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The investigation of thermal behaviour and physical properties of several types of contemporary gutta-percha points

Szu-Chin Liao[1,2] | **Hsin-Hui Wang**[2] | **Yung-Hao Hsu**[1,2] | **Haw-Ming Huang**[1,3] | **James L. Gutmann**[4] | **Sung-Chih Hsieh**[1,2]

[1]School of Dentistry, College of Oral Medicine, Taipei Medical University, Taipei, Taiwan

[2]Department of Dentistry, Taipei Municipal Wan-Fang Hospital, Taipei, Taiwan

[3]Graduate Institute of Biomedical Optomechatronics, College of Biomedical Engineering, Taipei Medical University, Taipei, Taiwan

[4]College of Dentistry, Nova Southeastern University, Ft. Lauderdale, Florida, USA

Correspondence
Sung-Chih Hsieh, Chairman of Endodontic Department, School of Dentistry, College of Oral Medicine, Taipei Medical University, 250 Wu-Hsing Street, Taipei 11031, Taiwan.
Email: endo@tmu.edu.tw

Abstract

Aim: To analyse the contents and thermal behaviour of several brands of contemporary gutta-percha points due to the variable nature of the components of gutta-percha, and the impact they can have on the physical properties of this unique material during canal filling.

Methodology: Six brands of gutta-percha were investigated: Conform Fit ™ Gutta-Percha Points for ProTaper Gold® (PTG) (Dentsply Sirona), ProTaper® Universal Gutta-Percha Points (PTU) (Dentsply Sirona), Autofit ™ Feathered Tip Gutta Percha (Kerr), Mtwo® Gutta-Percha (VDW), Gutta Percha Root Canal Points (GC, GC Corporation) and Gutta-Percha Points ISO Color-Coded (ISO; Dentsply Sirona). The organic and inorganic fractions of gutta-percha points were separated by quantitative chemical analysis. Thermal conductivity was detected using a laser flash method. In addition, the thermal behaviour of gutta-percha in response to temperature variations was analysed using differential scanning calorimetry (DSC). Kruskal–Wallis and Dunn tests were applied for comparisons amongst groups for chemical compositions and temperature obtained from DSC. The associations between compositions and thermal conductivity were determined using simple linear regression. A p value <.05 was considered to be statistically significant.

Results: There were significant difference in the inorganic fractions of the gutta-percha points in percentage by weight amongst the groups ($p < .05$). PTG had the lowest thermal conductivity (0.42 W/m K). A positive correlation was observed between the percentage of inorganic fraction and thermal conductivity ($r = 0.95$). The initial phase changing temperature and peak temperature measured by DSC were significantly different when the β-form transformed to α-form ($p < .05$), whereas no significant difference was found during the α-form to the amorphous-phase transition ($p > .05$).

Conclusions: Chemical compositions and initial phase changing temperature by DSC varied according to the various brands of gutta-percha points. Conform Fit ™

Szu-Chin Liao and Hsin-Hui Wang contributed equally to the study.

gutta-percha had the lowest percentage of inorganic fraction and thermal conductivity amongst these six brands of gutta-percha. Thermal conductivity had the strongest positive correlation with the percentage of inorganic components and zinc, whilst there was a negative correlation to the amount (ratio) of gutta-percha.

KEYWORDS

chemical composition, gutta-percha, thermal conductivity

INTRODUCTION

One of the clinical objectives of successful root canal treatment is the filling of the entire root canal system (Schilder, 1967). Since the introduction of gutta-percha into Dentistry, it has been the most commonly used material for root canal filling (Gatewood, 2007). Current dental gutta-percha points, composed of both organic and inorganic components, are biocompatible with low toxicity (Hauman & Love, 2003). The organic components are mainly gutta-percha (matrix) and wax/resin (plasticizer) that make the material more flexible and compressible; the inorganic components are metal sulphates (radiopacity material) and zinc oxide (filler) that increase brittleness and reduce tensile strength (Friedman et al., 1975, 1977; Gurgel-Filho et al., 2003).

