PATENT SPECIFICATION

NO DRAWINGS

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COMPLETE SPECIFICATION

Dentifrice Compositions

We, UNILEVER LIMITED, a company registered under the laws of Great Britain, of Port Sunlight, Birkenhead, Cheshire, England, 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:—

The present invention relates to dentifrice compositions.

In accordance with the present invention there is provided a dentifrice composition containing as the sole or principal cleansing and polishing ingredient from 5% to 50%, and preferably from 8% to 17%, by weight of the dentifrice of particulate dehydrated silica gel having an average particle diameter from 2 to 20 microns, and preferably from 5 to 15 microns, and a surface area of at least 600 square metres per gram. The present invention is based on the surprising discovery that the defined dehydrated silica gel has a good cleansing ability and imparts a high lustre to dental enamel without excessively abrading the tooth enamel surface.

The particulate dehydrated silica gel may be prepared in the following manner: a silica hydrosol is prepared by mixing an aqueous sodium silicate solution with a mineral acid, such as sulphuric acid. The hydrosol formed in this process is allowed to gel under suitable conditions; the resulting mass of hydrogel is then broken up mechanically and is washed free of salt. The hydrogel is appropriately dried (such as at 100—120°C) by thermal means to give an amorphous granular product with random microporous structure and about 5% by weight residual water content. This is then reduced to the desired small particle size.

The preferred dehydrated silica gel cleansing and polishing ingredient has an average particle diameter of about 10 microns and a surface area of about 700 square metres per gram.

In order to maintain the desired texture and thixotropic behaviour, the dentifrice compositions of the invention may contain, in addition to the above described silica cleansing and polishing ingredient, from 0.5% to 20%, and preferably from 2% to 15%, by weight of the dentifrice, of a synthetic silica which has thickening and gelling properties. Such particulate synthetic silicas have an average particle diameter below 4 microns and a surface area of less than 400 square metres per gram. Examples include pyrogenic silicas and silica aerogels. These silicas do not remove the stains from teeth.

Small amounts up to 10% by weight of the dentifrice of conventional dental abrasives, such as water-insoluble sodium metaphosphate, can be included.

The dentifrice of the invention can also contain various optional ingredients, for instance a soap or synthetic detergent as foaming agent; flavouring materials; buffers; sweeteners, such as saccharin; humectants; preservatives; colouring materials; opacifying agents, such as titanium dioxide; and powdered polymers such as an alpha-olefin polymer, for example polyethylene.

The dentifrice may contain a carrier and softener and a binder in amounts to give the dentifrice a smooth texture and good flowability. Glycerine and sorbitol are preferred carriers and softeners, but others, for example propylene glycol and polyethylene glycol can also be employed. Examples of suitable binders that can be used are gum tragacanth, sodium carboxymethylcellulose, hydroxyethyl cellulose, carragheen and its derivatives, starch derivatives and locust bean gum.

There can also be included in the dentifrice compositions of the invention prophy-

[*Price 5s. 0d.*]

lactic or therapeutic agents, such as germicides, antibiotics, astringents or fluorine-containing compounds. Typical examples thereof include tyrothrycin, chlorophyllins, hexachlorophene, the sarcosinates, astringent salts and water-soluble ionizable fluorine-containing compounds, for example sodium fluoride, stannous fluoride, and sodium monofluorophosphate.

Such prophylatic or therapeutic agents are employed in a beneficial amount normally ranging from 0.01% to 2% by weight of the dentifrice calculated as fluoride ions in the case of water-soluble therapeutic fluorine-containing compounds. The carriers and softeners are generally employed in an amount from 5% to 70%, the binders in an amount from 0.5% to 30%, flavouring agents in an amount from 0.1% to 5%, water in an amount up to 60% by weight of the dentifrice, foaming agents in an amount from 0.01% to 6%, buffers in an amount from 0.02% to 10%, and preservatives in an amount from 0.01% to 2%, all percentages being by weight of the dentifrice.

In the examples illustrating the dentifrice compositions of the invention set forth below the following test procedures were employed.

Abrasion Test

Abrasion testing (enamel loss) was done by measuring the thickness of extracted human teeth before and after each brushing with a given dentifrice; any difference represented enamel loss. The test tooth was first conditioned by storage in tap water until the thickness measurements were constant and remained constant for three successive days. The tooth was mounted in the bottom of a metal cup directly below a revolving brush of the type used by dentists in prophylatic work. The slurry of the test product was then poured into the cup and the machine was operated. The cup and tooth were moved slowly back and forth beneath the revolving brush for a period of one hour. The tooth thickness was then measured again. The difference between the initial and final measurements of tooth thickness represents the abrasion loss. Results on three teeth were averaged to obtain the abrasion loss value. Acceptable abrasion loss values are considered to be from 0 to 5 microns.

