

UNITED STATES PATENT OFFICE

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DENTAL PULP CAPPING PREPARATION

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19 Claims. (Cl. 106—35)

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This invention relates to a dental pulp capping preparation to be used in the art of dentistry, and more particularly to a new combination of medicinal agents and chemicals for forming a dental pulp capping agent and/or sedative cement having for its purpose a more successful capping and preserving of dental pulp in the filling of cavities in the teeth.

This application is filed as a continuation in part of my copending application filed December 17, 1943, Serial No. 514,655 now abandoned.

Dental pulp capping has long been practiced to preserve the dental pulp closely approached or exposed in dental operative procedures. In the capping processes heretofore employed, cement or capping agents, for example, mixture of zinc oxide or other metallic oxides and oil of cloves or eugenol were inserted within the tooth over the exposed pulp. Few, if any, of these cements or capping agents can be used without attendant disadvantages or irritation on tooth structure due to heat generated while hardening and/or pressure on the dental tissue. For example, the mixture of zinc oxide and oil of cloves or eugenol is a combination which must be mixed so thick that the insertion within the tooth many times causes pressure on the dental pulp, producing ache, often causing the loss of the dental pulp and necessitating the extraction of the tooth. Zinc oxide and eugenol mixture is very slow in its setting or hardening, taking many minutes to hours to reach its maximum hardness, during which time the patient can and does chew upon the tooth under anaesthesia, forcing the still soft material into the pulp canal with the possibility of destroying the pulp by a second method. Because of the slow reaction between these materials, the permanent filling cannot be immediately inserted in the tooth lest the pulp cap is forced into or upon the pulp, thus necessitating return of the patient for another sitting for final filling of the tooth, causing a loss of time both to the dentist and the patient.

Possibly 40% of exposed dental pulps are saved by the use of zinc oxide and eugenol, notwithstanding the above reasons for failure. It would be of value and advantage to the dentist and patient if dental pulp capping preparation could be made to permit the insertion of the material in the tooth over the dental pulp without pres-

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sure, yet have sufficient speed of set and hardening to permit final filling of the tooth at the same sitting of the patient, and it is the purpose of the present invention to do this.

I have found that the heretofore failures in the saving of the dental pulp can be prevented or at least greatly reduced by the use of a material more nearly the chemical nature of the dentine, and preferably one of alkaline nature to neutralize the acid of acid-forming bacteria and the bacteria themselves, and by the addition of other chemicals it is possible to prepare a pulp capping material that may be mixed to a very thin consistency causing no detrimental pressure on the dental pulp in the insertion operation. By means of my particular chemical mixture and obvious variations thereof the pulp capping material will set or harden in one to four minutes or more as desired and when set it is sufficiently strong, hard and inflexible to support most dental fillings and to sufficiently withstand compression or bending or on into the dental pulp in the further filling operation, thereby reducing the time and number of sittings necessary to the filling of the tooth in question, saving time for the dentist and patient. I have also found that materials may be introduced into the mixture to stimulate the formation of normal secondary dentine in the dental pulp canal, thus restoring the tooth to a condition approaching its normal condition and materials to give the mixture proper color and radiopacity.

Since there are many attendant advantages in a quick setting pulp capping material, I prefer to provide a mixture of dry chemicals in the form of a powder and other chemicals in the form of a liquid which may be proportioned for mixing to provide the consistency desired. The powder portion of the formula made in accordance with the invention contains in its preferred form as the principal ingredient an alkaline material such as a metallic hydroxide to produce the alkalinity necessary to neutralize the acid of acid-forming bacteria and the bacteria themselves. The preferred alkaline material which may be suitably used in the new capping material is calcium hydroxide, and tests prove that calcium hydroxide mixed with liquid eugenol or guaiaccol produces a rapid setting cement of sufficient hardness to withstand pressures necessary in in-

serting permanent fillings in teeth. Therefore, the principal ingredients of the liquid portion of the capping preparation formula are preferably eugenol or guaiacol or mixtures of both. Whereas a mixture of calcium hydroxide and eugenol or guaiacol, or both eugenol and guaiacol, may be used successfully to cap the dental pulp when atmospheric conditions are particularly favorable, that is, when the relative humidity is very low, it has been found favorable to add various quantities of linseed oil to the guaiacol or eugenol for the reason herein set forth.

