Journal of Dental Sciences 18 (2023) 414–419

Available online at www.sciencedirect.com

ScienceDirect

journal homepage: www.e-jds.com

Original Article

The investigation of composition and thermal behavior of two types of backfilling gutta-percha

Pai-Chun Huang [a], Yi-Ting Wu [a], Yung-Hao Hsu [a,b], Szu-Chin Liao [a,b], Ya-Hsuan Wang [a], James L. Gutmann [c], Haw-Ming Huang [a], Sung-Chih Hsieh [a,b*]

[a] School of Dentistry, College of Oral Medicine, Taipei Medical University, Taipei, Taiwan
[b] Department of Dentistry, Taipei Municipal Wan-Fang Hospital, Taipei, Taiwan
[c] College of Dentistry, Texas A&M University, Dallas, TX, USA

Received 24 June 2022; Final revision received 16 August 2022
Available online 2 September 2022

KEYWORDS
Backfilling;
Regular-type gutta-percha;
Soft-type gutta-percha;
Thermal behavior;
Warm vertical compaction

Abstract *Background/purpose:* In the warm gutta-percha technique, soft-type and regular-type gutta-percha are using for backfilling thermoplasticized injection system. However, there are limited reports about the properties of these backfilling gutta-percha. This study aimed to analyze and compare the composition, thermal behavior and compact force of two types of backfilling gutta-percha.

Materials and methods: Soft-type and regular-type backfilling gutta-percha (B&L BioTech, Fairfax, VA, USA) were investigated. The inorganic and organic fractions of these gutta-perchas were separated by quantitative chemical analysis ($n = 6$). Their composition was analyzed using energy dispersive spectroscopy. Thermal behavior in response to temperature variations was analyzed using differential scanning calorimetry. Additionally, a compaction model was used to investigate the relation between compaction force and temperature ($n = 10$).

Results: The soft-type contained more gutta-percha (3.69–5.85%), carbon ratio (38.96–48.52%) and less inorganic substance (86.51–90.45%), zinc ratio (29.36–35.67%). The composition ratio of two types gutta-percha were statistically significant different ($P < 0.05$). There were three phase transitions of the soft-type gutta-percha which started at 39.84 °C, 49.32 °C and 54.15 °C while the two phase transitions of the regular-type gutta-percha started at 40.48 °C and 53.45 °C. The glass transition temperature of the regular-type gutta-percha (44.24 °C) was higher than that of the soft-type gutta-percha (40.66 °C). Under various setting temperature, the higher compaction force in the regular-type gutta-percha was required ($P < 0.05$).

* Corresponding author. School of Dentistry, College of Oral Medicine, Taipei Medical University, 250 Wu-Hsing Street, Taipei 11031, Taiwan.
E-mail address: endo@tmu.edu.tw (S.-C. Hsieh).

https://doi.org/10.1016/j.jds.2022.08.014

Conclusion: The different components in gutta-percha contribute to its differences in thermal behavior. The soft-type had a higher proportion of gutta-percha and lower ZnO which makes the fluidity better than the regular-type.

Introduction

Successful root canal treatment relies through disinfection and three-dimensional obturation of the root canal system. Schilder popularized a technique known as "Schilder' s warm percha vertical compaction technique" to fill root canal systems.[1] This technique adapts warm gutta-percha under non-isothermal conditions. The transformation of the heated softened gutta-percha and sealer is compacted by a series of pluggers to hermetically seal the portals of exit and overcome the complexity of the root canal systems, thus creating a three-dimensional obturation to prevent leakage.[1–5]

In Schilder's method, once the apical third of the canal is obturated, the coronal portion of the canal is filled using a back-packing process, using precut segments of gutta-percha that are heated, and compacted into the root canal.[1] Presently, back-packing may also use a thermoplasticized, gutta-percha injection system. Regardless of the numerous variations among brands of gutta-percha used for backfilling techniques, they are roughly divided into a regular-type and a soft-type according to different thermoplastic temperatures.[6,7] In clinical operation, it is discernible by their qualitative differences in the texture and operational feeling.

