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[54] **GRANULAR NON-DUSTING ENZYME PRODUCT FOR DETERGENT USE**

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[63] Continuation of Ser. No. 864,176, Oct. 6, 1969, abandoned.

[52] **U.S. Cl.**..... **252/132,** 252/89, 252/108, 252/110, 252/135, 252/525, 252/539, 252/DIG. 12

[51] **Int. Cl.**..... **C11d 3/22,** C11d 7/42, C11d 9/40

[58] **Field of Search**..... 252/89, 99, 108, 132, 135, 252/DIG. 12

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[57] **ABSTRACT**

Granular non-dusting product of high enzyme content for use in detergent composition. A powdered enzyme preparation is blended with soap and a sugar.

3 Claims, No Drawings

GRANULAR NON-DUSTING ENZYME PRODUCT FOR DETERGENT USE

This is a continuation of application Ser. No. 864,176 filed Oct. 6, 1969 now abandoned.

This invention relates to a method of making a granular non-dusting detergent composition for use in laundry products.

Powdered enzymes have been employed in presoak and washing detergent compositions since they are particularly effective against various common stains which are fixed to textiles and laundry. In particular, proteolytic enzymes, which possess the ability to digest and degrade protein matter, are effective in removing from textiles and laundry proteinic stains such as blood, sweat, milk, cocoa, gravy and other sauces and the like. This digestion or degradation of protein matter facilitates removal of dirt by the detergent. Amylases and lipases are also useful in detergent cleaning.

However, the use of powdered enzymes in such compositions has resulted in certain problems including the presence of an excessive amount of dust. Furthermore, detergent compositions containing enzymes have been subject to discoloration, formation of undesirable odor and caking.

It has been suggested to bind various compounds which are common builder salts in their hydratable form with enzymes. This may be done by contacting enzyme with an anhydrous or partially hydrated salt and adding water in insufficient amount to fullyhydrate the salt. A considerable amount of enzyme dust is still present when this enzyme-hydratable salt composition is used. Discoloration and odor formation may also occur.

In accordance with one aspect of this invention, the enzyme powder is blended with a mixture of a water-soluble soap and a water soluble sugar to form a granular product which is resistant to dusting. This granular product is then blended with a granular detergent product to produce a highly effective enzyme-containing detergent product having good stability.

In the preferred form of the invention the enzyme comprises a proteolytic enzyme which is active upon protein matter and catalyzes digestion or degradation of such matter when present as in linen or fabric stain in a hydrolysis reaction. The enzymes may be effective at a pH range of say about 4–12, and may be effective even at moderately high temperatures so long as the temperature does not degrade them. Some proteolytic enzymes are effective at up to about 80°C. and higher. They are also effective at ambient temperature and lower to about 10°C. Particular examples of proteolytic enzymes which may be used in the instant invention include pepsin, trypsin, chymotrypsin, papain, bromelin, colleginase, keratinase, carboxylase, amino peptidase, elastase, subtilisia and aspergillopepidase A and B. Preferred enzymes are subtilisin enzymes manufactured and cultivated from special strains of spore forming bacteria, particularly Bacillus subtilis.

Proteolytic enzymes such as Alcalase, Maxatase, Protease AP, Protease ATP 40, Protease ATP 120, Protease L-252 and Protease L-423 are among those enzymes derived from strains of spore foaming bacillus, such as Bacillus subtillis.

Different proteolytic enzymes have different degrees of effectiveness in aiding in the removal of stains from textiles and linen. Particularly preferred as stain removing enzymes are subtilisin enzymes.

Metalloproteases which contain divalent ions such as calcium, magnesium or zinc bound to their protein chains are of interest.

The enzyme preparations are generally extremely fine, often substantially impalable, powders. In a typical powdered enzyme preparation the particle diameter is mainly below 0.15 mm, generally above 0.01 mm, e.g. about 0.1 mm; for example, as much as 75 % of the material may pass through a 100 mesh (U.S. Standard) sieve. In contrast, conventional spray dried granules of detergent compositions are usually of very much larger particle size, with the major portion of the granules being at least about 0.2 mm in diameter, e.g. about 0.3 or 0.4, or even 0.5 or 1 to 2 mm.