The varying compositions of dental gutta-percha from different manufacturers can affect the physical properties, such as brittleness, stiffness, flow, plasticity, elongation, tensile strength, inherent tension force, radiopacity and thermal behaviour (Marciano & Michailesco, 1989; Marciano et al., 1992; Tagger & Gold, 1988).

Natural gutta-percha, an isomerism of natural rubber, is a *trans*-1,4-polyisoprene polymer obtained from the coagulation of latex produced by trees of the family Sapotaceae and is primarily derived from *Palaquium gutta* bail (Marciano et al., 1993). Gutta-percha exists in two distinct crystalline phases: the alpha (α) phase and the beta (β) phase. These forms are different in the molecular repeat distance and single carbon bond configuration. The two crystalline phases are convertible, which indicates that the β-form transforms to the α-form and the α-form transforms to the amorphous phase (melt) during thermal enhancement. When the amorphous phase is cooled under the rate less than 0.5°C/h, the α-form will recrystallize (Bunn, 1942; Goodman et al., 1974). Schilder et al. (1974) proposed that the glass transition temperature of dental gutta-percha from the β-form to the α-form was around 42–49°C, and the melting temperature from the α-form to the amorphous phase was 53–59°C.

Due to the complexity of the root canal system (Çalişkan et al., 1995; Green, 1955; Vertucci, 1984), and the challenges encountered when using current filling materials, it is often difficult to achieve complete filling of the root canal space. Indeed, there are many canal filling techniques used in Endodontics (Whitworth, 2005), including cold lateral condensation and thermoplasticized techniques. Schilder (1967) described the use of warm gutta-percha compaction technique in an attempt to acquire a three-dimensional filling. The technique utilizes a non-isothermal operation and a heat source is required during manipulation. However, gutta-percha itself is not a good thermal conductor and will soften only near the heat source or slightly beyond. Adding zinc oxide, barium sulphate and other inorganic substance to the dental gutta-percha points can improve its thermal conductivity but will increase its hardness and change its rheology after heating (Goodman et al., 1981). There are limited reports about thermal conductivity of gutta-percha, and the relationship between chemical composition and thermal conductivity is also somewhat obscure.

The purpose of the present investigation was to determine the chemical composition and thermal properties of six commercially available gutta-percha points. Using these data, an attempt was made to determine whether a correlation exists between the gutta-percha composition and its thermal conductivity. The null hypothesis to be tested that there were no differences amongst the compositions of all gutta-percha brands analysed.

MATERIALS AND METHODS

Six commercially available brands of gutta-percha points were included: Conform Fit ™ Gutta-Percha Points for ProTaper Gold® (PTG) (Dentsply Sirona), ProTaper® Universal Gutta-Percha Points (PTU) (Dentsply Sirona), Autofit ™ Feathered Tip Gutta Percha (Kerr), Mtwo® Gutta-Percha (VDW), Gutta Percha Root Canal Points (GC) (GC Corporation) and Gutta-Percha Points ISO Color-Coded (ISO) (Dentsply Sirona). All samples were analysed before the expiration dates established by the manufacturers.

Quantitative chemical analysis

The chemical components of the gutta-percha points were determined according to the procedures described by Friedman et al. (1975). One gram of the gutta-percha points from the six different brands was weighed and dissolved in 10 mL of chloroform for 24 h prior to centrifugation. The resulting solution was then centrifuged at 10^4 *g* for 15 min. The solid phase (inorganic components: zinc oxide and metal sulphates), separated from the supernatant (organic components: gutta-percha and wax/resin), was acquired in this process. The gutta-percha polymer, insoluble in acetone, was coagulated by addition of this solvent and weighed after total solvent evaporation. The mass of soluble material (wax/resin) in acetone was determined following solvent evaporation. Four samples of gutta-percha points from each brand were quantitatively assayed.