In previous studies, the composition of commercially available dental gutta-percha consists of 18%–22% gutta-percha, 1%–4% waxes and resins, 59%–76% zinc oxide and 1%–18% metal sulfates.[8–11] And the glass transition temperature (T_g) of dental gutta-percha is around 40 °C–44 °C.[12] The purity, molecular weight and crystallinity can affect melting points, also called phase change temperature of dental gutta-perchas.[13] The change of melting points (T_g) affects not only molecular arrangement of gutta-percha polymer but also its physical state.[14] Thus, the study on the composition and thermal behavior of gutta-perchas is needed to understand clinical differences of two types of backfilling gutta-perchas. Previous research regarding thermal behavior of gutta-percha focused on the temperature change during compacting process, and the effects of temperature change using different heated "cone" gutta-percha delivery systems.[15] However, there is little information regarding the cooling temperature of the many gutta-perchas used for backfilling the canal. Formulations of these gutta-perchas produced by different manufacturers vary slightly. Therefore, it is necessary for the operator to know the thermal behavior and composition of the chosen gutta-percha filling to ensure smooth clinical operation.

The purpose of this experiment was to study the relationship between different compositions of gutta-perchas used for backfilling the canals and their thermal behavior, and to identify a suitable setting temperature of the heating element, for optimal delivery of the obturation material in clinical operations.

Materials and methods

Composition analysis-chemical

According to Friedman's method of measuring the composition and proportion of gutta-percha, the main composition and proportion of regular-type and soft-type backfilling gutta-percha (B&L BioTech, Fairfax, VA, USA) (each type n = 6) were measured.[1,8,9] One gram of gutta-percha was soaked in 10 ml chloroform solution for 24 h, then centrifuged for 15 min (6000 rpm/min, 25 °C); the supernatant was removed for later use. The pellet formed was the inorganic component which contains zinc oxide and metal sulphate in the gutta-percha. After drying, it was weighed and recorded as M1 (inorganic) and 10 ml of acetone solution was added to the supernatant, mixed well and centrifuged for 15 min (6000 rpm/min, 25 °C). The separated pellet was gutta-percha. After drying, the pellet was weighed and recorded it as M2 (gutta-percha.). M3 (wax/resin) = 1-M1-M2.

Energy dispersive X-ray microanalysis

Energy dispersive X-ray spectroscopy (EDS) was used to qualitatively analyzed the chemical compositions of the two types of gutta-percha materials. Both 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

Thermal behavior of two types of backfilling gutta-percha were analyzed according to Hsu's method.[14] 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, USA). Two heating and cooling cycles were characterized as follows: (each type n = 3)

a) Heating followed by slow cooling.
   1. Temperature increased from 25 °C to 70 °C with heating rate of 1 °C/min.
   2. Temperature increased from 70 °C to 130 °C with heating rate of 5 °C/min.
   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/min.

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/min.

Compaction model

The regular-type and soft-type backfilling gutta-perchas (B&L BioTech) were obturated in 0.25-ml centrifuge tubes. Ten microcentrifuge tubes (each group n = 10) were placed in a digital dry bath incubator with temperature settings of 37 °C, 40 °C, 42 °C, 44 °C, 46 °C, 48 °C, 50 °C and 52 °C. An Instron universal testing machine (E3366, Instron, MA, USA) was set for packing 1 mm down to the gutta-percha with a Schilder plugger No.12(Dentsply Sirona, Ballaigues, Switzerland) in each of the testing groups. Forces were recorded during the gutta-percha compaction procedure.[12]

Statistical analysis

Statistical analyses were performed using SPSS for Windows (Version 19, SPSS Inc., Chicago, IL, USA). All data for each sample was analyzed using one-way analysis of variance (ANOVA) (SPSS Inc.) with Mann–Whitney U test. A P-value < 0.05 was considered statistically significant. Mann–Whitney U test was used when comparing two groups of data. All data are presented as mean and standard deviation (mean, ± SD).

