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TOOTH POWDER

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This invention relates to an improved dentifrice. It relates more particularly to an improved tooth powder which, retaining all of the characteristics of ordinary tooth powders from the standpoint of effectiveness as a dentifrice, has improved physical characteristics in that it is free flowing and has little or no tendency to form large lumps or agglomerates, thus being distinguished from ordinary tooth powders of similar ultimate composition, which, because of the extremely fine state of subdivision of the particles of which they are essentially composed, do not flow readily and tend to form large lumps or agglomerates, which make them difficult to dispense from the type of container ordinarily used for such products.

In general, tooth powders or dentifrices of the type to which this invention relates, consist of admixtures of a finely divided polishing agent, such as precipitated calcium carbonate, calcium sulfate, dicalcium phosphate, or tricalcium phosphate together with a relatively small amount of a detergent and suitable flavoring ingredients. In many cases, a relatively small amount of a material such as magnesium carbonate is added to improve the retention of the flavoring ingredients. The essential cleaning ingredients are the polishing agent, which constitutes by far the major proportion of the compositions, and the detergent, which, while used in a relatively small proportion, nevertheless has important functions in use. In such compositions, the polishing agent is necessarily extremely finely divided, in general the particles being of an order of magnitude such that substantially all pass the finest available screens. The use of such extremely finely divided particles as the polishing agent is essential to avoid scratching, etc., of the teeth. Nevertheless, because of this extremely fine state of subdivision of the particles, necessary from the standpoint of dentifrice properties, tooth powders as heretofore produced have not been free flowing.

It has been proposed to overcome the objectionable tendency of tooth powders to cling and resist flowing, or to form lumps or agglomerates, by granulating the powders, as by forming them into a slurry, drying the slurry and then grinding or comminuting the dry product to form relatively finely divided granules or particles; but the products so produced have been subject to a number of disadvantages. One objection to such products is that extreme difficulty has been encountered in attempting to comminute the dried slurry into reasonably uniformly graded granules, an undue proportion being reduced to an impalpable powder which in properties resembles the usual tooth powder rather than a granulated product, and which requires separation from the granulated product as by screening or the like if a free flowing product is to be obtained. Furthermore, the granules so produced are extremely friable; and in adding perfume or the like to the granulated product, it is extremely difficult, if not impossible, to avoid reducing a very large proportion thereof to the impalpable fines which are objectionable in the final product. In the ordinary handling of the granulated product so produced, a large proportion is broken down from the granulated state into the impalpable powder, again more nearly resembling ordinary tooth powders than a granulated product.

In accordance with the present invention, tooth powders of the same general composition as heretofore produced are produced in the form of granules or small agglomerates having characteristic free flowing properties and sufficient strength to enable the addition and admixture therewith of flavoring ingredients, and to resist breaking down in the ordinary handling in packing and distribution, and which are of a nature such that when moistened, as in coming in contact with water or saliva, the granules, agglomerates or beads break down almost instantaneously into the ultimate fine particles which constitute a desirable polishing agent for dentifrice purposes, such that the product does not feel gritty in use and there is no possibility of the teeth being scratched or injured by large particles.

The new dentifrices may be produced by drying slurries containing the extremely finely divided polishing agent, such as precipitated calcium carbonate, tricalcium phosphate, dicalcium phosphate, calcium sulfate or the like, a suitable detergent in proper proportions, and a small amount of a binder, and grinding the dried product. The binder serves to hold the granules together, imparting to them sufficient strength to withstand the treatment to which they must be subjected in the addition of flavoring ingredients and the handling in packaging, storage, and distribution, while at the same time leaving them free from hard, gritty particles, so that they instantly break up upon contact with water, saliva, or other aqueous liquid. It is important that the amount of binder used be sufficiently small so that this characteristic quality of immediate disintegration of the particles or agglomerates on contact with aqueous media is retained; if too

much binder is used, the granules become relatively hard, and do not instantly break down upon contact with aqueous media, so that the possibility of scratching of the teeth or unpleasant grittiness in use is not avoided. The disintegration of the agglomerates or granules on contact with aqueous media is aided by the detergent present in the compositions, which in effect serves as a wetting agent to promote contact of the water with the granules and insures rapid and immediate penetration of the water with breaking down of their structure into the impalpable fines of the precipitated polishing agent used.

