PATENT SPECIFICATION

NO DRAWINGS

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COMPLETE SPECIFICATION

Toothpastes

We, INTERNATIONAL CHEMICAL COMPANY, LIMITED, of 12 Chenies Street, London, W.C.1., a British Company, do hereby declare the invention for which we pray that a patent may be granted to us and the method by which it is to be performed, to be particularly described in and by the following statement: —

This invention relates to toothpastes and to processes for preparing them.

In Patent Specification Serial No. 999,857 there is claimed a toothpaste comprising a calcium or magnesium salt abrasive having a water solubility of less than 0.01g/100 cc. at 20°C, a therapeutic amount of an alkali metal fluoride and sufficient silica in water soluble form to reduce the partial inactivation of the fluoride by the abrasive. We have now found that silica in water soluble form also reduces the partial inactivation of the fluoro-phosphate in toothpastes in which an alkali metal fluoro-phosphate is used as a source of fluoro-phosphate ions, in the presence of calcium salts of low solubility. In addition, the presence of the silica in water soluble form prevents the corrosion of the aluminium in unlined aluminium tubes containing the toothpaste.

By silica in water-soluble form is meant a silicic acid or an alkali metal salt of silicic acid which is soluble in water to give either a true solution or colloidal solution.

The invention provides a toothpaste comprising a calcium or magnesium salt abrasive, a therapeutic amount of an alkali metal fluorophosphate and sufficient silica in water-soluble form (as defined above) to reduce the inactivation of the fluorophosphate by the abrasive.

Desirably the abrasive has a water solubility of less than 0.03g/100cc. at 20°C and preferably less than 0.01g/100 cc. at 20°C.

As the alkali metal fluorophosphate it is preferable to use sodium monofluorophosphate, but potassium or lithium monofluorophosphate can also be employed. Preferably the toothpaste is one prepared by incorporation of from 0.01 to 0.4%, especially 0.05 to 0.15% by weight of fluorine as alkali metal fluorophosphate; these concentrations of fluorine can be obtained by adding 0.4 to 1.2% by weight of sodium monofluorophosphate to the toothpaste.

The silica can be incorporated in the form of a silica sol, preferably of alkaline pH, as a water-dispersible aerogel, or as an aqueous solution of a sodium or other alkali metal silicate, for instance Sodium silicate having a sodium oxide to silica ratio by weight of from 1:3.1 to 1:3.4. Conveniently an aqueous solution of sodium silicate of the type known as water-glass is used. Preferably the toothpaste is one prepared by incorporation of from 0.03 to 1.5% or as much as 3%, especially 0.15 to 0.6% by weight of silica in water-soluble form.

The abrasive preferably comprises calcium carbonate, and it is convenient to have the abrasive consisting substantially wholly of calcium carbonate. However, the other abrasives, preferably of low water-solubility, for example tricalcium orthophosphate and calcium pyrophosphate, can be employed as ingredients of the abrasive, for instance in an abrasive containing a major proportion of calcium carbonate. Calcium carbonate has a solubility of about 0.0015g./100 cc. at 20°C. and various figures up to 0.003 g./100 cc. have been reported for the solubility of tricalcium orthophosphate. A more water-soluble calcium abrasive which may be used is dicalcium orthophosphate. Preferably a toothpaste contains from 20 to 60% by weight of abrasive.

[*Price 4s. 6d.*]

The pH of a toothpaste of the invention is on the alkaline side of neutrality, and usually from 9 to 10.8, more often from 10.0 to 10.6. The pH of a toothpaste can be adjusted after mixing by the addition of alkali if desired. By pH of the paste is meant the pH as measured on the mixture obtained by dispersing the paste with 4 times its weight of water.