Results

Composition analysis-chemical

The organic and inorganic components of commercial gutta-perchas were separated by chloroform and acetone solvents. Fig. 1 and Table 1 show that there were significant differences in the ratio of proportion between the two types of gutta-percha ($P < 0.05$). Regular-type was 90.45 ± 0.15%; soft-type was 86.51 ± 0.46% in inorganic substance (zinc oxide and metal sulphate). The proportion of gutta-perchas, regular-type was 3.69 ± 0.19%; soft-type was 5.85 ± 0.51%. In the wax/resin ratio, regular-type was 5.86 ± 0.13%; soft-type was 7.63 ± 0.30%.

Composition analysis-energy dispersive spectroscopy

According to energy dispersive spectroscopy test results, both regular-type and soft-type gutta-perchas, showed energy spectrum dominated by carbon, oxygen, and zinc. The regular-type contained 38.96%(wt) carbon, 15.4%(wt) oxygen, and 35.67%(wt) zinc, whether soft-type carbon was accounts for 48.52%(wt), 15.4%(wt) oxygen, and 29.36%(wt) zinc. The remaining elements, magnesium, silicon, titanium and platinum's ratio were listed in Table 2.

Thermal behavior analysis

During the heating process, the soft-type gutta-percha underwent three phase changes, while the regular-type gutta-percha underwent two phase changes. The two phases' transitions of regular-type gutta-percha were 39.39–47.2 °C, 53.45–58.08 °C; the three phase transitions of soft-type gutta-percha were 39.84–44.71 °C, 49.32–52.09 °C, 54.15–57.33 °C (Table 3). The glass transition temperature of

Table 1 Median values and interquartile range for chemical compositions (%) of backfilling gutta-percha from different types ($n = 6$).

Backfilling gutta-percha type	Inorganic	Organic	
		Gutta-percha	Wax/resin
Regular-type	90.47(0.07)	3.63(0.04)	5.91(0.07)
Soft-type	86.53(0.45)	5.58(0.51)	7.62(0.12)

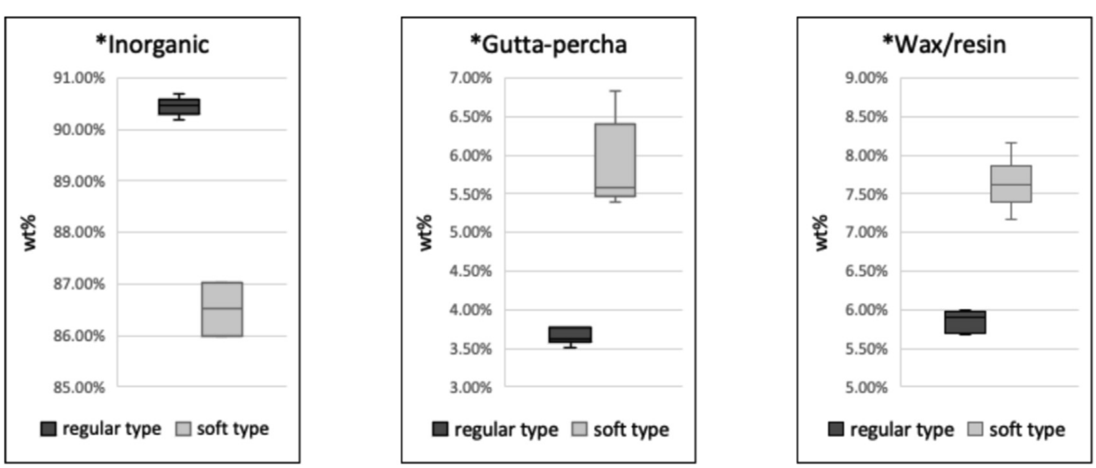

Figure 1 Box plot of inorganic, gutta-percha and wax/resin components of commercial dental backfilling gutta-percha. Statistical significance is shown as: *$P < 0.05$ (Mann–Whitney U test).

Table 2 Weight percentage (%) of components in two different types of backfill gutta-percha (n = 1).

