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(54) Title: TEMPERATURE INDICATING ENDODONTIC OBTURATION MATERIAL

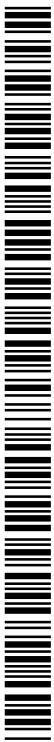
(57) **Abrégé/Abstract:**

A gutta-percha composition for filling a tooth root canal includes a temperature indicating component, such that the user has a visual indication that the material is ready for placement in the prepared canal. The material may include a thermochromatic dye, bismuth oxide or the like, in an otherwise conventional obturation material.

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(54) **Title:** TEMPERATURE INDICATING ENDODONTIC OBTURATION MATERIAL(57) **Abstract:** A gutta-percha composition for filling a tooth root canal includes a temperature indicating component, such that the user has a visual indication that the material is ready for placement in the prepared canal. The material may include a thermochromatic dye, bismuth oxide or the like, in an otherwise conventional obturation material.

## TEMPERATURE INDICATING ENDODONTIC OBTURATION MATERIAL

### RELATED APPLICATION

This application is a national phase entry of PCT/US2016/026179 which claims priority to Provisional US Application No. 62/143,919 filed on April 7, 2015.

### TECHNICAL FIELD

[0001] The present invention relates to endodontic obturation materials. More particularly, the invention relates to obturation materials that show a color change as an indication of temperature change.

### BACKGROUND OF THE INVENTION

[0002] Gutta-percha (Palaquium) is a genus of tropical trees native to Southeast Asia and northern Australasia, from Taiwan south to the Malay Peninsula and east to the Solomon Islands. The same term is used to refer to inelastic natural latex produced from the sap of these trees, particularly from the species *Palaquium gutta*. Chemically, gutta-percha is a polyterpene, a polymer of isoprene, or polyisoprene, specifically (trans-1, 4-polyisoprene). Examples of known gutta-percha compositions and certain uses thereof are disclosed for example in published patent application WO2014182570A1.

[0003] The bio inertness of gutta-percha makes it suitable for use within the human body. It is used in a variety of dental applications. In the 1900's this material was used to fill cavities. In the early 1900's the material was used in root canal therapy. Gutta-percha remained a staple well into the 20<sup>th</sup> century, when it was gradually replaced by alternative materials. A similar, cheaper natural material called balata is often used in gutta-percha's place. The two materials are almost identical, and balata is sometimes called gutta-ballata. For simplicity, all such materials will be encompassed by the term "gutta-percha" for purposes of this disclosure.

[0004] A material used to obturate or fill the empty space inside the root of a tooth after it has undergone root canal therapy, is often called "synthetic" in the dental arts. Synthetic became more readily available as the demand increased and the availability of natural material decreased. Its physical and chemical properties, including but not limited to its inertness and

biocompatibility, melting point, and malleability, make it an important material used in endodontics. Synthetic is also encompassed by the term "gutta-percha herein, again for simplicity sake. It will be understood that any conventional root canal obturation material is within the scope of the term "gutta-percha" herein.

**[0005]** Gutta-percha and more particularly, trans-1,4-polyisoprene was originally used as a cold material to fill the root canal. The material was rolled into a cone shaped point to fill the canal. The use of sealant was added to fill the voids around the points. Eventually the material was heated. The heated (melted) gutta-percha flowed to fill the voids and cavities within the canal. Heating was preferred because it allowed for a three dimensional fill, substantially absent of voids. The use of heated gutta-percha introduced new adjustable parameters, such as temperature, flow rate and tackiness.

**[0006]** When filling a root canal, heated gutta-percha is normally delivered by one of two methods. It can be placed with an electronic delivery device or placed on an endodontic carrier. Both methods deliver it hot into the canal, and both methods conventionally require compaction either with a metal plugger or the endodontic carrier. Further, these methods require the material to be within a temperature range ideal for the compaction.