A toothpaste of the invention may also contain such other of the normal ingredients of dentifrices as are compatible with fluorophosphates, including humectants (for example, glycerol and sorbitol), detergents (for example, water-soluble alkali metal fatty acid salts, alkali metal high alkyl sulphates and sulphonates, and alkali metal salts of sulphonated monoglycerides of higher fatty acids), thickeners (for example, methyl cellulose, sodium carboxymethyl cellulose, and gum tragacanth), flavouring agents (for example saccharin, sodium cyclamate, oil of peppermint, oil of spearmint, and oil of anise), and preservatives. Usually from 0.5 to 5% of determent, and from 0.5 to 5% of thickener by weight of the paste are employed.

The invention also provides a toothpaste comprising calcium carbonate as abrasive, an alkali metal fluorophosphate and a water-soluble silicate, the fluorine content of the paste being from 0.025 to 0.1% by weight.

The invention also comprises a process of preparing a toothpaste in which the separate ingredients as described above are mixed together.

The following test illustrates the effect of sodium silicate in reducing the inactivation of monofluorophosphate by calcium carbonate. Compositions A and B were prepared from the following materials by mixing them together in the relative amounts stated.

	A	B
Sodium monofluorophosphate	0.76	0.76
Calcium carbonate	52.22	52.22
40% Aqueous sodium silicate (having an $Na_2O:SiO_2$ ratio of about 1:3.2)	Nil	1.0
Other normal toothpaste ingredients	to 100	to 100

The resulting pastes were analysed for their water-soluble fluorine content immediately after mixing. Analysis was carried out by dilution with an amount of water twice the weight of the paste sample, mixing, centrifugation and determination of the fluorine in the supernatant liquid.

Samples of the pastes were stored in closed containers for 6 months at room temperature and their water-soluble fluorine content again determined. The results were as follows, the water soluble fluorine found being given as the proportion (%) of the amount incorporated in the paste.

Composition	Total soluble fluorine (%) A	B
Freshly made	74	92
After 6 month's storage	58	78

This shows clearly the efficacy of the silicate in reducing the insolubilisation of the fluorophosphate.

The following compositions illustrate toothpastes in accordance with the invention and are prepared in conventional manner.

EXAMPLE 1

	%(W/W)
Glycerol	20.00
Sodium carboxymethylcellulose	0.85
Sodium methyl-p-hydroxybenzoate	0.05
Saccharin, soluble	0.12
Sodium lauryl sulphate	1.13
Calcium carbonate	52.22
Sodium silicate (40% Solids)	1.00
Sodium monofluorophosphate	0.76
Flavouring oils	1.00
Water	22.87
	100.00

EXAMPLE 2

	% W/W
Sorbitol	20.00
Sodium alginate	3.70
Sodium methyl *p*-hydroxy benzoate	0.05
Saccharin soluble	0.10
Sodium lauryl sulphate	2.00
Calcium carbonate	30.00
Flavouring oils	1.30
Sodium silicate (40% Solids)	1.00
Disodium phosphate	0.25
Titanium dioxide	1.00
Sodium monofluorophosphate	0.76
Water	39.84
	100.00

EXAMPLE 3

Glycerin	20.00
Irish Moss Extract	1.50
Sodium methyl *p*-hydroxy benzoate	0.05
Saccharin soluble	0.10
Sodium lauryl sulphate	2.00
Calcium carbonate	53.00
Flavouring oils	1.00
Sodium silicate (40% Solids)	1.00
Sodium monofluorophosphate	0.76
Water	20.59
	100.00

EXAMPLE 4

	%W/W
Glycerine	25.000
Sodium carboxymethylcellulose	1.200
Saccharin soluble	0.300
Methyl-*p*-hydroxybenzoate	0.050
Calcium carbonate	36.000
Sodium silicate (40% Solids)	1.000
Sodium lauryl sulphate	1.800
Flavouring oils	1.186
Titanium dioxide	0.500
Sodium monofluorophosphate	0.740
Water	32.224
	100.000

WHAT WE CLAIM IS:—

1. A toothpaste comprising a calcium or magnesium salt abrasive, an effective amount of an alkali metal fluorophosphate and sufficient silica in water-soluble form to reduce the inactivation of the fluorophosphate by the abrasive.