The enzyme preparations are generally extremely diluted with salts such as calcium sulfate, sodium chloride, sodium sulfate and other inert materials. Chemically they are typically stable in the pH range of 5 to 10 and at an alkaline pH of 8.5 to 9. They can withstand temperatures of 49°C. to 77°C. with relatively little decomposition for time periods varying from 2 hours at the higher temperatures to more than 1 day at the lower temperatures. Different proteolytic enzymes have different degrees of effectiveness in aiding in the removal of stains from textiles and linen.

Instead of, of in addition to, the proteolytic enzyme, an amylase may be present such as a bacterial amylase of the alpha type (e.g. obtained by fermentation of B. subtilis). One very suitable enzyme mixture contains both a bacterial amylase of the alpha type and an alkaline protease, preferably in proportions to supply about 100,000 to 400,000 Novo alpha-amylase units per Anson unit of said alkaline protease.

In the most useful forms of the invention, the granular enzyme-soap-sugar product contains well over 50 % of powdered enzyme preparation. For instance, the latter may be present in proportions in the range of about 70–90% (e.g. 70, 80 or 85%). When a typical powdered enzyme preparation having an actual enzyme content of about 1.5 Anson units per gram is used, this range corresponds to an enzyme content of over 75 Anson units say about 105 to 135 Anson units, per 100 grams of the granular product. The ratio of the amount of sugar to the amount of soap may be, for instance, in the range of about 1:5 to 5:1 (e.g. about 1:2 or 1:1 or 2:1), on a dry basis. The product may also contain minor amounts of water, e.g. in the range of up to about 20% (such as about 10% or 5%).

The blending of the powdered enzyme preparation with the soap and sugar may be a simple dry blending of finely divided dry materials, followed by compaction under pressure to form a coherent mass, which can then be ground to granular form. In one very suitable method the sugar may be introduced in syrupy or pasty form so that a coherent mixture is formed on mixing; such a mixture can be mixed more thoroughly, and compacted, by roll milling giving a chip that can be broken up to form granules, e.g. granules such that a major portion thereof are about 0.2 mm in diameter, e.g. about 0.3 or 0.4, or even 0.5 or 1 to 2 mm. Any fines formed during the grinding or breaking operations can be compacted and reground. Granules can also be formed by prilling the mixture or squeezing it through a screen. The entire operation may be carried out at room temperature (e.g. 20°–25°C.) and atmospheric pressure. Higher or lower temperatures (and pressures) may be used, but it is preferred not to reach a tempera-

ture that would substantially degrade the enzyme. It is preferred to use a closed mixer for at least the initial mixing operation, to insure against loss of enzyme from any dust that may be entrained in the air before the enzyme preparation becomes bonded or wetted by the other ingredients.

The enzyme-containing granules or beads produced in accordance with this invention may be added to a wide variety of washing products. Thus, they may be incorporated in a laundry presoak product or in a laundry detergent or in a dishwashing product. A typical presoak product contains a relatively high concentration of builder salt such as about 30 to 95% pentasodium tripolyphosphate (calculated as anhydrous pentasodium tripolyphosphate), about 2 to 10% of organic surface active detergent, plus other ingredients such as sodium silicate (which acts as a builder salt and also acts to inhibit corrosion of aluminum surfaces), brightening agents and sodium sulfate. A laundry detergent generally has a lower ratio of builder salt to organic surface active agent (e.g. a ratio in the range of about 5:1 to 15:1). Dishwashing products, designed for use in automatic dishwashers, are on the other hand usually more alkaline, containing a very high proportion of alkaline builder salt, such as a mixture of the pentasodium tripolyphosphate and sodium silicate; they contain little, if any, organic surface active detergent, e.g. about 0.2 to 3%, and usually also contain a minor proportion (e.g. 0.5 to 5%) of an agent to prevent water-spotting such as a dry water-soluble compound which on contact with water, liberates hypochlorite chlorine (e.g. a heterocyclic dichloroisocyanurate); alternatively, a chlorinated phosphate (such as the well known chlorinated trisodium phosphate) may be used to supply both hypochlorite chlorine and some phosphate.