**[0007]** Currently, the dental or endodontic clinician relies on the appearance of the surface or the feel when touched with a gloved finger to determine if the gutta-percha is heated to the correct temperature for placement. When looking at the gutta-percha, the clinician can tell if the material has been heated, (the material often becomes more "plump") but not if it is currently the right temperature. If the material is too hot, it may not place well and the canal will not be properly obturated. Similarly, if the material is too cold it will not flow or place properly. The clinician will try to place the material into the canal and find that it will not fill the voids or not completely fill the canal.

**[0008]** The present invention overcomes these difficulties, giving the clinician a precise real time temperature indicator which allows for ease of placement and will substantially take the temperature estimate out of the procedure. This will give assurance that the material will place properly and fill the endodontically prepared root canal as needed.

## **DISCLOSURE OF THE INVENTION**

**[0009]** According to the invention, a gutta-percha composition for filling a tooth root canal includes a temperature indicating component. The component can be for example, a Leuco dye. The gutta-percha composition may include a trans 1,4-polyisoprene, wherein the temperature indicating additive is present in an amount of about 1% to about 70% by weight of the gutta-percha composition. The gutta-percha composition may also include a radiopacifier.

**[0010]** In another embodiment of the invention, a composition for filling a tooth root canal includes two different temperature indicating ingredients which indicate different temperature ranges. The two different temperature indicating ingredients may be for example, a Leuco dye and bismuth oxide.

**[0011]** In a further embodiment of the invention, a temperature indicating ingredient may be a wax which blooms to the surface and when warm, producing a glossy surface to the gutta-percha. Such a material may be used with a second temperature indicating ingredient such as a dye or bismuth oxide that indicates a different temperature.

**[0011a]** In another embodiment, the invention relates to a method for filing a root canal comprising the steps of: providing composition having a gutta-percha component and a first temperature indicating component, the composition having a first color; heating the composition until the composition changes from the first color to a desired color, wherein the desired color indicates that the composition is at a predefined temperature which results in the composition having optimum flow properties; filling the root canal with the composition having the desired color with optimum flow properties.

## **PREFERRED EMBODIMENTS FOR CARRYING OUT THE INVENTION**

**[0012]** According to the present invention, an endodontic obturation material is provided that indicates the current temperature of the material by a relative color indication. The Temperature Indicating material of the invention was developed to be

used with obturator devices because it was observed that clinicians often touch the gutta-percha with their finger to make sure the gutta-percha was hot and soft. The desire was to provide proof the oven or other conventional heating method or device properly heated the gutta-percha and to avoid any unnecessary contamination by foreign material or bacteria of the gutta-percha prior to inserting it into the prepared root canal.

**[0013]** Temperature indicating gutta-percha is a unique formulation with the same physical and thermal properties of otherwise conventional obturation materials such as Thermafil™ gutta-percha available from DENTSPLY SIRONA of York, PA. These gutta-percha materials have good tack and flow once the alpha phase process has been completed. The alpha phase process is a long term (8 to 12 hour) bake at elevated temperature (115 to 160°C). Many

standard gutta-percha formulations separate, even producing a brown oil at these temperatures. The remaining gutta-percha itself becomes brittle.

**[0014]** According to the present invention, gutta-percha survives the alpha phase process without separating or producing the brown oil and the gutta-percha does not become brittle. The thermal properties of the inventive gutta-percha materials after the alpha phase process are changed. The melt flow increases dramatically ( $\leq 5$  to  $>50$ g/10min at  $180^{\circ}\text{C}$ ). The change to the conventional materials includes a certain ratio of processing aids which are shown in the FORMULATION section below.

**[0015]** Another inventive characteristic of the present materials is a temperature indicating property. The following are examples of these gutta-percha formulations.

