coefficient of ceramic is 8×10^{-6}/℃ (20 - 50℃)[26], and it seems to be almost zero compared with that of gutta-percha and Resilon. The dilatometer used in this study can measure the dimensional change over temperature by micrometer unit.

In this study, the thermal coefficient of materials were larger than those of other studies[20,27]. The mean value of this study was more double than the one reported by Cohen *et al.*[20]. In addition, Tsukada *et al.*[19] showed that the percentage of specific volume change at cooling between 75 and 24℃ was 4-5%, which was much larger than our results although the equation for calculation and experimental condition were slight different. These differences are possibly due to the different materials used in experiment or sensitivities of the different thermal expansion recording systems. However they are worthwhile from a standpoint of comparing to each materials under the same condition. It is possible to say that the thermal expansion of Resilon was not different from the other gutta-percha materials.

The results of this study seem to indicate that concerns about the high temperature of thermoplasticized injectable gutta-percha are not justified. When the temperature was measured, there was evidently a rapid continuous heat loss from the time the heated gutta-percha leaved the injection needle and reached the thermocouple due to the effect of room temperature air causing a rapid cooling of the exposed experimental materials. Weller and Koch[28] measured the temperature produced with the Obtura II heated gutta-percha system and found intracanal temperatures ranging from 40℃ to 57℃ and external root surface temperatures ranging from 36℃ to 42℃. Sweatman et al[29] reported that with the Obtura II, the lowest mean internal temperature change was 5.22℃ at the 0 mm level, whereas the highest mean internal temperature change was 26.63℃ at 6 mm level. We did not measure the intracanal temperature during the root canal filling procedure, postulated that the intracanal temperature range during the canal filling procedure was 35-55℃ according to previous studies[28-33].

The thermal expansion of Obtura group appeared smaller than those of Diadent and Metadent groups ($p < 0.05$) and showed no difference with that of Resilon group at the range of 35℃-75℃. However there was no statistical difference between four material groups at the range of 35℃-55℃, the clinical meaningful temperature.

It is unfortunate that tests could not be carried out on the material as supplied by the manufacturer, and only during the heating procedure. Tsukada G *et al.*[19] showed that, on cooling, gutta-percha will shrink to its original length, although not at the same rate as it expanded on heating. However it may be assumed that the shrinkage on cooling from working temperature to intracanal temperature will be of an equal magnitude to the expansion over that temperature range. And, it is reasonable to assume that the shrinkage occurs in the range from the softening temperature down to intracanal temperature (55-35℃).

Schilder *et al.*[34] identified two major transitions, or phase changes, when dental gutta-percha was heated. They concluded that, on heating material, two transformations occurred at temperatures between 42-49℃ and 53-58℃. These appear to agree with the results calculated for phase-change temperature in another studies[20]. We found that there was the change of thermal expansion rate at near 45℃ and 55℃, a fiducial point, it suggested that the phase transition occurred at these temperatures. During the setting process, the crystallization of gutta-percha is thought to adversely affect the sealing ability in the root canal space. The root canal treatment failure is mainly caused by incomplete sealing of the root canal, and accordingly it is important to obturate the root canal hermetically. A marked specific volume change was observed at the temperature change of 45℃ to 55℃ in this study. On the basis of our findings, it seems to be better that thermoplasticized canal filling material is condensed with plugger until the intracanal temperature decreased at least under the temperature of 45℃ during the back-filling procedure.

Ⅴ. CONCLUSION

1. The temperature of extruded gutta-percha was increased in order of 23 G, 20 G, and heating chamber for all setting temperature.
2. There was almost no dimensional change of materials below 45℃. At the temperature range of 45℃-55℃, the volume of materials increased very rapidly, then slightly increased at over 60℃.
3. There was no statistical significant difference between four materials in the thermal expansion at the range from 35℃ to 55℃ ($p > 0.05$). However, Obtura gutta-pecha showed smaller thermal expansion than Diadent and Metadent gutta-perchas at the range from 35℃ to 75℃ ($p < 0.05$).
4. The thermal expansion of Resilon is similar to those of gutta-perchas ($p > 0.05$).

Therefore this results showed that Resilon had a potential to substitute for gutta-percha from a thermal expansion characteristic point of view. However there still remain many subjects to investigate this material compared with gutta-percha. More comparison about thermal properties of Resilon and Gutta-percha will be needed in the future.

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국문초록

수 종의 근관충전재의 열팽창 특성 측정 : Gutta-percha와 Resilon

전경아[1] · 이인복[2] · 배광식[2] · 이우철[2] · 백승호[2]*

[1]고려대학교 안암의료원 치과보존과, [2]서울대학교 치과대학 보존학교실

근관충전재로 사용되는 gutta-percha의 대체재로서 Resilon이라는 bioactive glass와 약간의 방사선 불투과성 성분을 포함하는 열가소성 고분자가 최근 소개되었다. 본 연구에서는 열연화 주입식 gutta-percha와 Resilon의 열팽창 특성을 측정하고 상호 비교하였다. 실험재료 중 gutta-percha 군으로는 Obtura, Diadent 그리고 Metadent 사의 gutta-percha를, Resilon으로 Pentron사의 Epiphany를 사용하였다. 열가압주입기인 Obtura II에 4가지 재료를 넣고 설정온도를 100℃, 150℃, 180℃ 그리고 200℃로 바꾸어 가며 각 온도에 대해서, heating chamber 입구에서의 온도와 23 게이지와 20 게이지의 needle에서 사출되는 재료의 온도를 디지털 thermometer를 이용하여 측정하였다. 열팽창을 측정하기 위해 세라믹으로 내경 3 ㎜, 외경 10 ㎜, 길이 27 ㎜의 원통형 주형을 제작하였고, 주형 안에 각 재료를 채워 넣은 후 양 끝을 세라믹 공이 (plunger)로 막았다. 이 시편을 dilatometer에 넣고 가열하여 25℃에서 75℃까지의 범위에서 열팽창 곡선을 얻었다. 온도에 따른 시편의 길이 변화로부터 각 재료의 열팽창 계수와 전체부피에 대한 팽창량을 계산하였다.

모든 재료에서 온도가 증가함에 따라 45℃ 이하에서는 재료의 부피변화가 거의 없었고, 45℃에서 55℃구간에서 급격히 팽창하였으며 그 이상의 온도에서는 완만한 부피의 증가를 보였다. 35℃에서 55℃사이에서의 부피의 변화는 재료들 사이에 통계적으로 유의한 차이가 없었으며 ($p > 0.05$), 35℃에서 75℃사이의 부피의 변화는 Obtura 사 gutta-percha가 Metadent사와 Diadent사의 gutta-percha에 비해 유의하게 작은 것으로 나타났다 ($p < 0.05$). Epiphany는 gutta-percha 군들과 비슷한 열팽창을 보였다 ($p > 0.05$).

주요어: 열팽창, Gutta-percha, Resilon, Dilatometer, 온도