Calcium hydroxide and eugenol when used for medically capping the exposed dental pulp under atmospheric conditions with humidities varying from 40 to 70 percent, have a very rapid chemical reaction. These same chemicals when used under atmospheric conditions with humidities of 10 to 30 percent do not react rapidly and can be used unmodified to successfully cap the exposed pulp, offering ample time to mix and insert the capping material into the tooth, this without generation of noticeable heat. No agent such as linseed or any other oil is necessary in the mixture during periods of low humidities.

Mixtures of calcium hydroxide and eugenol or guaiacol alone, and those of calcium hydroxide, eugenol and about 24 percent of linseed oil, and guaiacol and 35 percent of linseed oil, under conditions of low humidity set with about the same speed, requiring more than an hour to see setting change in any such mixture.

Therefore, apparently linseed oil has little or nothing to do with the chemical reaction of calcium hydroxide and eugenol or guaiacol, and in no way retards the chemical action of these chemicals directly. The function of the oil is to prevent the action of atmospheric moisture from attacking the liquid mixture, this moisture being the agent that causes rapid setting reaction.

The action of the linseed oil is demonstrated by the following test:

Through a rubber diaphragm and within a mixing receptacle, the air of which was quite completely dried by passing compressed air first through a container of calcium chloride and then through colloidal silicon dioxide, three similar mixes of pulp capping materials were made. One contained only calcium hydroxide and eugenol; another contained 23 percent of linseed oil in the eugenol and calcium hydroxide; the third contained guaiacol and 35 percent of linseed oil and calcium hydroxide. After contacting said mixtures in the drying receptacle for a period of sixty minutes no visible setting had occurred in any mix.

All mixes were removed simultaneously into a room in which the humidity was 60 percent. Within two minutes the material containing no linseed oil showed thickening signs of setting. In five minutes this material was of thick creamy nature. In nine minutes this material was of thick putty consistency. Only at the end of nine minutes was visible change noticed in the mixtures containing the linseed oil.

This test shows that linseed oil does not inhibit the setting of the calcium hydroxide and eugenol or guaiacol per se, but shows definitely that it inhibits or prevents to a necessary degree the attack of atmospheric moisture on the fresh liquid mixture of calcium hydroxide and eugenol or guaiacol.

Other known oils, for an example motor oil, olive oil, hydrogenated vegetable oil, tung oil, or poppy seed oil may be used in the eugenol or

guaiacol, and the speed of set is slowed thereby, but the set mixture is more crumbly and more easily fractured than when linseed oil is used. Therefore the addition of linseed oil, and it has been found that either boiled or raw oil may be used, has another specific function. When this oil is used with guaiacol or eugenol and calcium hydroxide the set mixture is much harder than with said other named oils. When linseed oil is used with eugenol or guaiacol the set hardened mixture is only slightly to no softer than when no linseed oil is used, a very vital result for a successful pulp capping material.

It is understood that sufficient linseed oil has not been added to the eugenol or guaiacol to prevent the attack of the moisture vapor on the liquid mixture placed in the mouth to prevent proper setting therein, nor will the amount herein stated prevent rapid setting in relative humidities close to 100 percent.

It has been found that suitable liquids to be used with calcium hydroxide alone or with its modifying chemicals are as follows: Eugenol 100 parts, linseed oil 15 to 30 parts; guaiacol 100 parts, linseed oil 25 to 50 parts. Eugenol 1 to 100 parts, guaiacol 100 to 1 parts, linseed oil 15 to 50 parts, said parts of the liquid components being volume. It is understood that these proportions may be varied depending upon atmospheric condition. The quantities of linseed oil as stated sufficiently prevent the attack of atmospheric moisture of areas of average relative humidity, that the liquid mix may be carried to the tooth with ample time, but when so placed the mixture will set hard in from one to four minutes, a very valuable characteristic in pulp capping agents.

