United States Patent Office 3,781,228 Patented Dec. 25, 1973

3,781,228

LAUNDRY PRODUCT CONTAINING ENZYME

Robert William McDonnell, Teaneck, and Maung Hla Win, Berkeley Heights, N.J., assignors to Colgate-Palmolive Company, New York, N.Y.

No Drawing. Continuation-in-part of abandoned application Ser. No. 733,298, May 31, 1968. This application Dec. 17, 1971, Ser. No. 209,355

Int. Cl. C11d 3/22, 7/42, 11/00

U.S. Cl. 252—539 6 Claims

ABSTRACT OF THE DISCLOSURE

Laundry product containing granules of a binding agent, such as polyvinyl alcohol or caroxyalkyl cellulose, and a detergent builder salt, such as sodium tripolyphosphate, and bound to and substantially homogeneously distributed on the surface of the granules, an enzyme.

This is a continuation of application Ser. No. 733,298, filed May 31, 1968, now abandoned.

This invention relates to a laundry product useful as a component of presoak or washing detergent compositions. More particularly, the laundry product contains granules of a binding agent, such as a polyvinyl alcohol or a carboxyalkyl cellulose, and a detergent builder salt, and bound to and substantially evenly distributed on the surface of the granules, a powdered enzyme.

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 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. Some individuals experience allergic reactions to the enzyme 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 fully hydrate the salt. A considerable amount of enzyme dust is still present when the enzyme-hydratable salt composition is used. Discoloration and odor formation may also occur.

It is an object of this invention to provide a laundry composition containing granular particles having bound thereto enzyme wherein the granular particles are hard, spheroidal and sizable particles which do not substantially dust when in use, do not discolor upon storage and are essentially free of disagreeable odor. Other objects of the invention will be apparent from the disclosure below.

In accordance with certain of its aspects, this invention relates to a laundry product containing granules of water-soluble binding agent selected from the group consisting of polyvinyl alcohol and alkali metal carboxyalkyl cellulose, and a detergent builder salt; and bound to and substantially homogeneously distributed on the surface of said particles, a powdered enzyme.

The laundry composition of this invention can be made by dry mixing or by blending powdered enzyme with granular particles of detergent builder salt and then agitating, typically by tumbling and spraying a water solution

of the binding agent on the enzyme-builder salt blend. The ingredients are typically blended in amounts of about 3–15 percent by weight, preferably about 5–10 percent, and most preferably about 7.5 percent of enzyme; the builder salt and binding agent solution are in a ratio of about 49:1 to 17:3 and preferably about 16:1. The total of enzyme, builder salt and binding agent solution comprises the essential components of the laundry composition of the invention. A preferred laundry composition contains 7.5 parts of enzyme; about 87 parts of builder salt; and about 5.5 parts of a water solution containing about 0.5–25 parts by weight, and most preferably about 10 parts of binding agent in 100 parts by weight of solution. Agitation, such as tumbling, is continued until the laundry product has a desired degree of dryness. Agitation may be performed in a Dravo pan and continuously or batchwise in a rotary drum or a twin-shell blender. Optionally, the degree of dryness can be increased by passing a stream of air over the blend during or following the agitation.

When the laundry product of the invention is dried, it typically contains about 3–15 parts by weight of enzyme; about 67 to 95 parts by weight of builder salt; and binding agent in an effective amount up to about 8 parts by weight.

The granular particles of the laundry product so produced are essentially spheroidal in shape. The enzyme is bound to and substantially evenly distributed over the surfaces of the granular particles. The size of the particles formed during tumbling may be controlled so that the particles are substantially free of fines (finer than 100 mesh) and have a range in size typically of about 0.15–2 mm. and preferably about 0.25–0.84 mm. The dried sizable particles are relatively hard and resistant to crushing during metering and blending in the formulation of a finished detergent. Likewise, being sizable and hard, they are free-flowing and do not tend to dust when being poured or otherwise used. Typically, the specific gravity of the particles ranges from 0.4–0.9 and may be roughly equivalent to the specific gravity of the detergent with which the laundry product is admixed, although the specific gravity of the laundry product may differ from that of the detergent without undesirable settling.