**[0016]** Example 1 – replace the majority of the radiopacifier used in a conventional gutta-percha material with bismuth oxide. Standard gutta-percha uses zinc oxide and barium sulfate to generate a radiopacity of 6mmAl (mm of aluminum). The bismuth oxide is known to be a good radiopacifier however it is also an indicator of temperature. At low temperatures it is a mild yellow color. At elevated temperature it is a bright yellow color.

**[0017]** Example 2 – replace the conventional colorant with thermochromic dyes. A red colorant is often used in standard gutta-percha to give it a standard and well-known pink color. Thermochromic dyes used in the present invention are designed to change from a color to clear at a specified temperature. They preferably fall into two temperature ranges; 40 to  $50^{\circ}\text{C}$  and 60 to  $70^{\circ}\text{C}$ . These temperature ranges are ideal for gutta-percha obturation materials. Gutta-percha begins to melt at about  $42^{\circ}\text{C}$ . The 40 to  $50^{\circ}\text{C}$  dye indicates the gutta-percha has melted and the 60 to  $70^{\circ}\text{C}$  dye indicates the gutta-percha is much hotter than needed to melt.

Color:

Example 1 – Bismuth Oxide

Ingredients	Weight %
Trans-1, 4 - polyisoprene	25
Zinc Oxide	28.5
Bismuth Oxide	36
Antioxidant	1
Antiozidant	2.5
Processing Aid	1.5
White Colorant	5
Red Colorant	0.5
Total	100

## Example 2 – Thermochromic Dye

Ingredients	Weight %
Trans-1, 4 - polyisoprene	25
Zinc Oxide	51
Barium Sulfate	10
Antioxidant	1
Antiozidant	2.5
Processing Aid	1.5
White Colorant	5
TP060B Blue	2
TP047R Red	2
Total	100

Another exemplary formulation is shown below in Table 1.

Table 1: Example Formulation

Ingredient	Weight %
Trans-1,4-polyisoprene	24.2
Zinc Oxide	70.7
QCR TP047K (47C Black Thermo)	4.9
Total	100

**[0018]** The trans-1,4-polyisoprene is first baked in a 130°C oven for 7.5 hours. The baked polyisoprene was then allowed to cool for at least 24 hours. The polyisoprene was placed into a 71°C oven for a half-hour, and then the polyisoprene was formed into a ball and placed back into the oven. After a half-hour, the polyisoprene was removed from the oven and placed onto a heated two-roll mill that was set to 60°C. The Zinc Oxide was then added to the polyisoprene on the mill a little at a time. Once all of the Zinc Oxide was incorporated, the material was cross-cut at least 5 times. The QCR TP047K (47C Black Thermo) was then added in the same way as the Zinc Oxide. Once all of it was added, the formulation was again cross-cut at least 5 times in order to fully mix in the colorant. The formulation was then pigged at least 3 times, and then removed from the mill, laid down on a flat surface covered with release liner, and allowed to cool for at least 24 hours.

**[0019]** The material produced in Table 1 was then converted into pellets with a nominal diameter of 1mm and 14mm length. The pellets were then applied to carriers. This process can be used to produce finished obturators.

**[0020]** The pellets produced in the above example can also be loaded into cartridges and used in obturation devices such as the Calamus™ brand obturation devices available from DENTSPLY International of York, PA.

**[0021]** Table 1 presents an example of the present invention. Table 2 presents several examples with a similar functionality.

Table 2: Several Example Formulations

Ingredient	Example 1	Example 2	Example 3	Example 4
Trans-1,4-polyisoprene	25%	25%	25%	25%
Zinc Oxide	70%	65%		65%
Leuco Dye 47°C	5%	5%		
Leuco Dye 69°C		5%		
Bismuth Oxide			75%	
Wax				10%
Total	100%	100%	100%	100%

**[0022]** Example 1 has already been explained. Example 2 has the unique property to identify when the material is not only warm so that it is soft but also when it is too hot and may be too runny. The material is a bas color about 60°C. Between 47 and 69°C it is the color of the Leuco Dye 69°C. And the gutta-percha is the optimum temperature with the optimum properties. As the material cools to below 47°C the color changes to a blend of Leuco Dye 47°C and Leuco Dye 69°C (red and blue = purple).