A number of suitable binders are available for use in accordance with the present invention, including such materials as gelatin, gum tragacanth, sodium alginate, gum arabic and pectin, as well as crystallizable materials, such as sugars, sodium metaphosphate or other phosphate, and other materials adapted to form a matrix holding the polishing agent. The amount of binder used is relatively small, e. g., in the neighborhood of a percent or a fraction of a percent based upon the weight of the total final composition, and the amount which is used varies with the different binders, some producing the desired effect in smaller quantities than others. As pointed out, the amount of binder used should be such as to give the necessary resistance to breaking down to permit the addition of flavoring materials and to permit the necessary handling, while at the same time it must not be so much as to produce unduly hard particles.

In the final product, the extremely finely divided particles of the polishing agent apparently are held in a continuous phase of the binding material, perhaps with the detergent serving as a supplementary binding material; and the breaking down or disintegration of the granules into the ultimate particles is due, we believe, to the disintegration of this continuous phase of the binder which, being attenuated and offering a large surface, readily dissolves or breaks down when brought into contact with water or the like.

The new granules may be readily produced by grinding a dried slurry containing the detergent, the polishing agent and the binder, with separation of the extreme fines and unduly coarse particles after the grinding by screening or by other means. In the operation, the important advantages imparted to the composition by the inclusion of a binder become apparent, as the grinding operation may be carried out with the production of the granules of relatively uniformly graded sizes, with a minimum amount of impalpable fines or dust requiring separation by screening or the like and returning for reprocessing.

In order to produce the free flowing products of the present invention, it is important that the granules be properly sized, as if they are too small they do not have the desirable free flowing characteristics. Advantageously, their size should be such that substantially all are retained upon a 100 mesh screen, but pass through a 40 mesh screen.

In general, the slurry which is dried and subsequently ground, or otherwise comminuted, includes only the polishing agent, the detergent and the binder, although it may include sweetening materials, such as saccharin. The slurry should not contain the volatile ingredients of the final product, such as the flavoring materials, which, of course, would be removed to a substantial extent at the same time that the water is removed. After the product has been dried and converted into the form of small agglomerates or granules, the flavoring materials are added, as by spraying into a mass of the granules with agitation, or by the addition of magnesium carbonate treated with flavoring materials.

Another precaution which should be taken in the production of the dried product from the slurry involves avoidance of the removal of too much water in the case of those polishing agents which contain water or crystalization. Dicalcium phosphate, for example, in the precipitated form used in dentifrices, contains 2 molecules of water of crystallization, and care should be taken that the drying is carried out under such conditions, if this polishing agent be used, that substantially all of the free water is removed, without removal of any very substantial proportion of this water of crystallization, which might tend to make the polishing agent too harsh for satisfactory use in dentifrices.

The invention will be further illustrated by the following specific examples, but it is not limited thereto.

Example 1.—A slurry is prepared containing about 2.5% of the sodium sulfate of a monoglyceride of a higher fatty acid, about 50% of dicalcium phosphate, about 0.4% of saccharin, and about 0.1% of gum tragacanth, the remainder being water. The slurry is dried by evaporation of the water and the dried product is comminuted to form granules, the granules being sized so that substantially all are retained on a 100 mesh screen and substantially all pass a 40 mesh screen. The product flows very freely. It is flavored by the addition of flavoring materials as by spraying and with agitation; and because of the strength or rigidity imparted to the granules by the binder present, that is, the gum tragacanth, the product retains its free flowing characteristics despite the handling incidental to such treatment.

Example 2.—A slurry containing a little less than 2.5% of the detergent referred to in Example 1, about 50% of dicalcium phosphate, about 0.04% of saccharin and about 0.05% of sodium alginate, the remainder being water, is prepared, dried and converted into granules as in Example 1, after which flavoring materials are incorporated. The product is free flowing and the granules have sufficient strength to withstand ordinary handling and the incorporation of flavoring materials, let disintegrate immediately upon contact with aqueous media.

Example 3.—A slurry containing about 3.5% of soap, 50% of precipitated calcium carbonate, 0.1% of gum tragacanth and 0.04% of saccharin, the remainder being water, is prepared, dried and converted into granules as in Example 1, after which flavoring materials are incorporated. The product is free flowing, and the granules have sufficient strength to withstand ordinary handling and the incorporation of flavoring materials, yet disintegrate immediately on contact with aqueous media.