Lustre Test

The lustre test to evaluate ability to improve tooth lustre was carried out as follows.

The labial surface of an extracted human anterior incisor was smoothed with sandpaper, ending with fine grade sandpaper, followed by levigated alumina, and then dulled by buffing with a slurry of chalk. The enamel surface then contained pits characteristic of a chalk-dulled surface. The degree of dullness was carried to a uniform low reflectance level as measured by a light source-photocell assembly. This measurement is the initial lustre of the tooth surface. The tooth was then brushed with the test composition on a brushing machine for a period of two hours, following which the lustre was again measured. The difference between the prebrushing lustre figure and the figure obtained after brushing with the test dentifrice represents the lustre increase. Results on three different teeth were average to obtain the lustre increase value.

Cleansing Test

The cleansing ability of the dentifrice was evaluated *in vitro* using the following technique.

A zein film was prepared on strips of a copper alloy surface, which film was intended to simulate in hardness the pellicle on the teeth. This film was brushed for 6 minutes with a slurry of dentifrice (1 part by weight) in water (2 parts by weight) using the reciprocating brushing action of a brushing machine yielding 150 double strokes per minute. The brushing head was equipped with flat bristle heads of medium nylon brushes. A weight of 230 grams was placed on top of the brushing head.

The evaluation of the degree of abrasion of the zein film was only semi-quantitative, i.e., the strips were compared visually to a strip brushed with a slurry (1 part by weight dentifrice, 2 parts by weight water) of a commercial toothpaste containing about 50% by weight of dicalcium phosphate dihydrate, and to a second strip brushed with water. Each slurry was run three times. While water brushing did not remove any appreciable amount of film, brushing with the commercial toothpaste removed a considerable amount.

During the testing of all dentifrices the same commercial toothpaste standard and a water standard were run simultaneously in order to allow for deviations in wear due to possible differences in the zein film.

In the following Examples the silica cleansing and polishing agent employed was a silica xerogel having an average particle diameter in the range 8 to 13 microns, typically about 10 microns, and a surface area of about 700 square metres per gram. It has a microporous structure, the average pore diameter being about 2 millimicrons which accounts for the large surface area. It is available commercially from W. R. Grace and Company, Davison Chemical Division (an American company) under the trade name "Syloid 63".

The silica thickening agent employed was a silica aerogel having an average particle diameter of about 3 microns and a surface area of about 300 square metres per gram. The pore diameter is in excess of 10 millimicrons. It is available commercially from W. R. Grace and Company, Davison Chemi-

cal Division under the trade name "Syloid 244".

In the following Examples percentages are by weight.

EXAMPLE 1

Ingredients	%
Silica Cleansing and Polishing Agent	12.00
Silica Thickening Agent	5.00
Hydroxyethyl Cellulose	1.50
Saccharin	0.20
Glycerin	34.76
Water	39.00
Stannous Fluoride	0.41
FDC Blue No. 1 (1%)	0.03
Flavour	1.10
Sodium-Lauryl Sulphate-Glycerine Mixture (21%)	6.00
	100.00

Cleansing: Same as the standard dentifrice
Lustre Increase Value: 60
Abrasion Loss Value: 3 microns

EXAMPLE 2

Ingredients	%
Silica Cleansing and Polishing Agent	15.00
Silica Thickening Agent	8.00
Sodium Carragheenate	1.30
Saccharin	0.20
Glycerin	31.53
Water	32.54
Polyethylene Glycol	4.00
FDC Blue No. 1 (1% solution)	0.02
Hexachlorophene	0.05
Tribromosalicylanilide	0.05
Sodium-Lauryl Sulphate-Glycerine Mixture (21%)	6.00
Stannous Fluoride	0.41
Flavour	0.90
	100.00

Cleansing: Same as the standard
Lustre Increase Value: 60
Abrasion Loss Value: 5 microns

EXAMPLE 3

Ingredients	%
Silica Cleansing and Polishing Agent	12.00
Insoluble Sodium Metaphosphate	6.00
Silica Thickening Agent	5.00
Sodium Carrhagheenate	1.30
Saccharin	0.20
Glycerin	31.53
Water	32.95
Polyethylene Glycol	4.00
Colorant (1% solution)	0,02
Flavour	0.90
Hexachlorophene	0.05
Tribromosalicyanilide	0.05
Sodium-Lauryl Sulphate-Glycerine Mixture (21%)	6.00
Total	100.00

