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DENTIFRICE

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This invention relates to an improved dentifrice. It relates more particularly to an improved tooth powder or dentifrice in the form of small, hollow bodies or beads of generally spherical or globular shape which, retaining all of the characteristics of ordinary tooth powders from the standpoint of effectiveness as a dentifrice, has improved physical characteristics such that it is free-flowing with little or no tendency to form lumps or large 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 composed, do not flow readily and tend to form lumps or large 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 large agglomerates, by granulating the powders, as by forming them into a slurry, drying the slurry and then grinding or comminuting the dried 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 diffi-

culty 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 resembles the usual tooth powder rather than a granulated product, requiring separation of this fine powder from the granulated product as by screening or the like and its return for reprocessing. The product so produced is composed of irregularly shaped granules which must be of relatively large size, e. g., of a size such that substantially none passes a 100 mesh screen, in order that it be free-flowing. Also, products so produced are extremely friable; and in adding flavoring materials to such granulated products, 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 that they interfere with its free-flowing properties, their friability being such that ordinary handling results in the breaking down or disintegration of a large proportion of the granules into the impalpable powder.

In accordance with the present invention, tooth powders are produced in the form of hollow bodies or beads of generally spherical or globular shape having characteristic free-flowing properties. It should be understood that the beads or discrete bodies produced in accordance with the invention are neither strictly globular nor spherical, but are irregular, though of a generally globular or spherical shape, and hollow. Such a product has extremely good free-flowing characteristics, even when formed of relatively small beads, as compared with irregularly shaped granules having free-flowing properties, for example, of beads of a size such that substantially all pass a 100 mesh screen but are retained on a 200 mesh screen. For practical purposes, the size of the beads should be such that substantially all pass a 40 mesh screen, which is about the upper limit of size for commercial purposes, and substantially all are retained upon a 200 mesh screen, which is about the lower limit of size of beads having the characteristic free-flowing properties of the products of the present invention.

The new dentifrices are produced by subjecting 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 advantageously, a small amount of a binder of such nature as to form a matrix or web enmeshing or

holding the fine particles of polishing agent, to a suitable spray drying operation, the slurry being sprayed into a current of hot gas, such as air, at a sufficiently high temperature, and in sufficient volume, to insure the production of a product with the properly reduced moisture content. The inclusion of a small amount of a binder in the slurry which is spray dried has important advantages, as it imparts to the beads or agglomerates obtained strength to withstand the treatment required in the addition of flavoring ingredients and the handling in packaging, storage and distribution, while at the same time, if properly selected, leaving them free from hard, gritty particles, so that they are readily pulverized and so that they instantly break up or disintegrate upon contact with water, saliva, or other aqueous liquids. The purpose of the binder is to impart strength and rigidity to the agglomerates produced; and the binder may be any one of a number of diverse substances, and may produce the required increase in strength or rigidity in various ways. For example, the binder may be some such material as gum tragacanth, gum arabic, gelatin, sodium alginate, pectin, etc., in which case I believe that it imparts rigidity to the product by forming a more or less continuous phase or matrix enmeshing the extremely finely divided particles of polishing agent, which continuous phase or matrix, while attenuated because of the small amount of binder used, is nevertheless sufficient to impart the desired strength or rigidity. On the other hand, crystallizable materials, such as sugar or the like, may be used as the binder; such materials effectively imparting the desired strength or rigidity to the particles produced. If, in the production of the new products, a binder is included in the slurry which is spray dried, care must be taken that the amount of binder used is within limits hereinafter described, as if too much binder is used, the beads or agglomerates produced are too 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. It is important that the agglomerates or beads immediately disintegrate upon contact with water or the like; and while the use of a binder has important advantages in imparting strength or rigidity to the beads or agglomerates, the amount used should be properly adjusted.

Where the binder is used in the small amounts contemplated by the present invention, it is necessarily present in the final product in a greatly attenuated form, offering a large surface; and I believe that the almost immediate disintegration of the particles or beads upon contact with aqueous media is due to the fact that the binder, if used, is so attenuated that it readily dissolves or breaks down when brought into contact with water or the like, the detergent present in the composition, which in effect serves as a wetting agent to promote contact of the water with the agglomerates or beads and insures rapid and immediate penetration of the water into them, aiding in the rapid disintegration into the almost impalpable fines of the precipitated polishing agent used.

In general, the slurry which is spray dried to form the new agglomerates or beads includes only the polishing agent, the detergent and the binder, although it may include such additional materials as sweetening materials, such as saccharin or the like. The slurry should not contain the vola-

tile ingredients of the final product, such as the usual flavoring ingredients, as such materials would, of course, be removed to a substantial extent at the time when the water is removed by exposure to a current of hot air. After the product has been spray dried, the flavoring ingredients are added, as by spraying into a mass of the granules with agitation, or by the addition of magnesium carbonate treated with the flavoring materials; and it is at this time that the important advantages of the use of a binder are most apparent. This operation of adding flavoring materials necessarily subjects the product to a certain amount of mechanical action, and if no binder has been used in the formation of the product, care must be taken to avoid breaking down the particles into the fine particles of polishing agent with the formation of a product very similar to ordinary tooth powders.

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 of crystallization. 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.