Backfilling gutta-percha type	Chemical elements						
	C	O	Zn	Mg	Si	Ti	Pt
Regular-type	38.96	15.4	35.67	1.36	1.1	0.74	6.77
Soft-type	48.52	15.84	29.36	0.67	0.44		5.16

the two were 44.24 °C (regular-type) and 40.66 °C (soft-type) respectively (Table 3). The peak temperature of each phase transition and the glass transition temperature of regular-type were significantly higher than those of soft-type ($P < 0.05$).

During the rapid cooling process, the regular-type showed an exothermic peak at 77.94 °C, while the soft-type showed an exothermic peak at 48.01 °C. During the slow cooling process, the regular-type showed an exothermic peak at 71.38 °C, while the soft-type showed two exothermic peaks at 31.55 °C and 52.55 °C.

Relationship between compact force and heating temperature

A dry bath was used to simulate the experiment of using a universal testing machine and Schilder plugger to soften the gutta-percha under different temperature settings. As the heating temperature increased, the force required to compact the gutta-percha was reduced. Fig. 2 and Table 4 show that under various setting temperature, the force required to compact regular-type gutta-percha was significantly higher than that required to compact soft-type gutta-percha ($P < 0.05$).

Discussion

The manufacturer provides little information about the soft-type and regular-type gutta-perchas used for the backfilling thermoplasticized injection system. A recent study has shown that these two types backfilling gutta-percha have different viscoelastic behavior.[14] However, the literature lacks detailed scientific insights into the chemical composition and the thermal behaviors properties of two types of backfilling materials. Those properties should be taken into consideration when using backfilling gutta-perchas.

Inorganic substances were used as radial transparent plasticizers. We slightly modified the method, combining zinc oxide and metal sulfate into inorganic substances to calculate the ratio and compare the differences.[8,9] The experimental results (Fig. 1; Table 1), compared with the results of Friedman, Gurgel-Filho's and Liao's research

Table 3 Temperature (°C) of phase transition during heating cycle ($n = 3$).

Backfilling gutta-percha type	β to α phase		α to amorphous phase		Third peak		Glass transition temperature
	Initial phase change	Peak	Initial phase change	Peak	Initial phase change	Peak	
Regular-type	39.39	47.20	53.41	58.09	–	–	44.24
Soft-type	39.84	44.71	49.32	52.08	54.15	57.33	40.66