A small quantity of powdered calcium phosphate, preferably 1 to 10 parts by weight to 100 parts by weight of calcium hydroxide, is desirable in the capping agent as the calcium phosphate being essentially the same composition as dentine may act as a splint or stimulant for the formation of secondary dentine in the pulp canal by the same principles by which bone splints are used in bone surgery to stimulate the growth of bones. Although it is desirable to incorporate small amounts of calcium phosphate in the powder it is not essential and successful cappings are possible without it.

Calcium hydroxide, eugenol and linseed oil set to a dark greenish brown color. It is usually desirable to obtain a hard set capping material approximately the color of natural teeth. Therefore, the color of the capping material is preferably controlled by incorporating in certain formulae a suitable amount of a white agent. Such control agent should provide beneficial results as regards color without great detrimental effects on the setting properties and hardness of the capping material or cement. I have found that titanium dioxide produces very good results with eugenol, linseed oil and calcium hydroxide mixtures. However, I prefer to add a small quantity of yellow ochre to provide a more perfect tooth tint and to make the dry powder visibly different in color than other capping powders used in the dental office. By the addition of these coloring agents the color characteristics of the capping material are susceptible to close control. In general, I have found that from nine (9) to fifteen (15) parts by weight of titanium dioxide and $\frac{1}{16}$ to 1 part by weight of ochre (yellow) to 100 parts by weight of calcium hydroxide effects desirable results when eugenol and linseed oil is the liquid, although more or less may be used depending

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upon the particular color characteristics desired.

A set mixture of calcium hydroxide, titanium dioxide, calcium phosphate and ochre (yellow) mixed with eugenol and/or guaiacol and linseed oil is radiolucent and when a tooth is X-rayed the dental capping material is falsely represented beneath the metal fillings as carious dentine. I have discovered that additions of five (5) to fifteen (15) parts by weight of barium sulphate to 100 parts by weight of calcium hydroxide produces sufficient radiopacity to cause the set material to appear the same in density as the undecayed tooth or to appear as a whitish layer beneath the various fillings. Radio opaque materials other than barium sulphate may also be used to produce the proper X-ray interference as desired, such materials being metallic particles such as finely divided copper, silver or silver alloy particles. It has also been found that amounts of abietic acid may be added to the above powders to make the set material more adhesive and/or harder. One to twenty-five parts of abietic acid may be used.

It has been further discovered that although excellent pulp capping agents can be made by combinations of the chemicals as already set out, an additional group of formulae can also be used successfully. When the liquid guaiacol with linseed oil is used with calcium hydroxide alone a light grey solid is formed. It follows from this that little to no white titanium dioxide need be used, but pure zinc oxide may be substituted in the proper amounts for a triple reason. Zinc oxide is first, white; second, radio opaque, and third, it apparently goes into the chemical reaction with guaiacol to make a more hardened end result. Being radio opaque it may also replace the barium sulphate. Yellow ochre may be added to this formula or not, at will. To make the set mixture of calcium hydroxide and zinc oxide, with or without ochre, more adhesive powdered abietic acid, which dissolves in either of the above liquids, may be added to the various powders in the amount of 1 to 25 parts to 100 parts calcium hydroxide.

Guaiacol and linseed oil as set out react with any of the previously described powders to produce set hardened products that are of more grayish tint than the tooth color. Small quantities of eugenol added to the guaiacol linseed oil mixture tend to produce a set product of a more yellow nature, more the color of the usual dental cements and tooth tint. Such a liquid would have a formula as follows: guaiacol 100 parts, eugenol 1 to 15 parts, linseed oil 15 to 50 parts. As the eugenol is increased in the guaiacol so is the yellow tint deepened. However, except as regards color any mixture of eugenol and guaiacol with 15 to 50 parts of linseed oil can be used successfully as a pulp capping liquid.