As has been pointed out above, the size of the agglomerates or beads is advantageously such that substantially all pass through a 40 mesh screen and are retained on a 200 mesh screen, beads of this size having excellent free flowing properties and a sufficiently small size to be commercially desirable. It will be understood that such spray dried beads or agglomerates will ordinarily be quite uniform in size, and their size may be regulated readily by proper regulation of the spinner or nozzle in the spray drying equipment. It is advantageous to make the beads relatively small, e. g., as small as is practical without interference with the desirable free flowing characteristics, as the physical strength, and ability to withstand handling, etc., without breaking down, increases as the size of the beads decreases.

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

Example 1.—A slurry is prepared containing about 2.5% of sodium sulfate of a monoglyceride of a higher fatty acid, about 50% of dicalcium phosphate, about 0.04% of saccharine, and about 0.1% of gum tragacanth, the remainder being water. The slurry is spray dried using a concurrent stream of hot air with an entrance temperature of from about 350° F. to about 400° F. and an exit temperature of about 200° F., with the production of the product in the form of a white powder consisting of small hollow beads or bodies of generally spherical or globular shape, of a size such that substantially all are retained on a 200 mesh screen. The product flows very freely. It is flavored by the addition of flavoring materials as by spraying and with agitation, and retains its free flowing characteristics despite the handling incidental to such treatment. Of course, the spray drying may be equally well accomplished with countercurrent spraying, with proper control over the degree of removal of water.

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 saccharine and about 0.05% of sodium alginate, the remainder being water, is prepared and spray dried as in Example 1, after which flavoring materials are incorporated. The product is free flowing and disintegrates immediately upon contact with aqueous media.

Example 3.—A slurry containing about 2.5% of the detergent of Example 1, about 50% of dicalcium phosphate, and about 0.04% of saccharine, the remainder being water, is prepared and spray dried as in Example 1. The product obtained is free flowing, but requires care in handling and in the incorporation of flavoring ingredients to prevent an undue breaking down of the beads or agglomerates.

Example 4.—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 and spray dried as in Example 1. The product obtained is free flowing, and disintegrates immediately upon contact with aqueous media, yet has sufficient strength to withstand handling, the incorporation of flavoring materials, etc.

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 agglomerated form, advantageously with the use of a suitable binder, such as gum tragacanth, sodium alginate, gum arabic, gelatin, pectin, and crystallizable substances such as sugar, etc. The amount of detergent, and the particular detergent used, is 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 amount 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 sulfated or sulfonated alcohols may be used in amounts corresponding to about 1% of the final composition. The detergents referred to in the Examples 1 to 3, sulfated monoglycerides of higher fatty acids, are advantageously used in amounts corresponding to about 4½% to 5% of the final composition, such amounts giving a final product having an advantageous detergent action due to the presence of this wetting agent.

Similarly, the particular polishing agent selected is of no particular importance, any of the conventional polishing agents, such as precipitated calcium carbonate, dicalcium phosphate, tricalcium phosphate, calcium sulfate, or mixtures thereof, etc., being usable, and giving products fully comparable to products presently marketed in dentifrice properties, but having free flowing characteristics.

Similarly, if a binder is used, it may be varied over relatively wide ranges, although in any case the binder is used in quite small amounts. The amount of binder used, of course, will depend largely upon the particular material selected. If

sodium alginate is used as the binder, it may advantageously be used in amounts corresponding to about ¼% of the final composition or less, for example, ⅙%, but in any event care should be taken that too much is not used, else the beads or agglomerates obtained will be too hard and will not disintegrate with sufficient rapidity when used. 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 beads or agglomerates which are too hard.

I claim:

1. A dentifrice or tooth powder in the form of small, discrete hollow bodies or beads of a generally globular or spherical shape, said product consisting of a major proportion of an extremely finely divided polishing agent together with a detergent and other conventional dentifrice ingredients, the size of such small, discrete bodies or beads being such that substantially all are retained on a 200 mesh screen and substantially all pass a 40 mesh screen, said product being of a free flowing nature and said bodies or beads disintegrating immediately upon contact with aqueous media.

2. A dentifrice in the form of small, discrete hollow bodies or beads of generally globular or spherical shape, consisting of a major proportion of an extremely finely divided polishing agent together with a binder which imparts to said bodies or beads strength with rigidity sufficient to permit them to withstand the handling incident to the incorporation of flavoring materials, packaging and the like, said bodies or beads disintegrating instantaneously upon contact with aqueous media, said dentifrice also containing a detergent and other conventional dentifrice ingredients.

3. A dentifrice in the form of small, discrete hollow bodies or beads of generally spherical or globular shape, said dentifrice comprising a major proportion of an extremely finely divided polishing agent held in said form by means of a binder adapted to form a more or less continuous phase or matrix 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 small, discrete hollow bodies or beads of generally spherical or globular shape, said dentifrice comprising a major proportion of an extremely finely divided polishing agent held in said form by means of a binder adapted to form a more or less continuous phase or matrix enmeshing the fine particles of polishing agent, said dentifrice also containing a detergent and other conventional dentifrice ingredients, said bodies or beads being of a size such that substantially all pass a 40 mesh screen but are retained on a 200 mesh screen.

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