In formulating the washing products, the water-soluble builder salts may be phosphates and particularly condensed phosphates (e.g. pyrophosphates or tripolyphosphates), silicates, borates and carbonates (including bicarbonates), as well as organic builders such as salts of nitrilotriacetic acid or ethylene diamine tetracetic acid. Sodium and potassium salts are preferred. Specific examples are sodium tripolyphosphate, potassium pyrophosphate, sodium hexametaphosphate, sodium carbonate, sodium bicarbonate, sodium sesquicarbonate, sodium tetraborate, sodium silicate, salts (e.g. Na salt) of methylene diphosphonic acid, disodium diglycollate, trisodium nitrilotriacetate, or mixtures of such builders, including mixtures of pentasodium tripolyphosphate and trisodium nitrilotriacetate in a ratio, of these two builders, of 1:10 to 10:1, e.g. 1:1.

The organic surface active agent may be an anionic, nonionic or amphoteric surface active agent; mixtures of two or more such agents may be used.

The anionic surface active agents include those surface active or detergent compounds which contain an organic hydrophobic group and an anionic solubilizing group. Typical examples of anionic solubilizing groups are sulfonate, sulfate, carboxylate, phosphonate and phosphate. Examples of suitable anionic detergents which fall within the scope of the invention include the soaps, such as the water-soluble salts of higher fatty acids or resin acids, such as may be derived from fats, oils and waxes of animal, vegetable origin, e.g. the sodium soaps of tallow, grease, coconut oil, tall oil and mixtures thereof; and the sulfated and sulfonated synthetic detergents, particularly those having about 8 to 26, and preferably about 12 to 22, carbon atoms to the molecule.

As examples of suitable synthetic anionic detergents there may be cited the higher alkyl mononuclear aromatic sulfonates such as the higher alkyl benzene sulfonates containing from 10 to 16 carbon atoms in the alkyl group in a straight or branched chain, e.g., the sodium salts of higher alkyl benzene sulfonates or of the higher alkyl toluene, xylene and phenol sulfonates; alkyl naphthalene sulfonate, ammonium diamyl naphthalene sulfonate, and sodium dinonyl naphthalene sulfonate. In one preferred type of composition there is used a linear alkyl benzene sulfonate having a high content of 3- (or higher) phenyl isomers and a correspondingly low content (well below 50%) of 2- (or lower) phenyl isomers; in other terminology, the benzene ring is preferably attached in large part at the 3 or higher (e.g. 4, 5, 6 or 7) position of the alkyl group and the content of isomers in which the benzene ring is attached at the 2 or 1 position is correspondingly low. Particularly preferred materials are set forth in U.S. Pat. No. 3,320,174, May 16, 1967, of J. Rubinfeld.

Other anionic detergents are the olefin sulfonates, including long chain alkene sulfonates, long chain hydroxyalkane sulfonates or mixtures of alkenesulfonates and hydroxylalkane sulfonates. These olefin sulfonate detergents may be prepared, in known manner, by the reaction of SO_3 with long chain olefins (of 8–25, preferably 12–21, carbon atoms) of the formula $RCH{=}CHR_1$, where R is alkyl and R_1 is alkyl or hydrogen, to produce a mixture of sultones and alkenesulfonic acids, which mixture is then treated to convert the sultones to sulfonates. Examples of other sulfate or sulfonate detergents are paraffin sulfonates having, for example, about 10–20, preferably about 15–20, carbon atoms such as the primary paraffin sulfonates made by reacting long chain alpha olefins and bisulfites (e.g. sodium bisulfite) or paraffin sulfonates having the sulfonate groups distributed along the paraffin chain such as the products made by reacting a long chain paraffin with sulfur dioxide and oxygen under ultraviolet light followed by neutralization with NaOH or other suitable base (as in U.S. Pat. Nos. 2,503,280; 2,507,088; 3,260,741; 3,372,188 and German Pat. No. 735,096); sulfates of higher alcohols; salts of α-sulfofatty esters (e.g. of about 10 to 20 carbon atoms, such as methyl α-sulfomyristate or α-sulfotallowate).