Energy dispersive X-ray microanalysis

Energy-dispersive X-ray (EDX) microanalysis was used to qualitatively establish the presence of chemical elements in the samples. The samples of all brands were mounted on aluminium stubs and examined using a JSM-6510 Scanning Electron Microscope (JEOL) fitted with an energy-dispersive X-ray spectrometer.

Differential scanning calorimetry

The thermal analyses of all samples were performed by differential scanning calorimetry (Diamond DSC, PerkinElmer). A total of three specimens from each brand were heated from 25°C to 70°C at a heating rate of 1°C/min followed by a rate of 5°C/min heating up to 130°C; subsequently, the temperature decreased from 130°C to 25°C at a cooling rate of 5°C/min.

Thermal conductivity analysis

Thermal conductivity was measured indirectly using the laser flash method, which was first introduced by Parker et al. (1961). The samples of the six brands were prepared by hot pressing the pellets of the solid gutta-percha polymer to obtain six small thin disks, 12.7 mm in diameter and 0.3 ~ 0.5 mm in thickness. These specimens were tested using the laser flash method by LFA 467 HyperFlash (Netzsch) to acquire thermal diffusivity, allowing the thermal conductivity to be calculated using the following equation:

$$\alpha = \frac{k}{\rho C_p}$$

where α is thermal diffusivity (m^2/s), K is thermal conductivity (W/m K), ρ is density (kg/m^3) and C_p is specific heat capacity (J/kg °C).

Statistical analysis

Data from these tests were evaluated statistically using the Kruskal–Wallis test with Dunn's *post hoc* to compare the compositions of all gutta-percha brands and temperature obtained from differential scanning calorimetry. The Pearson correlation and simple linear regression analyses were used to explore the association between chemical compositions and thermal conductivity. The significance level for all analyses was accepted for p values lower than .05.

RESULTS

Quantitative chemical analysis

The quantitative chemical analysis of the six brands of gutta-percha points is shown in Figure 1 and Table 1. Amongst the organic fraction, the median value of percentage of gutta-percha was between 8.5% and 17%. PTG had the highest concentrations of gutta-percha, and GC had the lowest. There was a significant difference for the gutta-percha percentages amongst the groups ($p < .05$). With regard to the percentage of wax/resin, no significant difference was found ($p > .05$). The median value of percentage of inorganic components was between 77% and 86.5%. GC had the highest percentage of inorganic components, followed by Autofit, ISO, PTU, Mtwo and PTG. There was a significant difference amongst the groups ($p < .05$).

Energy-dispersive X-ray microanalysis

Table 2 shows the chemical elements detected by EDX microanalysis. The data indicated that carbon, zinc and oxygen were the main elements in all of the six brands. Barium and aluminium were also present except in GC gutta-percha points. In addition, small amounts of other elements were found depending on the manufacturer.