The proteolytic enzymes which are employed in the instant invention are active upon protein matter and catalyze digestion or degradation of such matter when present as in linen or fabric stain in a hydrolysis reaction. The enzymes are effective over a pH range of about 4–12, preferably about 7–10, such as usually prevails in detergent cleaning procedures. Moreover, they 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 aspergillopepitase A and B. They are available also under names such as Alcalase (Novo Industri, Copenhagen, Denmark), Maxatase (Royal Netherlands Fermentation, Delft, Netherlands), Protease AP (Sandoz-Ferment, Basel, Switzerland), Protease B–400 (Sandoz-Ferment), Protease ATP 40 (Sandoz-Ferment), Pancreatin NF (Pfizer), Pancreatin 6xNF (Armour), Fungal Protease (Miles), DSE Numbers 4–9 (Rohm and Haas), Exzyme DPX (Premier Malt), Protease L–252 Digester (Premier Malt), Protease L–253 Digester (Premier Malt), Protease L–423 (Premier Malt), Protease L–516 (Premier Malt), Protease L–517 (Premier Malt), Exzyme WT–2 (Premier Malt), Texzyme PX 1 (Premier Malt), Protease P–G (Pfizer), Compound 37B (Miles), Serizyme (Wallerstein), Papain 100 (Wallerstein), Alkaline Protease HA (Wallerstein), Optimo Papain (Penick), Ficin (Miles),

Bromelain (Miles), HT Proteolytic Concentrate (Miles), Protease ATP 40 (Rapidase), Protease ATP 120 (Rapidase), Rhozyme P–11 (Rohm and Haas), Monzyme (Monsanto Chemical Company), and Rhozyme PF (Rohm and Haas).

Proteolytic enzymes such as Alcalase, Maxatase, Protease AP, Protease ATP 40, Protease ATP 120, Protease L–252 and Protease L–423 are derived from strains of spore-forming 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 Alcalase, Maxatase, Protease AP, Protease ATP 40, and Rapidase.

The above enzymes may have other than proteolytic activity. Thus, Monzyme also has amylase activity.

In addition to or in combination with the proteolytic enzymes set forth above, amylases such as the microbial amylases or other carbohydrases, such as maltase, saccharase, pectinase, lycozyme and glycosidases; and lipases, such as microbial lipases, pancreatic lipase, plant lipases or gastric lipase may be employed.

The builder salts with which the powdered enzyme is mixed prior to contact with the binding agent typically have a pH of about 4–12, when dissolved in water. The particles of the builder salts may be in fully hydrated form or may be anhydrous or partially hydrated. Process may be performed in a rotary drum (batch or continuous) Dravo pan or twin-shell blender (batch or continuous).

The particles of builder salt typically have about the same size as the finished laundry product, although they contain somewhat more fines and, therefore, are more prone to dusting than the finished laundry product.

Typical examples of builder salts which may be employed in the practice of this invention are organic builder salts in hydrated or less than fully hydrated form, such as the trisodium salt of nitrilotriacetic acid and the di-, tri- and tetrasodium salts of ethylenediamine tetraacetic acid and inorganic builder salts, preferably polyphosphate salts, and most preferably in the form of the normal or completely neutralized salts which have the property of inhibiting precipitation of calcium and magnesium material in aqueous solution and of contributing to the heavy-duty performance of the liquid detergent product. They may be considered as derived from orthophosphoric acid or the like by the removal of molecularly-bound water, though any suitable means of manufacture may be employed if desired. Such complex or molecularly dehydrated polyphosphate salts may be used in the form of the normal or completely neutralized salt, e.g., pentapotassium tripolyphosphate, or partially neutralized salt, e.g. potassium acid tripolyphosphate. It is preferred to use the pentapotassium tripolyphosphate or tetrapotassium pyrophosphate salts including any desired combination of the same. The alkali metal salts of tetraphosphoric acid may be used also. Other suitable materials which may be employed are sodium tripolyphosphate and its hexahydrate, and tetrasodium pyrophosphate in suitable proportions.

Other alkaline builder salts may be employed also such as the soluble alkali metal borates and silicates. The silicates may be employed as the sole builder salt if desired or in suitable combination with the polyphosphates. Suitable silicates are those available in solid form and having an alkali oxide to silica ratio within the range of about 1:1 to 1:4, and preferably from about 1:2 to 1:3. Examples are sodium silicates having an Na_2O to SiO_2 ratio of 1:2.35, 1:2.5, 1:3.2, 1:2.0, 1:1.6 and 1:1. The most highly preferred builder salt is anhydrous sodium tripolyphosphate.

The binding agent employed in accordance with this invention is a polyvinyl alcohol or an alkali metal carboxyalkyl cellulose. The specific or exact degree of polymerization of the vinyl polymer is not critical, provided the material has the desired water solubility or dispersibility to produce the desired effect. It is usually a mixture of specific polymers containing different numbers of monomeric units, depending upon the manner and degree of polymerization. In general, these polymers are linear in structure and have an average molecular weight of less than about one million, preferably up to 100,000, depending upon the type of polymer and usually within the range of about 5,000 to about 500,000. The term "polyvinyl alcohol" includes polymers of vinyl alcohol, prepared from polyvinyl acetate or similar polyvinyl esters by replacement of acetate groups by hydroxyl groups. This chemical reaction is generally known as hydrolysis in the art.