Examples of sulfates of higher alcohols are sodium lauryl sulfate, sodium tallow alcohol sulfate, Turkey Red Oil or other sulfated oils, or sulfates or mono- or di-glycerides of fatty acids (e.g. stearic monoglyceride monosulfate), alkyl poly (ethenoxy) other sulfates such as the sulfates of the condensation products of ethylene oxide and lauryl alcohol (usually having 1 to 5 ethenoxy groups per molecule); lauryl or other higher alkyl glyceryl ether sulfonates; aromatic poly (ethenoxy) ether sulfates such as the sulfates of the condensation products of ethylene oxide and nonyl phenol (usually having 1 to 6 oxyethylene groups per molecule).

The suitable anionic detergents includes also the acyl sareosinates (e.g. sodium lauroylsarcosinate) the acyl esters (e.g. oleic acid ester) of isethionates, and the acyl N-methyl taurides (e.g. potassium N-methyl lauroyl- or oleyl tauride).

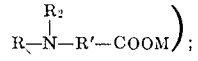
The most highly preferred water soluble anionic detergent compounds are the ammonium and substituted ammonium (such as mono-, di- and triethanolamine), alkali metal (such as sodium and potassium) and alkaline earth metal (such as calcium and magnesium) salts of the higher alkyl benzene sulfonates, olefin sulfonates, the higher alkyl sulfates, and the higher fatty acid monoglyceride sulfates. The particular salt will be suitably selected depending upon the particular formulation and the proportions therein.

Nonionic surface active agents include those surface active or detergent compounds which contain an organic hydrophobic group and a hydrophilic group which is a reaction product of a solubilizing group such as carboxylate, hydroxyl, amido or amino with ethylene oxide or with the polyhydration product thereof, polyethylene glycol.

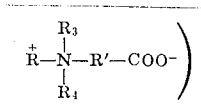
As examples of nonionic surface active agents which may be used there may be noted the condensation products of alkyl phenols with ethylene oxide, e.g., the reaction product of isooctyl phenol with about 6 to 30 ethylene oxide units; condensation products of alkyl thiophenols with 10 to 15 ethylene oxide units; condensation products of higher fatty alcohols such as tridecyl alcohol with ethylene oxide; ethylene oxide addends of monoester of hexahydric alcohols and inner ethers thereof such as sorbitan monolaurate, sorbitol monooleate and mannitan monopalmitate, and the condensation products of polypropylene glycol with ethylene oxide.

A particularly suitable composition, for use as a granular detergent material contains a mixture of a linear alkylbenzenesulfonate, as previously described, soap and a nonionic detergent, with the soap and nonionic detergent being present in minor portions. The ratios of the amounts of (A) soap, and (B) nonionic detergent, to (C) the total amount of the synthetic anionic sulfate and sulfonate detergent, in this mixture, are preferably as follows: A:C, about 1:10 to 1:2, preferably about 1:4 to 1:6, on an anhydrous basis; B:C about 1:10 to 1:3, e.g. about 1:4 to 1:6, on an anhydrous basis. The component (C) may comprise a blend of the linear alkylbenzenesulfonate detergent with other anionic synthetic sulfate or sulfonate detergents (e.g. olefin sulfonates, paraffin sulfonates having the sulfonate groups distributed along the paraffin chain, or alkyl sulfates) with the alkylbenzenesulfonate constituting, say, one-third, one-half or two-thirds of this blend.