It will be understood that the ingredients used in the new tooth powders of the present invention may be widely varied, the essence of the invention being the production of the powders in the new granular form, with the use of a suitable binder, such as gum tragacanth, sodium alginate, gum arabic, gelatin, pectin, crystallizable substances such as sugars, sodium phosphates, etc., or other material adapted to form a matrix hold-

ing the polishing agent in agglomerated form. The amount of detergent, and the particular detergent used, may be the same as in conventional practice, such materials as soaps, sulfonated alcohols, sulfated monoglycerides, the Igepons, and other wetting agents or detergents being usable, it being understood that the amounts used will depend upon the particular detergent selected and upon the amount of foaming action desired in the product. For example, if ordinary soaps are used, they may be used in amounts corresponding to about 6½% of the final composition, whereas such materials as the sulfated or sulfonated alcohols may be used in amounts corresponding to about 1% of the final composition. The sulfated monoglycerides of higher fatty acids are advantageously used in amounts corresponding to about 4½% to 5% of the final composition, such amounts of these detergents giving a final product having an advantageous detergent action.

Any of the conventional polishing agents, such as precipitated calcium carbonate, dicalcium phosphate, tricalcium phosphate, calcium sulfate, etc., or mixtures thereof, may be used with the production of products fully comparable to tooth powders presently marketed in dentifrice properties, but having the free flowing characteristics of the new, properly sized, granular products.

The amount of the binder used may be varied over relatively wide ranges, although in any case the binder is used in quite small amounts. The amount of binder used will, of course, depend largely upon the particular materials selected, as well as the degree of hardness or resistance to disintegration desired in the final product. If sodium alginate is used as the binder, for example, it may advantageously be used in amounts corresponding to about ¼% of the final composition or less, for example, $\frac{1}{10}$%, but in any event care should be taken that too much is not used, or the product will not break down with sufficient ease. Gum tragacanth may be used in amounts ranging from about 0.15% up to about 0.5%, based upon the final composition, while gelatin may be used in even larger amounts, e. g., up to 2% or even a little more. 3% of gelatin produces a product which is believed to be too hard for satisfactory use. Similarly, such binders as gum arabic and pectin may be used in relatively widely varied proportions, but in no case in amounts that will result in the production of granules which are too hard.

It will thus be seen that the present invention involves the production of tooth powders or dentifrices in the form of small, discrete granules containing the polishing agent and a detergent, together with a small amount of binding material which imparts to the granules sufficient strength and rigidity to withstand ordinary handling and the incorporation of flavoring materials without breaking down, the granules having a selected size which imparts to them free flowing characteristics, which sharply distinguishes the new products from the previously known powders.

We claim:

1. As a new product, a dentifrice in the form of small, discrete granules consisting of a major proportion of an extremely finely divided polishing agent and containing a binder adapted to impart to the granules sufficient strength and rigidity to withstand ordinary handling without disintegration, said composition also containing a detergent and other conventional dentifrice ingredients.

2. A dentifrice in the form of small, discrete granules, comprising a major proportion of an extremely finely divided polishing agent agglomerated in the form of granules by the use of a binder, said dentifrice also containing a detergent and other conventional dentifrice ingredients, said granules being of a size that substantially all pass a 40 mesh screen and are retained on a 100 mesh screen, said granules disintegrating instantaneously upon contact with aqueous media, but having sufficient strength to permit incorporation of flavoring materials and ordinary handling without disintegrating.

3. A dentifrice in the form of small, discrete agglomerates having characteristic free-flowing properties, said agglomerates comprising a major proportion of an extremely finely divided polishing agent agglomerated by means of a binder adapted to form a more or less continuous phase or web enmeshing the fine particles of polishing agent, said dentifrice also containing a detergent and other conventional dentifrice ingredients.

4. A dentifrice in the form of agglomerates or granules which disintegrate instantaneously upon contact with aqueous media, composed essentially of an extremely finely divided polishing agent held in agglomerated form by means of a binder, together with a detergent and other conventional dentifrice ingredients, said agglomerates being of a size such that substantially all pass a 40 mesh screen and are retained on a 100 mesh screen, said product having free-flowing characteristics.

5. The method of producing tooth powder or dentifrices in granular form which comprises forming a slurry containing the polishing agent, the detergent and a binder, drying the slurry, comminuting the dried product to form a granular product and then adding volatile flavoring ingredients.

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