The polyvinyl alcohol is often identified by its viscosity, and percent hydrolysis or percent ester content. The polyvinyl alcohol may be of low, medium or high viscosity and may be selected from the range of about 1.8 to 65, and preferably up to 15 centipoises. Reference to centipoises herein refers to determinations made in a 4% aqueous solution at 20° C. The product may contain minor amounts of polyvinyl acetate such as up to about 30% by weight and preferably from about 10 to 30% ester content. The degree of hydrolysis is thus usually within the range from about 70 to 100%. Suitable examples are polyvinyl alcohol which has a viscosity of about 10 to 15 cps.

It is preferred to employ polyvinyl alcohol having a viscosity of 10 to 15 centipoises and a polyvinyl acetate content of about 10 to 30% by weight. This material exhibits maximum stability upon storage of the composition at elevated temperature resulting in maximum soil-suspending power for the composition. It may be prepared by any suitable manner such as by control of the degree of polymerization and alcoholysis of the polyvinyl acetate to produce a product of the above characteristics. The above viscosity range corresponds to an approximate weight average degree of polymerization of about 35 to 100.

If desired, a small amount of a plasticizer or color extender, typically up to about 5 parts per 100 parts of the polyvinyl alcohol solution may be present. Typical plasticizers include propylene glycol and glycerin.

The pH of the solution of polyvinyl alcohol is typically about 6.5 to 7.5, preferably about 7. The pH of the solution is compatible with enzyme stability.

The water-soluble alkali metal carboxyalkyl cellulose is a hydrophilic colloid which is soluble or dispersible in water, and is generally classified as water-soluble. It is preferred to use the alkali metal salts of a carboxy lower alkyl cellulose having up to 3 carbons in the alkyl group, such as the sodium and potassium salts of carboxymethylcellulose. Sodium carboxymethylcellulose and the like are available usually in the form of powders in various grades of purity and viscosity in solution. Commercial grades of sodium carboxymethylcellulose having a purity from about 40 to 100% on a dry basis and which are of low, medium or high viscosity may be employed. The degree of substitution of the carboxymethyl group per anhydroglucose unit in the cellulose molecule is variable, but may be in the range from about 0.5 to 2. Another known water soluble carboxyalkylcellulose salt is sodium carboxyethylcellulose.

The binding agents discussed above are dissolved in water, typically at a concentration of 2–25% by weight, preferably about 2–15% and most preferably about 10% in the case of polyvinyl alcohol and about 3% in the case of alkali metal carboxyalkyl cellulose prior to contact with the enzyme-builder salt blend. The amount of solution contacted with the enzyme builder salt blend is such that the ratio of builder salt to solution is between about 49:1 and 17:3.

The laundry product containing binding agent and builder salt having bound thereto enzyme may be used in presoaking detergents or in washing detergents. In such compositions it is mixed, preferably by post-adding with

other commonly employed detergent components, in particular, an organic surface active agent, typically a spray dried organic surface active agent, is employed. Typically, the laundry product is present in amount of about 1–50% by weight of the finished detergent, and most preferably about 5%.

The surface active agent which may be employed may be any commonly used compound having surface active or detergent properties. Most preferred are those water-soluble surface active compounds having anionic or nonionic properties. 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, 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 rosin acids, such as may be derived from fats, oils and waxes of animal, vegetable or marine 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 decyl, undecyl, dodecyl (lauryl), tridecyl, tetradecyl, pentadecyl, or hexadecyl benzene sulfonate and the higher alkyl toluene, xylene and phenol sulfonates; alkyl naphthalene sulfonate, ammonium diamyl naphthalene sulfonate, and sodium dinonyl naphthalene sulfonate; sulfated aliphatic alcohols such as sodium lauryl and hexadecyl sulfates, triethanolamine lauryl sulfate, and sodium oleyl sulfate; sulfated alcohol ethers, such as lauryl, tridecyl, or tetradecyl sulfates including 2–4 ethylene oxide moieties; sulfated and sulfonated fatty oils, acids or esters, such as the sodium salts of sulfonated castor oil and sulfated red oil; sulfated hydroxyamides such as sulfated hydroxy-ethyl lauramide; sodium salt of lauryl sulfoacetate; sodium salt of dioctyl sulfosuccinate; and the sodium salt of oleyl methyl tauride.

Other anionic surface active agents which may be employed in the practice of this invention include olefin sulfonates, typically containing 8–25 carbon atoms.

Also included within the ambit of the invention are the sulfuric acid esters of polyhydric alcohols incompletely esterified with higher fatty acids, e.g. coconut oil monoglyceride monosulfate, tallow diglyceride monosulfate; and the hydroxy sulfonated higher fatty acid esters such as the higher fatty acid esters of low molecular weight alkylol sulfonic acids, e.g., oleic acid ester of isethionic acid.