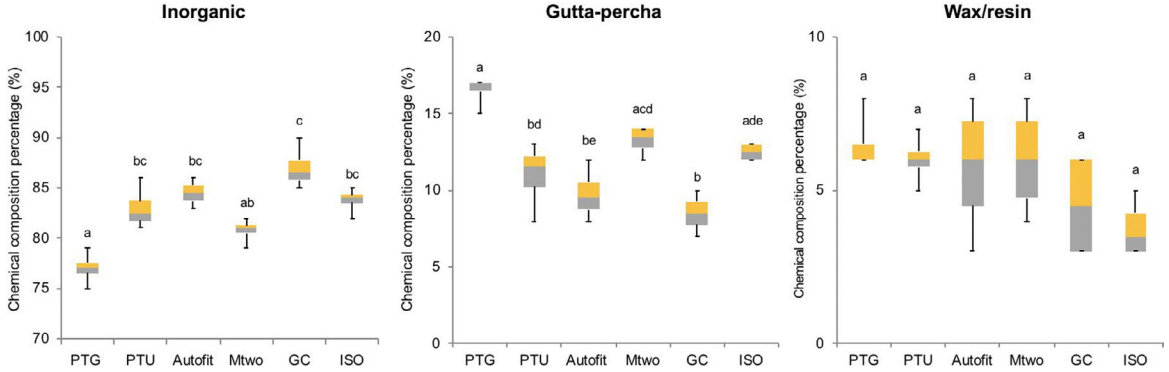

FIGURE 1 Box plot of inorganic, gutta-percha and wax/resin components of commercial dental gutta-percha points. Different lowercase superscript letters indicate significant differences according to the Kruskal–Wallis and Dunn tests ($p < .05$). [Colour figure can be viewed at wileyonlinelibrary.com]

TABLE 1 Median values and IQR for chemical compositions (%) of gutta-percha points from different brands ($n = 4$)

Gutta-percha brand	Inorganic components	Organic components	
		Gutta-percha	Wax/resin
PTG	77.0 (1.0)^{a}	17.0 (0.5)^{a}	6.0 (0.5)^{a}
PTU	82.5 (2.0)^{bc}	11.5 (2.0)^{bd}	6.0 (0.5)^{a}
Autofit	84.5 (1.5)^{bc}	9.5 (1.8)^{be}	6.0 (2.8)^{a}
Mtwo	81.0 (0.8)^{ab}	13.5 (1.3)^{acd}	6.0 (2.5)^{a}
GC	86.5 (2.0)^{c}	8.5 (1.5)^{b}	4.5 (3.0)^{a}
ISO	84.0 (0.8)^{bc}	12.5 (1.0)^{ade}	3.5 (1.3)^{a}
p value	.0024	.0025	.1646

Different lowercase superscript letters indicate significant differences according to the Kruskal–Wallis and Dunn tests ($p < .05$).

Abbreviation: IQR, interquartile range.

TABLE 2 Weight percentage (%) of components in six different brands of gutta-percha points

Gutta-percha brand	Chemical elements									
	C	O	Zn	Ba	Al	S	Mg	Si	Ti	Ca
PTG	41.81	16.80	28.58	8.27	0.25	1.82	0.68	0.79	1.00	
PTU	37.05	16.84	43.75	2.13	0.22					
Autofit	34.48	16.53	41.12	6.07	0.39					1.41
Mtwo	34.52	16.82	45.83	2.59	0.24					
GC	27.85	17.99	54.16							
ISO	31.62	16.66	51.09		0.33			0.30		

Differential scanning calorimetry

All products had a thermal behaviour with two endothermic peaks during the cycle. Table 3 presents the median values and interquartile range of phase changing temperature. From β- to α-phase transition, the initial phase transition occurred when the gutta-percha was heated from 39.85°C to 45.80°C and the peak temperature value in the phase transition ranged from 47.98°C to 50.63°C. There was significant difference for the temperature of β- to α-phase transition amongst the groups ($p < .05$). When the α-form transformed to the amorphous phase, the temperature of initial phase transition was from 53.16°C to 54.63°C and the peak temperature value in the phase transition was from 59.78°C to 60.04°C. No significant difference was verified amongst the groups ($p > .05$). No obvious changes in the heat flow were observed for all analysed materials at temperatures higher than 120°C during the

heating process; neither were exothermic peaks observed in the cooling process.

Thermal conductivity analysis

Thermal diffusivity, specific heat capacity and density are listed in Table 4. GC gutta-percha points had the highest thermal conductivity (0.96 W/m K), whilst PTG had the lowest thermal conductivity (0.42 W/m K). When applying the Pearson correlation analysis, a significant correlations between the percentage of inorganic fraction and thermal conductivity ($r = 0.95$, $p < .05$), the percentage of zinc element and thermal conductivity ($r = 0.93$, $p < .05$) and the percentage of gutta-percha and thermal conductivity ($r = -0.90$, $p < .05$) were revealed. In the simple linear regression analysis, the percentage of inorganic fraction and zinc were positively related to thermal conductivity. The coefficient of determination, r^2, were 0.90 and 0.87 respectively ($p < .05$). However, there was a negative correlation between the percentage of gutta-percha and thermal conductivity ($r^2 = 0.81$, $p < .05$; Figure 2).