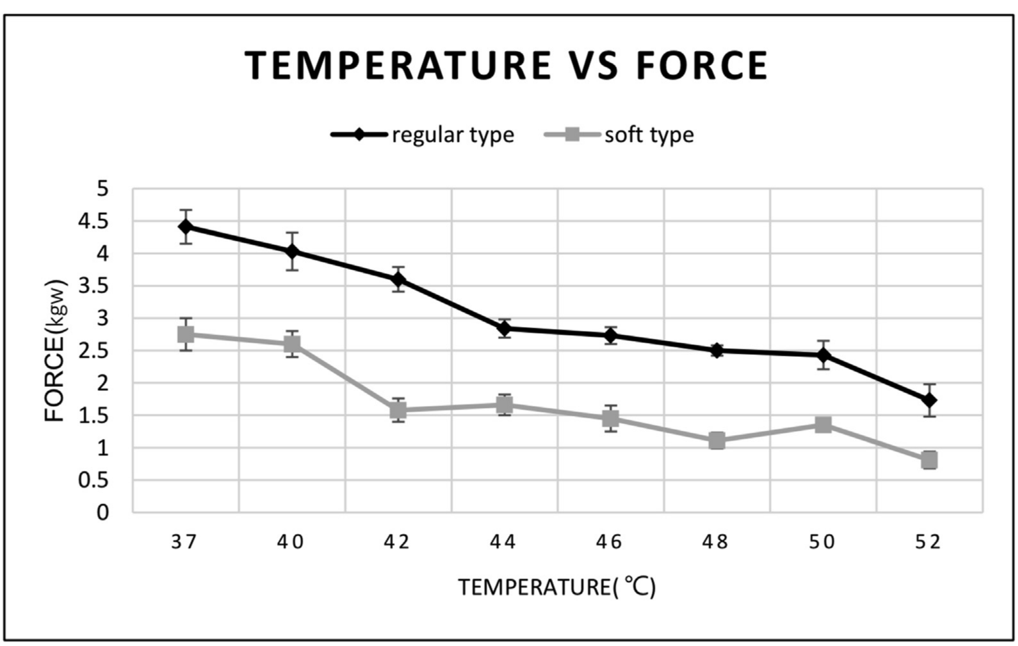

Figure 2 Relationship between compact force (kgw) and heating temperature (°C) (each temperature n = 10).

Table 4 Relationship between compact force (kgw) and heating temperature (°C) (each group n = 10).

Temperature (°C)	Regular-type (kgw)	Soft-type (kgw)
37	4.41 ± 0.26	2.75 ± 0.25
40	4.03 ± 0.29	2.60 ± 0.20
42	3.60 ± 0.19	1.58 ± 0.18
44	2.84 ± 0.14	1.66 ± 0.16
46	2.73 ± 0.13	1.45 ± 0.20
48	2.50 ± 0.08	1.11 ± 0.12
50	2.43 ± 0.22	1.35 ± 0.10
52	1.73 ± 0.25	0.81 ± 0.13

were different in composition ratio.[8–10,15] The inorganic composition ratio of regular-type and soft-type backfilling gutta-percha was 10–15% significantly higher ($P < 0.05$); the composition ratio of wax and resin was 3–4% significantly higher ($P < 0.05$); the ratio of gutta-percha was 14–16% significantly lesser ($P < 0.05$). The results of this experiment indicated that the gutta-percha segments from cones were different from the backfilling gutta-percha delivered through an injection needle due to the difference in material composition ratio. However, most of the current literature has focused on gutta-percha segments obtained from cones, and there were few discussions on the analysis of the components of backfilling gutta-percha in the injection systems.

According to the results of Friedman's research, the ratio of inorganic content (ZnO) to gutta-percha will affect the hardness, brittleness, and tensile strength of the backfilling gutta-percha.[8,9] Higher inorganic content (ZnO) would make the backfilling gutta-percha more brittle and reduced fluidity. The regular-type had more inorganic content (ZnO) than the soft-type, and the soft-type had more gutta-percha and wax and resin than the regular-type from our experiment. There was a significant difference between the two, $P < 0.05$ (Mann–Whitney U test), the results matched the clinical operating characteristics of soft-type gutta-percha, which was softer and more fluid than the regular-type. Recent research also indicated that the ratio of zinc in the gutta-percha could affect the thermal conductivity and volume shrinkage of the product.[15,16] The soft-type had a higher proportion of gutta-percha which makes the fluidity better than the regular-type; the regular-type had a higher proportion of zinc oxide which made the brittleness higher than the soft-type. The different properties of these types of gutta-percha led to different thermal behaviors and clinical portraits.

Gutta-percha used to backfill the canal and gutta-percha cone points were similar in the main element composition. Energy dispersive spectroscopy indicated that the element spectrum of C, O, Zn, Si, Ti appears to be common. The experiments of Gurgel-Filho and Maniglia-Ferreira detected elements such as H, N and S;[10,11]; however, we did not detect them in the experiments (Table 2). According to the experiments of Gurgel-Filho, element Ba was only detected in two samples of all five gutta-percha cone points.[10] In this study, element Ba wasn't be detected in two types of gutta-perchas evaluated (Table 2). We thought the absence of Ba was because of the presence of zinc oxide, which is as same function as radio-opacifier as Ba. The trace amounts of Mg and Pt were detected, but the experiments of Gurgel-Filho, Maniglia-Ferreira did not detect these elements.[10,11]

According to the experiments of Combe, Ferrante and Maniglia-Ferreira, this experiment was designed to detect the phase change of gutta-percha and all wave crests through a slow heating method.[11,17,18] The results of this experiment were also in line with the study by Chen, the transformation temperatures of dental gutta-percha are 42–49 °C for the beta to alpha transition and 53–59 °C for the alpha to amorphous transition.[12] During the heating process, the soft-type had undergone three phase changes, while the regular-type had undergone two phase changes from Table 3. The first phase change was from beta form to alpha form, and the second and third phase changes were from alpha form to amorphous form. But the soft-type gutta-percha had undergone three phase changes which is different from the previous study by Hsu.[14] Further study is needed to confirm the meaning of the third phase change of soft-type gutta-percha.