It is understood that successful cappings may be made with calcium hydroxide and eugenol or guaiacol alone when proper atmospheric conditions are present. The other chemicals have been added and varied as shown, to adapt the preparation to particular conditions, practice, and desires of dentists and patients. However, each of the other chemicals may be omitted or added at will with apparently the same successful result, except as already set out, not excluding calcium phosphate.

The following examples illustrate exemplary compositions of the powder and liquid components used in the capping material of the invention, it being understood that any of the powder

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components may be used with any of the liquid components and that the invention is not to be in any way limited by the examples:

	Parts
5 Calcium hydroxide (powder)	100
Eugenol (liquid)	100

Other powder components

	Parts by weight
10 Calcium hydroxide	100
Titanium dioxide	9 to 15
Calcium phosphate	1 to 10
Barium sulphate	5 to 15
Ochre (yellow)	1 to 2

	Parts by weight
15 Calcium hydroxide	100
Titanium dioxide	9 to 15
Calcium phosphate	1 to 10
Metallic-silver particles	1 to 5
20 Yellow ochre	1 to 2

	Parts by weight
Calcium hydroxide	100
Zinc oxide	5 to 25
25 Ochre	1 to 2

	Parts by weight
Calcium hydroxide	100
Zinc oxide	5 to 25
Abietic acid	1 to 25
30 Ochre	1 to 2

	Parts by weight
Calcium hydroxide	100
Titanium dioxide	9 to 15
Calcium phosphate	1 to 10
35 Barium sulphate	5 to 15
Ochre	1 to 2
Abietic acid	1 to 25

	Parts by weight
40 Calcium hydroxide	100
Titanium dioxide	9 to 15
Abietic acid	1 to 25

	Parts by weight
45 Calcium hydroxide	100
Titanium dioxide	9 to 15
Calcium phosphate	1 to 10
Abietic acid	1 to 25

	Parts by weight
50 Calcium hydroxide	100
Titanium dioxide	9 to 15
Calcium phosphate	1 to 10
Barium sulphate	5 to 15
Abietic acid	1 to 25

	Parts by weight
55 Calcium hydroxide	100
Titanium dioxide	9 to 15
Calcium phosphate	1 to 10
Metallic particles	1 to 5
60 Abietic acid	1 to 25
Yellow ochre	1 to 2

Other liquid components

	Parts by volume
65 Eugenol U. S. P.	100
Linseed oil (boiled)	15 to 30

	Parts by volume
Guaiacol	100
70 Linseed oil	25 to 50

	Parts by volume
Guaiacol	100
Eugenol	1 to 15
75 Linseed oil	15 to 50

	Parts by volume
Guaiacol	1 to 99
Eugenol	99 to 1
Linseed oil	15 to 50

It has been further discovered that abietic acid may be added to or dissolved in the eugenol, guaiacol, linseed oil liquids of the above examples of the liquid components to obtain a more adhesive and/or harder pulp capping material, the results being comparable to those obtained when the abietic acid is incorporated in the powders. The quantity of abietic acid in the liquid component is limited according to the quantity in the powder component or vice versa whereby the final mixture of the liquid and powder contains approximately one (1) to twenty-five (25) parts by weight of abietic acid to one hundred (100) parts by weight of calcium hydroxide. The following are examples of suitable liquids containing abietic acid:

	Parts by volume
Eugenol	100
Linseed oil	15 to 30
Abietic acid	1 to 15

	Parts by volume
Guaiacol	100
Linseed oil	25 to 50
Abietic acid	1 to 15

The procedure for using the dental pulp capping material is as follows:

After the pulp in a tooth is exposed in the operative procedure, the tooth is dried. A desired amount of a selected powder component of the dental pulp capping material is then mixed with a selected liquid component to form a cream-like consistency only sufficiently thick or viscous to adhere to an instrument used to carry it to the tooth. A small quantity is placed over the exposure of the pulp and quickly teased or flowed over the pulp and adjacent cavity walls by the instrument or by a tiny pellet of cotton held by cotton pliers, causing no appreciable pressure on the pulp.