DISCUSSION

Gutta-percha has been used as a root canal filling material for over a century. Different manufacturers produce distinct designs and proprietary compositions for dental gutta-percha points. Similar to previous study (Maniglia-Ferreira et al., 2013), thermoplastic gutta-percha systems (PTG, PTU, Autofit and Mtwo gutta-percha points) were included in the present study due to their widespread clinical application for root canal filling. In this group, PTG (conform fit points) was the most recent intracanal material; however, there is limited information on its

TABLE 3 Median values and IQR for temperature (°C) of phase transition during heating cycle ($n = 3$)

	β to α phase		α to amorphous phase	
Gutta-percha brand	**Initial phase change**	**Peak**	**Initial phase change**	**Peak**
PTG	40.02 (1.41)^{ab}	48.54 (0.27)^{ab}	53.19 (0.23)^{a}	59.90 (0.15)^{a}
PTU	39.85 (0.09)^{bc}	47.98 (0.01)^{bc}	53.16 (0.30)^{a}	60.03 (0.03)^{a}
Autofit	46.52 (0.11)^{d}	50.07 (0.04)^{ae}	53.17 (0.56)^{a}	59.79 (0.08)^{a}
Mtwo	40.05 (0.05)^{abe}	48.52 (0.01)^{ab}	53.17 (0.18)^{a}	60.02 (0.03)^{a}
GC	45.80 (0.07)^{ad}	50.63 (0.02)^{de}	54.63 (0.32)^{a}	59.78 (0.06)^{a}
ISO	45.67 (0.85)^{ad}	50.12 (0.26)^{abd}	53.78 (0.10)^{a}	60.04 (0.05)^{a}
p value	.0101	.0066	.0522	.0515

Different lowercase superscript letters indicate significant differences according to the Kruskal–Wallis and Dunn tests ($p < .05$).

Abbreviation: IQR, interquartile range.

TABLE 4 Thermal conductivity analysis in six different brands of gutta-percha points

Gutta-percha brand	Thermal diffusivity (m^2/s)	Specific heat capacity (J/kg °C)	Density (kg/m^3)	Thermal conductivity (W/m K)
PTG	0.26	0.91	1.82	0.42
PTU	0.26	1.07	2.00	0.55
Autofit	0.35	0.83	2.68	0.78
Mtwo	0.35	0.84	2.56	0.75
GC	0.41	0.75	0.78	0.96
ISO	0.40	0.74	2.63	0.78

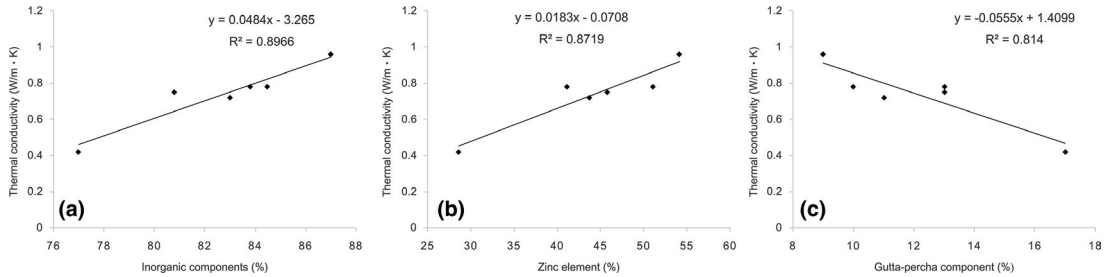

FIGURE 2 The simple linear regression analyses. (a) The correlation between thermal conductivity and inorganic components. (b) The correlation between thermal conductivity and zinc element. (c) The correlation between thermal conductivity and gutta-percha component.

composition. This formulation is designed to be used with warm vertical compaction. The other two brands of gutta-percha points, GC and ISO, are usually used in cold lateral compaction technique in most sectors and were included for comparison. All materials included were identified by quantitative chemical analysis, and their relative percentages were determined by chemical composition and EDX microanalysis.