The regular-type did not show a significant exothermic peak, while the soft-type did at 48.41 °C in the cooling part. In previous studies, the meaning of the exothermic peak during the cooling process was the crystallization process from the amorphous form back to the beta form.[12] Therefore, it could be inferred from the experimental results that the soft-type underwent a more obvious crystallization process. Moreover, there was third exothermic peak in our DSC curve of soft-type, which differed from the previous studies focused on gutta-percha cone.[13,14,17,18]

The glass transition temperature was the first phase change and the crystalline structure change temperature of the gutta-percha. Previous studies focused on the thermal behavior around the glass transformation temperature of different commercial types of gutta-percha segments from cones.[15,19] The glass transition temperature of regular-type was significantly higher than that of the soft-type. It could be inferred that the optimal clinical operation temperature of cone gutta-percha and backfilling gutta-percha was different. This difference also existed between two types of gutta-percha. Therefore, there could be a difference in clinical operation when using different gutta-percha. It was easier for the doctor to operate around this temperature.

The thermal behavior of gutta-percha affected its mechanical properties strongly.[13–15] The result in our DSC test might indicate that soft-type backfilling gutta-percha had unexpectedly potential (the third thermal peak) to change its thermal behavior during higher temperature above glass transformation temperature. The need of further investigation regarding to thermal behavior of soft-type gutta-percha at higher temperature above glass transformation temperature should be addressed. Above all, the result might indicate that clinicians should aware when using soft-type backfilling gutta-percha in clinical use.

Comparing Fig. 1, Table 1, and Table 3, there was some correlation between chemical composition and glass transition temperature. The percentage of inorganic substance (ZnO) was positive related to the glass transition temperature. On the contrary, the percentage of gutta-percha was negative related to the glass transition temperature. These results were the same as previous studies.[14,15] Thus, it was speculated that the ratio of inorganic (ZnO) and gutta-percha affected the glass transition temperature of regular-type and soft-type backfilling gutta-percha. The of the soft-type was lower than that of the regular-type, which made the thermal behavior of the two different. The soft-type underwent phase change earlier during the heating process than the regular-type which made them clinical characteristics different.

The compact forces of the regular-type and the soft-type gutta-perchas at the glass transition temperature were significant different (Table 4). It was speculated that the required compact force decreased because of the crystalline structure change and volume shrinkage. According to Dong, the more inorganic substance (ZnO) the lower the volume shrinkage.[16] The soft-type had lower compact force compared with the regular-type. The soft-type with lower inorganic substance, higher components of gutta-percha needed and lower clinical heating temperature than regular-type. Possibly the soft-type backfilling gutta-percha had a higher proportion of molecular crystal changes and less zinc oxide to decrease volume shrinkage when heated, making it easier to change the volume, causing the compact force to decrease. This finding demonstrated that the soft-type gutta-percha changes more rapidly from a thermoplastic state to a non-thermoplastic state during clinical backfilling and therefore has relatively lower physical stability than regular-type gutta-percha at clinical heating temperature. Consequently, soft-type gutta-percha may need a more technique-sensitive compaction process than regular-type gutta-percha to reach a similar quality of root canal backfilling.

In summary, the different components in gutta-percha contribute to its differences in thermal behavior. The soft-type gutta-percha shows a higher proportion of fluidity compared to the regular-type. The regular-type has higher brittleness compared to the soft-type. Based on the findings of this study, it is imperative that clinicians be aware of individual properties of different types of backfilling gutta-percha, so clinical techniques can be adjusted accordingly to ensure success in root canal treatment.