Setting of the capping material requires one to four or five minutes. At the end of this time the excess material is removed, the cavity completed in the usual well known manner and silver, silicate, or cement fillings may be inserted, or the wax pattern for gold inlays may be made immediately the setting of the capping material is complete. Temporary fillings of gutta percha or zinc oxide eugenol may be inserted over the capper when lack of time prevents the insertion of the permanent filling.

Special emphasis is brought to bear that by my combination of powder and liquids a tooth pulp protecting and capping material can be mixed very thin and carried into the cavity in this state and within one to four minutes the mixture hardens or sets sufficiently to withstand the pressure of inserting any known filling except gold foil. Unlike other capping agents, no cement is usually needed over the capper to protect it from compression or bending onto or into the pulp when inserting the filling immediately after the pulp is capped and setting occurs.

Furthermore, my pulp protecting and capping materials can be used as sedative cements and insulating materials in the deep cavity having no exposure of the pulp. It is further understood that germicidal agents, sulphanilamide and other derivatives, may be added for bacterial action. It might also be mentioned that various

combinations of the drugs may be employed if desired, the amount of which drugs or combinations thereof required to obtain the beneficial results will not appreciably affect the setting properties and hardness of the capping material.

What I claim and desire to secure by Letters Patent is:

1. A dental plug capping material consisting of approximately equal parts of calcium hydroxide and eugenol.

2. A dental pulp capping material consisting of a powder and liquid in combination, the powder containing one hundred (100) parts by weight of calcium hydroxide and the liquid containing one hundred (100) parts by volume eugenol and fifteen (15) to thirty (30) parts by volume of linseed oil.

3. A dental pulp capping material consisting of a powder and liquid in combination, the powder containing one hundred (100) parts by weight of calcium hydroxide, nine (9) to fifteen (15) parts titanium dioxide, one (1) to five (5) parts calcium phosphate, and the liquid containing one hundred (100) parts by volume eugenol and fifteen (15) to thirty (30) parts by volume of linseed oil.

4. A dental pulp capping material consisting of a powder and liquid in combination, the powder containing one hundred (100) parts by weight of calcium hydroxide, nine (9) to fifteen (15) parts titanium dioxide, one (1) to five (5) parts calcium phosphate, and five (5) to fifteen (15) parts barium sulphate, all of said parts being by weight, and the liquid containing one hundred (100) parts by volume eugenol and fifteen (15) to thirty (30) parts by volume of linseed oil.

5. A dental pulp capping material consisting of a powder and liquid in combination, the powder containing one hundred (100) parts by weight of calcium hydroxide, nine (9) to fifteen (15) parts titanium dioxide, one (1) to five (5) parts calcium phosphate, five (5) to fifteen (15) parts barium sulphate, one-sixteenth (1/16) to two (2) parts yellow ochre, and the liquid containing one hundred (100) parts by volume eugenol and fifteen (15) to thirty (30) parts by volume of linseed oil.

6. A dental pulp capping material consisting of a powder and liquid in combination, the powder containing one hundred (100) parts by weight of calcium hydroxide, nine (9) to fifteen (15) parts titanium dioxide, one (1) to ten (10) parts calcium phosphate, one-sixteenth (1/16) to five (5) parts metallic silver particles, one-sixteenth (1/16) to two (2) parts yellow ochre, all of said parts being by weight, and the liquid containing one hundred (100) parts by volume eugenol and fifteen (15) to thirty (30) parts by volume linseed oil.

7. A dental pulp capping material consisting of a powder and liquid in combination, the powder containing one hundred (100) parts calcium hydroxide, nine (9) to fifteen (15) parts titanium dioxide, one (1) to twenty-five (25) parts abietic acid, all of said parts being by weight, and the liquid containing one hundred (100) parts by volume eugenol and fifteen (15) to thirty (30) parts by volume of linseed oil.

8. A dental pulp capping material consisting of a powder and liquid in combination, the powder containing one hundred (100) parts calcium hydroxide, nine (9) to fifteen (15) parts titanium dioxide, one (1) to ten (10) parts calcium phosphate, five (5) to fifteen (15) parts barium sulphate, one (1) to twenty-five (25) parts abietic

acid, all of said parts being by weight, and the liquid containing one hundred (100) parts by volume eugenol and fifteen (15) to thirty (30) parts by volume of linseed oil.