It is well known that the amounts of inorganic and organic components will influence physical properties. It was observed that gutta-percha content was positively correlated with yield strength and high zinc oxide levels were associated with low percentage elongation. The desirable root canal filling material should incorporate flexibility and rigidity, even though they are two opposite characteristics (Friedman et al., 1977). Hence, it seems difficult to develop the ideal material for canal filling. The chemical components of the gutta-percha points were determined carefully using the time-test methodology of Friedman et al. (1975). According to their studies and others (Friedman et al., 1975; Gurgel-Filho et al., 2003; Maniglia-Ferreira et al., 2013), the proportion of components of gutta-percha points was about 72.5% to 84.3% by weight in inorganic substances; 14.5% to 22.4% in gutta-percha and 1.2% to 7.8% in wax/resin, which is similar to the results of the present work (Table 1). However, there was a significant difference in the proportion of inorganic substances and gutta-percha amongst the groups. Thus, the null hypothesis is rejected. It may reflect attempts by the manufacturers to enhance the performance of the gutta-percha points over the past few decades. Due to the clinical manipulation based on the filling techniques, the data also revealed heterogeneity amongst the different brands of dental gutta-percha points. The thermoplastic gutta-percha points tended to have lower percentages of inorganic compounds, which was similar to a previous study (Maniglia-Ferreira et al., 2013).

A survey of the chemical elements of gutta-percha points was provided by the EDX microanalysis (Table 2) and, according to the results, zinc was found to be generally present in large amounts. It indicates that zinc oxide is the main ingredient in these brands of gutta-percha points (although in variable percentages), which is in accordance with the earlier assessments (Gurgel-Filho et al., 2003, 2006; Möller & Ørstavik, 1984). The occasional occurrence of small amounts of Cl, Ti, Ca and Si is most probably due to impurities or small volumes of compounds added during manufacturing either for processing or to enhance material properties. Combining the data of chemical compositions (Table 1) and chemical elements (Table 2), GC gutta-percha had the highest level of inorganic substances, as well as the presence of zinc; PTG gutta-percha points (conform fit) had the lowest proportion of inorganic substances and zinc; reflecting that zinc oxide accounts for the majority of inorganic fraction. Zinc oxide has high thermal conductivity (Janotti & Van de Walle 2009). Therefore, it provides an explanation why that material with high ratio of zinc oxide (inorganic fraction) would exhibit better thermal conduction.

In the past, many investigations (Blum et al., 1997; Goodman et al., 1981; Marroquín et al., 2015) have attempted to explore the thermal conductivity of gutta-percha by detecting temperature changes subsequent to heating. Nevertheless, whilst thermal conductivity of the material is a direct indicator of its handling properties, there is insufficient literature addressing this aspect. Fan et al. (2017) revealed that thermal conductivity of gutta-percha points ranged from 0.60 to 2.25 W/m K. However, the data of the present study detected by a laser flash method indicated 0.42–0.96 W/m K (Table 4), which differed from the previous work detected using a transient plane source method. The laser flash method was implemented due to several advantages, such as a short time required for measurement, a relatively small effect of heat loss from the sample and the requirement of a small sample size (Takahashi & Murabayashi, 1975). This method was quite suitable for testing polymers, especially a relatively miniature material like gutta-percha points (dos Santos et al., 2005).