Declaration of competing interest

The authors have no conflicts of interest relevant to this article.

Acknowledgments

This study was supported by the Ministry of Science and Technology, Taiwan (109-2813-C-038-033-B).

The authors would like to thank Taipei Medical University for providing all the research facilities for this study.

References

1. Schilder H. Filling root canals in three dimensions. *J Endod* 2006;32:281—90.
2. Diemer F, Sinan A, Calas P. Penetration depth of warm vertical gutta-percha pluggers: impact of apical preparation. *J Endod* 2006;32:123—6.
3. Venturi M, Breschi L. Evaluation of apical filling after warm vertical gutta-percha compaction using different procedures. *J Endod* 2004;30:436—40.
4. Johnson BT, Bond MS. Leakage associated with single or multiple increment backfill with the Obtura II gutta-percha system. *J Endod* 1999;25:613—4.
5. Gilbert SD, Witherspoon DE, Berry CW. Coronal leakage following three obturation techniques. *Int Endod J* 2001;34: 293—9.
6. Li GH, Niu LN, Zhang W, et al. Ability of new obturation materials to improve the seal of the root canal system: a review. *Acta Biomater* 2014;10:1050—63.
7. Frantzeska K, Christopoulos D, Chindrokoukis P. Gutta percha and updated obturating techniques. *J Dent Health Oral Disord Ther* 2017;8:470—4.
8. Friedman CM, Sandrik JL, Heuer MA, Rapp GW. Composition and mechanical properties of gutta-percha endodontic points. *J Dent Res* 1975;54:921—5.
9. Friedman CE, Sandrik JL, Heuer MA, Rapp GW. Composition and physical properties of gutta-percha endodontic filling materials. *J Endod* 1977;3:304—8.
10. Gurgel-Filho ED, Andrate-Feitosa JP, Teixeira FB, Monterio de Paula RC. Chemical and X-ray analyses of five brands of dental gutta-percha cone. *Int Endod J* 2003;36:302—7.
11. Maniglia-Ferreira C, Gurgel-Filho ED, Araújo Silva Jr JB, Paula RCM, Andrade-Feitosa JP, Sousa-Filho FJ. Chemical composition and thermal behavior of five brands of thermoplasticized gutta-percha. *Eur J Dermatol* 2013;7:201—6.
12. Chen CH, Shen YK, Hsieh SC. The investigation of gutta-percha temperature and compaction force change when using the vertical compaction of warm gutta-percha technique. *J Polym Eng* 2014;34:219—23.
13. Goodman A, Schilder H, Aldrich W. The thermomechanical properties of gutta-percha. II. The history and molecular chemistry of gutta-percha. *Oral Surg Oral Med Oral Pathol* 1974;37:954—61.
14. Hsu YH, Wang HH, Shen YK, Gutmann JL, Hsieh SC. Thermal behavior and viscoelastic properties of gutta-percha used for back-filling the root canal. *J Dent Sci* 2020;15:28—33.
15. Liao SC, Wang HH, Hsu YH, Huang HM, Gutmann JL, Hsieh SC. The investigation of thermal behaviour and physical properties of several types of contemporary gutta-percha points. *Int Endod J* 2021;54:2125—32.
16. Dong M, Zhang J, Liu L, Yu Y, Yuan C, Wang X. Volume shrinkage mechanism of gutta-percha point studied by differential scanning calorimetry and volume dilatometer in vitro. *Polym Test* 2019;73:115—23.
17. Combe EC, Cohen BD, Cummings K. Alpha-and beta-forms of gutta-percha in products for root canal filling. *Int Endod J* 2001;34:447—51.
18. Ferrante M, Trentini P, Croce F, Petrini M, Spoto G. Thermal analysis of commercial gutta-percha. *J Therm Anal Calorim* 2011;103:563—7.
19. Chang J, Baek SH, Lee IB. Rheological characterization of thermoplasticized injectable gutta percha and resilon. *J Korean Acad Conserv Dent* 2011;36:377—84.