Examples of suitable amphoteric detergents are those containing both an anionic and a cationic group and a hydrophobic organic group, which is advantageously a higher aliphatic radical, e.g. of 10–20 carbon atoms. Among these are the N-long chain alkyl aminocarboxylic acids (e.g. of the formula



the N-long chain alkyl iminodicarboxylic acids (e.g. of the formula $RN(R'COOM)_2$) and the N-long chain alkyl betains (e.g. of the formula



where R is a long chain alkyl group, e.g. of about 10–20 carbons, R′ is a divalent radical joining the amino and carboxyl portions of an amino acid (e.g. an alkylene radical of 1–4 carbon atoms), N is hydrogen or a salt-forming metal, R_2 is a hydrogen or another monovalent substituent (e.g. methyl or other lower alkyl), and R_3 and R_4 are monovalent substituents joined to the nitrogen by carbon-to-nitrogen bonds (e.g. methyl or other lower alkyl substituents). Examples of specific amphoteric detergents are N-alkyl-beta-aminopropionic acid; N-alkyl-beta-iminodipropionic acid, and N-alkyl, N,N-dimethyl glycine; the alkyl group may be, for example, that derived from coco fatty alcohol, lauryl alcohol, myristyl alcohol (or a lauryl-myristyl mixture), hydrogenated tallow alcohol, cetyl, stearyl, or blends of such alcohols. The substituted aminopropionic and iminodipropionic acids are often supplied in the sodium or other salt forms, which may likewise be used in the practice of this invention. Examples of other amphoteric detergents are the fatty imidazolines such as those made by reacting a long chain fatty acid (e.g. of 10 to 20 carbon atoms) with diethylene triamine and monohalocarboxylic acids having 2 to 6 carbon atoms, e.g. 1-coco-5-hydroxethyl-5-carboxymethylimidazoline; betaines containing a sulfonic group instead of the carboxylic group; betaines in which the long chain substituent is joined to the carboxylic group without an intervening nitrogen atom, e.g. inner salts of 2-trimethylamino fatty acids such as 2-trimethylaminolauric acid, and compounds of any of the previously mentioned types but in which the nitrogen atom is replaced by phosphorous.

Various other materials may be present in the granular washing products. Thus, materials such as the higher fatty acid amides may be added to improve detergency and modify the foaming properties in a desirable manner. Examples thereof are the higher fatty acid alkanolamides, preferably having 2–3 carbons in each alkanol group and a fatty acyl radical within the range of 10–18 carbons, preferably 10–14 carbons, such as lauric or myristic monoethanolamides, diethanolamides and isopropanolamides. Tertiary higher alkyl amino oxides such as having about 10–18 carbons in one alky group, e.g. lauryl or myristyl dimethylamino oxide, may be added also. Fatty alcohols of 10–18 carbons such as lauryl or coconut fatty alcohols, or cetyl alcohol are suitable additives also. A hydrotropic material such as the lower alkyl aryl sulfonates, e.g. sodium tuluene or xylene sulfonates, can assist processing also. In general, these materials are added in minor amounts, usually from about ½ to 10%, preferably 1 to 6%, based on the total solids.

The washing products may also contain optical brightening agents or fluorescent dyes (e.g. in amounts in the range of about one-twentieth to one-half percent); germicidal ingredients such as halogenated carbanilides, e.g. trichlorocarbanilide, halogenated salicylanilide, e.g. tribromosalicylanilide, halogenated bisphenols, e.g. hexachlorophene, halogenated trifluoromethyldiphenyl urea, zinc salt of 1-hydroxy-2-pyridinethione and the like (e.g. in amounts in the range of about one-fiftieth to 2%); soil-suspending agents such as sodium carboxymethyl cellulose or polyvinyl alcohol, preferably both, or other soluble polymeric materials, such as methyl cellulose (the amount of suspending agent being, for example, in the range of about one-twentieth to 2%); antioxidants such as 2,6-

di-tert-butylphenol, or other phenolic antioxidant materials (e.g. in amounts in the range of about 0.001 to 0.1%), coloring agents, bleaching agents and other additives.

The washing products, to which the enzyme-soap-sugar granules are added, are most often in the form of spray-dried hollow beads or spongeous low density granules. As previously indicated, their particle sizes are usually such that a major portion is at least about 0.2 mm in diameter, e.g. about 0.3 or 0.4, or even 0.5 or 1 to 2 mm.

The amount of the enzyme-soap-sugar granules added to the washing product is relatively small, usually being such that the content of powdered enzyme preparation in the final product is, for example, in the range of about 0.1 to 4%, preferably about 0.3 to 2%; for a powdered enzyme preparation having an alkaline protease content of 1.5 Anson units per gram this represents about 0.15 to 6 Anson units (preferably about 0.45 to 3 Anson units) per 100 grams of the final product.