2. A toothpaste according to Claim 1, in which the alkali metal fluorophosphate is sodium monofluorophosphate.

3. A toothpaste according to any one of the preceding claims, in which the silica is incorporated in the form of an aqueous alkali metal silicate.

4. A toothpaste according to Claim 3, in which the alkali metal silicate is sodium silicate.

5. A toothpaste according to any one of the preceding claims, in which there is incorporated from 0.01 to 0.4% of fluorine as alkali metal fluorophosphate.

6. A toothpaste according to Claim 5, in which there is incorporated from 0.05 to 0.15% of fluorine as alkali metal fluorophosphate.

7. A toothpaste according to any one of the preceding claims, in which there is incorporated from 0.03 to 3% of silica in water soluble form.

8. A toothpaste according to Claim 7, in which there is incorporated from 0.15 to 0.6% of silica in water-soluble form.

9. A toothpaste according to any one of the preceding claims having a pH of from 9 to 10.8.

10. A toothpaste according to any one of the preceding claims in which the abrasive has a water solubility of less than 0.01 g./100c. at 20°C.

11. A toothpaste according to Claim 10 in which the abrasive comprises calcium carbonate.

12. A toothpaste according to Claim 11, in which the abrasive is substantially wholly calcium carbonate.

13. A toothpaste comprising calcium carbonate as abrasive, an alkali metal fluorophosphate and a water-soluble silicate, the fluorine content of the paste being from 0.025 to 0.1% of weight of the paste.

14. A toothpaste according to any one of claims 1—9, in which the abrasive comprises dicalcium orthophosphate.

15. A toothpaste containing an alkali metal monofluorophosphate and silica substantially as described in Example 1.

16. A toothpaste containing an alkali metal monofluorophosphate and silica substantially as described in Example 2.

17. A toothpaste containing an alkali metal monofluorophosphate and silica substantially as described in Example 3.

18. A toothpaste containing an alkali metal monofluorophosphate and silica substantially as described in Example 4.

19. A process for preparing a toothpaste according to any one of the preceding claims, characterised in that the ingredients of the paste are mixed together.

20. A toothpaste whenever prepared by a process according to Claim 19.

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EXAMPLE 4

	%W/W
Glycerine	25.000
Sodium carboxymethylcellulose	1.200
Saccharin soluble	0.300
Methyl-*p*-hydroxybenzoate	0.050
Calcium carbonate	36.000
Sodium silicate (40% Solids)	1.000
Sodium lauryl sulphate	1.800
Flavouring oils	1.186
Titanium dioxide	0.500
Sodium monofluorophosphate	0.740
Water	32.224
	100.000

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1. A toothpaste comprising a calcium or magnesium salt abrasive, an effective amount of an alkali metal fluorophosphate and sufficient silica in water-soluble form to reduce the inactivation of the fluorophosphate by the abrasive.

2. A toothpaste according to Claim 1, in which the alkali metal fluorophosphate is sodium monofluorophosphate.

3. A toothpaste according to any one of the preceding claims, in which the silica is incorporated in the form of an aqueous alkali metal silicate.

4. A toothpaste according to Claim 3, in which the alkali metal silicate is sodium silicate.

5. A toothpaste according to any one of the preceding claims, in which there is incorporated from 0.01 to 0.4% of fluorine as alkali metal fluorophosphate.

6. A toothpaste according to Claim 5, in which there is incorporated from 0.05 to 0.15% of fluorine as alkali metal fluorophosphate.

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8. A toothpaste according to Claim 7, in which there is incorporated from 0.15 to 0.6% of silica in water-soluble form.

9. A toothpaste according to any one of the preceding claims having a pH of from 9 to 10.8.

10. A toothpaste according to any one of the preceding claims in which the abrasive has a water solubility of less than 0.01 g./100c. at 20°C.

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14. A toothpaste according to any one of claims 1—9, in which the abrasive comprises dicalcium orthophosphate.

15. A toothpaste containing an alkali metal monofluorophosphate and silica substantially as described in Example 1.

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17. A toothpaste containing an alkali metal monofluorophosphate and silica substantially as described in Example 3.

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