There is limited information available on the thermal conductivity and chemical compositions of gutta-percha. GC gutta-percha points had the highest thermal

conductivity corresponding to the highest percentage of inorganic components (Tables 1 and 4). Interestingly, a similar correlation also occurred in the result of PTG gutta-percha points, which demonstrated the lowest thermal conductivity corresponding to the lowest percentage of inorganic components. Furthermore, thermal conductivity revealed a high positive correlation between the percentage of inorganic components and zinc, as assessed using the Pearson correlation. Also, a high negative correlation was found between thermal conductivity and the ratio of gutta-percha. The explanatory power of linear regression model between the percentage of inorganic components and thermal conductivity was 90%. According to the regression formula, the thermal conductivity could be estimated not only by the percentage of inorganic components but also by the percentage of elemental zinc and gutta-percha. The model correlation powers were 87% and 81%, respectively, with these two factors. These results provide evidence to support that material with high ratio of zinc oxide (inorganic fraction) would have better thermal conduction.

The thermal analysis technique has been proven to be a credible method for analysing gutta-percha (Combe et al., 2001; Ferrante et al., 2011; Marciano et al., 1992; Schilder et al., 1974). According to previous studies (Combe et al., 2001; Ferrante et al., 2011; Hsu et al., 2020), the heating procedure of this experiment was divided into two steps. The results of the present study revealed that all products had two typical major endothermic peaks (Table 3), which are similar to those of the study by Schilder et al. (1974). In the six brands of commercial gutta-percha points, the initial phase changing temperatures of PTG and PTU were relatively low (approximately 40°C), which would tend to indicate that the appropriate temperatures for warm gutta-percha techniques would vary depending on the specific brand of gutta-percha points used (Roberts et al. 2017).

The Conform Fit ™ gutta-percha had the lowest percentage of inorganic fraction and the highest percentage of organic fraction in the present study (Table 1). Such characteristic composition will reduce the brittleness and hardness and increase the plasticity of gutta-percha (Friedman et al., 1977), which would allow it to adapt to canal irregularities during compaction. Due to the relatively low initial temperature for the β- to α-phase transition and the difference in physical properties caused by the composition, it might explain why Conform Fit ™ gutta-percha is easier to soften after heating than other brands in the same clinical situation. The findings are consistent with the statement of Dentsply Sirona that a lower heating temperature is required for Conform Fit ™ gutta-percha (See https://www.chooseyourendosolution.com/storage/app/media/downloads/Obturatie.pdf). However, Dentsply Sirona did not disclose the relevant experimental methods and results for thermal conduction of the characteristics of Conform Fit ™ gutta-percha. Depending on the present result, Conform Fit ™ gutta-percha had the lowest thermal conductivity, which might offer suitable time for manipulation during a warm gutta-percha filling technique.

Although limited information was obtained due to only six brands of gutta-percha points included, it is reasonable to suggest that the zinc oxide (inorganic fraction) plays an important role in thermal conduction based on the correlations found. In addition, the data presented in this study can be a useful reference for further evaluation with more brands of gutta-percha points.

CONCLUSIONS

The chemical composition of several commercially available dental gutta-percha points varied greatly. The ratio of organic and inorganic substances affects the physical properties. Increasing the inorganic component elevated their thermal conductivity.

ETHICAL STATEMENT

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AUTHOR CONTRIBUTION

Sung-Chih Hsieh: conceptualization and project administration; Sung-Chih Hsieh, Szu-Chin Liao, Hsin-Hui Wang, Yung-Hao Hsu: data curation; Sung-Chih Hsieh, Szu-Chin Liao, Hsin-Hui Wang, Haw-Ming Huang: methodology; roles/writing - original draft and writing - review & editing: all authors.

CONFLICT OF INTERESTS

The authors have stated explicitly that there are no conflicts of interest in connection with this article.

ORCID

Szu-Chin Liao https://orcid.org/0000-0003-0666-2796
Yung-Hao Hsu https://orcid.org/0000-0001-7199-4629
Haw-Ming Huang https://orcid.org/0000-0003-4184-3744

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