The amount of the enzyme mixture present in the detergent composition will, of course, depend to some extent on the amount of the detergent composition which is to be added to the wash water. For detergent compositions which are intended for use at concentrations of, say about 0.15% in the wash water of an automatic home laundry machine, one suitable amount of enzyme mixture is such as to provide 1 Anson unit of the alkaline protease for each 100 to 500 (e.g. 200 to 400) grams of the detergent composition.

The following Examples are given to illustrate this invention further. In these Examples, as in the rest of the application, all proportions are by weight unless otherwise specified. Also, in these Examples, the pressure is atmospheric unless otherwise specified. All mesh and screen sizes are U.S. Standard.

EXAMPLE 1

a. Seventy parts of powdered proteolytic subtilisin enzyme preparation (Alcalase, 1.5 Anson Units/g), 15 parts of corn syrup ("Karo Crystal Clear" syrup containing about 50% water and about 50% corn sugar which comprise mainly glucose) and 15 parts of granulated soap (sodium soap of a mixture of 4 parts of tallow fatty acids and 1 part of coconut oil fatty acids, the soap granules being about 2 mm in diameter and their water content being about 12%) are mixed thoroughly at room temperature in a conventional Hobart mixer, the corn syrup first being mixed with the dry powdered enzyme preparation to form a dough, after which the soap granules are added. The resulting blend, which is a doughy mass, is then passed through a soap mill, such as a three-roll Lehman soap mill in which the material passes between parallel closely spaced rolls and then is peeled off the last roll by means of a serrated knife, which, owing to its serrations, forms chips of the milled material. The resulting chips are typically about 1 to 2 inches long, about one-fourth to one-half inches wide and about 0.01 to 0.02 inch thick; thinner flakes or chips (e.g. about 0.001 inch thick) may be produced as by using a 5-roll mill. The tough, dusting-resistant chips thus produced are then ground to form free-flowing granules of, say 40 to 60 mesh particle size, as in a ball mill.

The resulting dusting-resistant granules are then blended with a heavy duty built granular spray-dried detergent composition, supplied as hollow beads (or clumps of hollow beads) of the following approximate composition: 10% sodium linear tridecylbenzenesulfonate, 2% nonionic detergent which is an adduct of 11 mols of ethylene oxide per mol of a linear C14-C15 alkanol (Neodol 45-11), 2% water soluble sodium soap of higher fatty acid, 33% pentasodium tripolyphosphate, ½% sodium carboxymethylcellulose, 8.5% total water (including water of hydration), the balance being sodium sulfate together with small amounts of optical brighteners and antioxidant. The amount of enzyme-containing granules is such that the resulting product contains about 0.5% of the Alcalase. The product shows good retention of its enzyme activity on aging.

b. Example 1*a* is repeated except that the proportions are 70 parts of the enzyme powder, 10 parts of the corn sugar syrup and 20 parts of the soap.

The proteolytic subtilisin enzyme preparation used in the foregoing Example has its maximum proteolytic activity at a pH of 8–9. This activity as measured at pH 7.5 on the commercial enzyme preparation available from Novo Industri A/S, Copenhagen, Denmark is about 1.5 Anson Units per gram of the enzyme. The commercial enzyme preparation is a raw extract of Bacillus subtillis culture and contains about 6% of pure crystallized proteolytic material. The preparation is an extremely fine powder; typically the particle diameter is mainly below 0.15 mm, generally above 0.01 mm, e.g. about 0.1 mm, and as much as 50% or even 75% of material may pass through a 100 mesh sieve. The preparation contains about 70% NaCl and about 15–18% Na_2SO_4. Its organic content is in the neighborhood of 11%.