**[0023]** Example 3 has been explained that bismuth oxide will also indicate a temperature change and doubles as a radiopacifier.

**[0024]** Example 4 indicates the gutta-percha is heated by the blooming wax melting. This would provide a glassy look to the surface.

**[0025]** It is therefore evident that an endodontic obturation material having desirable and inventive characteristics has been provided by the present invention. The invention has been characterized herein by way of examples and discussion for exemplary purposes only.

CLAIMS:

1. A composition for filling a tooth root canal comprising a gutta-percha component and a temperature indicating component.
2. A composition as in claim 1, wherein said gutta-percha component includes trans 1,4-polyisoprene.
3. A composition as in claim 1 or 2 wherein said temperature indicating component is a thermochromic dye.
4. A composition as in claim 3 wherein said thermochromic dye is present in an amount of from about 1 to about 70 percent by weight of said composition.
5. A composition as in any one of claims 1 to 4 further comprising a second temperature indicating component, wherein said temperature indicating component and said second temperature indicating components provide temperature indication at at least two different temperature ranges from each other.
6. A composition as in claim 5, wherein said temperature indicating component changes color upon reaching a temperature of from about 40 to about 50 degrees C, and said second temperature indicating component changes color upon reaching a temperature of from about 60 to about 70 degrees C.
7. A composition as in claim 5 or 6 wherein said temperature indicating component is a thermochromic dye and said second temperature indicating component is bismuth oxide.
8. A composition as in claim 1 or 2, wherein said temperature indicating component is a wax which blooms to the surface when warmed, producing a glossy surface to the gutta-percha.
9. Use of a composition for filling a root canal, said composition comprising a gutta-percha component and a temperature indicating component,

wherein the composition has a first color; and

wherein the composition changes color when heated from the first color to a desired color; and

wherein the desired color indicates that the composition is at a predefined temperature at which the composition has optimum flow properties; and

wherein the composition with the desired color and optimum flow properties is for use in filling a root canal; and

wherein the temperature indicating component is a thermochromic dye.

10. Use of a composition for filling a root canal as in claim 9, wherein said gutta-percha component includes trans 1,4-polyisoprene.

11. Use of a composition for filling a root canal as in claim 9 or 10, wherein said thermochromic dye is present in an amount of from about 1 to about 70 percent by weight of said composition.

12. Use of a composition for filling a root canal as in any one of claims 9 to 11, wherein said composition further comprises a second temperature indicating component, wherein said temperature indicating component and said second temperature indicating components provide temperature indication at at least two different temperature ranges from each other.

13. Use of a composition for filling a root canal as in claim 12, wherein said temperature indicating component changes color upon reaching a temperature of from about 40 to about 50 degrees C, and said second temperature indicating component changes color upon reaching a temperature of from about 60 to about 70 degrees C.

14. Use of a composition for filling a root canal as in claim 12 or 13, wherein said temperature indicating component is a thermochromic dye and said second temperature indicating component is bismuth oxide.

15. Use of a composition for filling a root canal, said composition comprising a gutta-percha component and a temperature indicating component, wherein said temperature indicating component is a wax which blooms to the surface when warmed producing a glossy surface to the gutta-percha to indicate that the composition is at a predefined temperature at which the composition has optimum flow properties.

16. Use of a composition for filling a root canal as in any one of claims 9 to 15, wherein said gutta-percha component includes trans 1,4-polyisoprene.

17. Use of a composition for filling a root canal as in any one of claims 9 to 15, wherein said gutta-percha component includes cis 1,4-polyisoprene.

18. A composition as in any one of claims 1 to 6, further comprising a radiopacifier.

19. Use of a composition as in any one of claims 9 to 13, wherein the composition further comprises a radiopacifier.