9. A dental pulp capping material consisting of a powder and liquid in combination, the powder containing one hundred (100) parts calcium hydroxide, five (5) to twenty-five (25) parts zinc oxide, one-sixteenth ($\frac{1}{16}$) to two (2) parts yellow ochre, all of said parts being by weight, and the liquid containing one hundred (100) parts by volume eugenol and fifteen (15) to thirty (30) parts by volume of linseed oil.

10. A dental pulp capping material consisting of a powder and liquid in combination, the powder containing one hundred (100) parts calcium hydroxide, five (5) to twenty-five (25) parts zinc oxide, one (1) to twenty-five (25) parts abietic acid and one-sixteenth ($\frac{1}{16}$) part yellow ochre and the liquid containing one hundred (100) parts by volume guaiacol and twenty-five (25) to fifty (50) parts by volume linseed oil.

11. A dental pulp capping material consisting of a powder and liquid in combination, the powder containing one hundred (100) parts calcium hydroxide, and five (5) to fifteen (15) parts barium sulphate, all of said parts being by weight, and the liquid containing one (1) to ninety-nine (99) parts by volume guaiacol, ninety-nine (99) to one (1) part by volume eugenol and fifteen (15) to fifty (50) parts by volume linseed oil.

12. A dental pulp capping material consisting of a powder and liquid in combination, the powder containing one hundred (100) parts calcium hydroxide, one (1) to ten (10) parts calcium phosphate, all parts being by weight, and the liquid containing one hundred (100) parts by volume of eugenol, and fifteen (15) to thirty (30) parts by volume linseed oil.

13. A dental pulp capping material consisting of a powder and liquid in combination, the powder containing one hundred (100) parts by weight of calcium hydroxide and the liquid containing one hundred (100) parts by volume guaiacol, one (1) to fifteen (15) parts by volume eugenol, and fifteen (15) to fifty (50) parts by volume of linseed oil.

14. The liquid of a dental pulp capping material including one hundred (100) parts by volume eugenol, fifteen (15) to thirty (30) parts by volume linseed oil, and one (1) to fifteen (15) parts by volume abietic acid.

15. The liquid of a dental pulp capping material including one hundred (100) parts by volume guaiacol, twenty-five (25) to fifty (50) parts by

volume linseed oil, and one (1) to fifteen (15) parts by volume abietic acid.

16. A dental pulp capping material consisting of a powder and liquid in combination, the powder containing calcium hydroxide 100 parts by weight, titanium dioxide 9 to 15 parts by weight, yellow ochre $\frac{1}{16}$ th to 2 parts by weight, calcium phosphate 1 to 10 parts by weight, barium sulphate 5 to 15 parts by weight, and the liquid containing eugenol 100 parts by volume, linseed oil 15 to 30 parts by volume, and a germicidal agent.

17. A dental pulp capping material consisting of a powder and liquid in combination, the powder containing calcium hydroxide 100 parts by weight, yellow ochre $\frac{1}{16}$ th to 2 parts by weight, calcium phosphate 1 to 10 parts by weight, barium sulphate 5 to 15 parts by weight and the liquid containing eugenol 100 parts by volume and linseed oil 15 to 30 parts by volume.

18. A dental pulp capping material consisting of a powder and liquid in combination, the powder containing calcium hydroxide 100 parts by weight, yellow ochre $\frac{1}{16}$ th to 2 parts by weight, calcium phosphate 1 to 10 parts by weight, barium sulphate 5 to 15 parts by weight and the liquid containing eugenol 100 parts by volume, linseed oil 15 to 30 parts by volume and a germicidal agent.

19. A dental pulp capping material consisting of a powder and liquid in combination, the powder containing calcium hydroxide 100 parts by weight, zinc oxide 5 to 25 parts by weight, yellow ochre $\frac{1}{16}$ th to 2 parts by weight, and the liquid containing eugenol 100 parts by volume, and linseed oil 15 to 30 parts by volume.

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