EXAMPLE 2

a. 80 parts of the powdered enzyme preparation used in Example 1 are dry blended with 10 parts of micropulverized refined cane sugar and 10 parts of finely powdered soap (sodium soap of a mixture of 4 parts tallow fatty acids and 1 part coconut oil fatty acids). The mixture of powders is pressed into tablets in a Carver press at a high pressure (e.g. of about 10,000 to 25,000 lbs/in^2), the resulting hard glassy tablets, which are about 1 inch in diameter and about three-eighths inch thick, are then ground to form free flowing, non-dusting granules of 40 to 60 mesh particle size, which are then blended with a detergent composition, as in Example 1. The granules dissolve in water much more rapidly than granules containing only soap and the enzyme preparation.

The tablets are readily water soluble and can be added, as such, to the water used for washing or soaking clothes.

The fines from the grinding operation can be repressed into the tablet form, in the same way, and reground to produce more granules. Before re-pressing the fines can be mixed, if desired, with additional powdered enzyme preparation, soap powder and sugar.

b. Example 2*a* is repeated, except that the proportions are 70 parts of the enzyme powder, 15 parts of the sugar, and 15 parts of the soap.

c. Example 2*a* is repeated except that the proportions are 85 parts of the enzyme powder, 10 parts of the pulverized sugar and 5 parts of the pulverized soap.

Each of the granular products of this Example 2 is dry blended with spray dried hollow beads of a mixture of pentasodium tripolyphosphate, an organic detergent

(sodium linear tridecylbenzenesulfonate), sodium silicate ($Na_2O:SiO_2$ ratio 1:2.35), borax, sodium carboxymethyl cellulose, polyvinyl alcohol, optical brighteners, and sodium sulfate to give, in each case, a composition having the following approximate analysis: total anhydrous phosphate solids 34%, organic detergent 17.3%; borax 1%; sodium silicate 7%; brighteners 0.46%; sodium carboxymethyl cellulose 0.4%; polyvinyl alcohol 0.2%; H_2O 9.5%; the enzyme preparation 0.5%, and the balance being sodium sulfate (in the neighborhood of 30%), together with the sugar and soap present in the granules containing the enzyme preparation. The resulting heavy duty built detergent blend shows good retention of its enzyme activity on aging.

It is also within the broader scope of this invention to prepare a granular enzyme concentrate in which the soap is omitted. Such a granular product can be made, for example, by dry-blending the enzyme preparation and corn sugar, humidifying the mixture so that it becomes a cohesive mass and then breaking up this mass into granules. The granules may be used in the same way as the soap-containing granules described above.

Instead of glucose and sucrose other water-soluble sugars may be used in the practice of the invention, such as other aldohexoses, ketohexoses, monosaccharides and disaccharides.

It will be apparent that variations of the invention may be made and equivalents substituted therefor.

We claim:

1. A process for making an enzyme concentrate, for use as an additive to detergent compositions to make them enzyme-active, in which, on an enzyme preparation-soap-sugar product basis, from 50 to about 90% by weight of a powdered enzyme preparation, of particles of diameters less than 0.15 millimeter, is dry blended with a mixture of a water-soluble granular soap and a water soluble granular sugar in a soap-sugar ratio of about 1:5 to 5:1, the soap being in finely divided powder form and the sugar being micropulverized, and said blended mixture is compacted under pressure between opposed solid surfaces by roll milling or pressing to form a coherent mass which is then size-reduced to granular form, the granules of which are of particle sizes of up to 40 mesh and contain proteolytic enzyme preparation in an amount sufficient to provide a final washing product having 0.15 to 6 Anson units of activity per hundred grams when incorporated therein in a proportion containing from 0.1 to 4% of powdered enzyme preparation.

2. A process according to claim 1 wherein the proportion of powdered enzyme preparation dry blended with a mixture of water-soluble granular soap and water-soluble granular sugar is from 70 to 90%, the powdered enzyme preparation is of particle sizes of diameters of 0.01 to 0.15 millimeter, the compacting is by milling to chips and the size-reduction is by grinding to free-flowing granules.

3. A process for making an enzymatic detergent which comprises making an enzyme concentrate according to the process of claim 1 and blending it with a particulate built synthetic organic detergent composition in such proportion as to produce an enzymatic detergent having 0.15 to 6 Anson units of activity per hundred grams of detergent composition when incorporated therein, whereby said blending is effected without dusting due to the presence of the enzyme preparation.

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