Cleansing: Same as the standard
Lustre Increase Value: 62
Abrasion Loss Value: 5 microns

EXAMPLE 4

Ingredients	%
Silica Cleansing and Polishing Agent	14.00
Silica Thickening Agent	7.50
Carboxymethyl cellulose	0.60
Saccharin	0.20
Sorbitol Solution (70%)	67.82
Dye Solution (red)	0.47
Flavour	2.00
Sodium-Lauryl Sulphate-Glycerine Mixture (21%)	6.00
Hexachlorophene	0.10
Sodium Hydroxide (30% solution)	0.31
Total	100.00

This toothpaste gives cleansing and lustre increase and abrasion loss values similar to that of the toothpaste of Example 1.

The following comparative Examples A and B illustrate the advantages of using the dehydrated silica gel as the cleansing and polishing ingredient.

Comparative Example A

This example differs primarily from Example 2 in that the silica cleansing and polishing agent has been replaced by dicalcium phosphate dihydrate, a common dentifrice cleansing ingredient.

Ingredients	%
Dicalcium Phosphate Dihydrate	15.00
Silica Thickening Agent	8.00
Sodium Carragheenate	1.30
Saccharin	0.20
Glycerin	31.53
Water	32.56
Polyethylene Glycol	4.00
Hexachlorophene	0.10
Sodium-Lauryl Sulphate-Glycerine Mixture (21%)	6.00
Stannous Fluoride	0.41
Flavour	0.90
Total	100.00

Cleansing: Significantly poorer than the standard
Lustre Increase Value: Low; 33
Abrasion Loss Value: 2 microns

This Example demonstrates that the use of a conventional cleansing ingredient results in a significant decrease in lustre and cleansing ability.

Comparative Example B

In this Example, a test was conducted to determine whether pyrogenic silica (known as a toothpaste thickening agent) could ful-

fill the function of the silica xerogel. The pyrogenic silica had a particle size of about 20 millimicrons and a surface area of about 200 square meteres per gram. Because of the swelling properties of the pyrogenic silica, a significantly smaller amount of this silica must be used if one is to obtain acceptable consistencies.

Ingredients	%
Pyrogenic Silica	6.70
Sodium Carragheenate	1.00
Saccharin	0.20
Glycerin	40.20
Water	40.49
Polyethylene Glycol	4.00
Stannous Fluoride	0.41
Flavour	0.90
Hexachlorophene	0.05
Tribromosalicylanilide	0.05
Sodium-Lauryl Sulphate-Glycerine Mixture (21%)	6.00
Total	100.00

Cleaning: None, same as brushing with water
Lustre Increase Value: 60
Abrasion Loss Value: 1 micron

This example indicates that the use of pyrogenic silica results in a dentifrice with no cleansing ability.

Another type of silica which has been proposed as a dentifrice abrasive is diatomaceous silica. This has a surface area of 2 to 38 square metres per gram. It is, however, much more abrasive to dental enamel than the silicas employed in dentifrice compositions of this invention at comparable particle sizes. This is also the case with the crystalline type of silicas. However, in contrast to these other silicas, the highly porous form of silica used in this invention is sufficiently hard to effect good cleansing of the tooth surface but, probably due to its very porous nature, it causes relatively little abrasion of tooth enamel.

WHAT WE CLAIM IS:—

1. A dentifrice composition containing as the sole or principal cleansing and polishing ingredient from 5% to 50% by weight of the dentifrice of particulate dehydrated silica gel having an average particle diameter from 2 to 20 microns and a surface area of at least 600 square metres per gram.

2. A dentifrice composition as claimed in Claim 1, wherein the amount of the dehydrated silica gel is from 8 to 17% by weight of the composition.

3. A dentifrice composition as claimed in Claim 1 or Claim 2, wherein the dehydrated silica gel has an average particle diameter from 5 to 15 microns.

4. A dentifrice composition as claimed in any of the preceding claims, wherein the composition also contains a silica thickening agent.

5. A dentifrice composition as claimed in Claim 4, wherein the thickening agent is a pyrogenic silica.

6. A dentifrice composition as claimed in Claim 4, wherein the thickening agent is a silica aerogel.

7. A dentifrice composition as claimed in any of the preceding claims containing up to 10% by weight of water-insoluble sodium metaphosphate.

8. A dentifrice composition as claimed in any of the preceding claims, wherein the dehydrated silica gel has an average particle diameter of about 10 microns and a surface area of about 700 square metres per gram.

9. A dentifrice composition substantially as herein described with reference to any of Examples 1 to 4.

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