Nonionic surface active agents are 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 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 monoesters of hexahydric alcohols and inner ethers thereof such as sorbitan monolaurate, sorbitol mono-oleate and mannitan monopalmitate and the condensation products of polypropylene glycol with ethylene oxide.

Cationic surface active agents may also be employed. Such agents are those surface active detergent compounds which contain an organic hydrophobic group and a cationic solubilizing group. Typical cationic solubilizing groups are amine and quaternary groups.

As examples of suitable synthetic cationic detergents there may be noted the diamines such as those of the type $RNHC_2H_4NH_2$ wherein R is an alkyl group of about 12 to 22 carbon atoms, such as N-aminoethyl stearyl amine and N-aminoethyl myristyl amine; amide-linked amines such as those of the type

$$R'CONHC_2H_4NH_2$$

wherein R' is an alkyl group of about 12 to 18 carbon atoms, such as N-amino ethyl-stearyl amide and N-amino ethyl myristyl amide; quaternary ammonium compounds wherein typically one of the groups linked to the nitrogen atom is an alkyl group of about 12 to 18 carbon atoms and three of the groups linked to the nitrogen atom are alkyl groups which contain 1 to 3 carbon atoms, including such 1 to 3 carbon alkyl groups bearing inert substituents, such as phenyl groups, and there is present an anion such as halogen, acetate, methosulfate, etc. Typical quaternary ammonium detergents are ethyl-dimethyl-stearyl ammonium chloride, benzyl-dimethyl-stearyl ammonium chloride, benzyl-diethyl-stearyl ammonium chloride, trimethyl-stearyl ammonium chloride, trimethyl-cetyl ammonium bromide, methyl-ethyl-distearyl ammonium chloride, dimethyl-propyl-myristyl ammonium chloride, and the corresponding methosulfates and acetates.

The surface active agent is typically present in amount of about 2–95% by weight of the detergent composition, preferably about 2–15% by weight in presoak detergents and about 7–35% by weight in washing detergents. The preferred surface active compounds are those having anionic properties.

The detergent to which the laundry product of the invention is post-added also typically contains a builder salt, which may be the same type of organic or inorganic builder salt as is present in the binding agent-builder salt-enzyme laundry product. The builder salt in the final detergent is generally present in the range of about 20–90% by weight, preferably about 30–90% by weight in presoak detergents and about 20–65% in washing detergents.

The polyvinyl alcohol and alkali metal carboxyalkyl cellulose binding agents, besides being effective in forming the novel laundry product of the instant invention, are also effective in inhibiting or preventing redeposition of soil. Thus, a complete detergent composition, if desired, may be formulated, omitting anti-redeposition agents or such agents may be added in smaller amounts than are usual. When employed, the anti-redeposition agent may be agents such as polyvinyl pyrrolidone or additional polyvinyl alcohol or sodium carboxymethyl cellulose. It is typically present in amounts of up to about 1% by weight of the detergent composition, although amounts up to about 5% by weight may be employed too, if desired.

The final detergent composition may also include minor amounts of dyes and pigments, optical brighteners, perfumes and preservatives. If desired, dyes and pigments can also be incorporated in the binding agent-builder salt-enzyme laundry product.

The detergent is typically added to an aqueous wash solution in concentration of about 1 to 10 grams, preferably 1.5 grams per liter of water. The temperature of the solution is 60° F.–140° F. usually 120° F. typically about 15.5° C.–60° C., usually 50° C.

The following examples are set forth in order to illustrate the invention. All amounts recited are by weight unless otherwise specified.

EXAMPLE I

7.5 parts of the proteolytic enzyme Alcalase are dry blended with 87 parts granular anhydrous sodium tripolyphosphate.

The protetolytic enzyme Alcalase is characterized as having its maximum protetolytic activity at a pH of 8.9. This activity as measured on the commercial enzyme available from Novo Industri A/S, Copenhagen, Denmark is about 1.5 Anson units per gram of the enzyme. The commercial enzyme is a raw extract of *Bacillus subtillis* culture and contains about 6% of pure crystallized proteolytic material.

5.5 parts of a 10% by weight aqueous solution of polyvinyl alcohol are then sprayed on and simultaneously agitated with the dry blend of Alcalase and sodium tripolyphosphate. The laundry product thereby formed is a granular material wherein the enzyme is substantially evenly distributed over the surface and the product is resistant to caking upon storage.

A solution of sodium carboxymethyl cellulose may be substituted for the polyvinyl alcohol solution to obtain a similar laundry product. Likewise, the enzymes Maxatase and Monzyme may replace Alcalase. Similarly sodium pyrophosphate and trisodium nitrilotriacetic acid may be substituted for sodium tripolyphosphate.

EXAMPLE II

The Alcalase-sodium tripolyphosphate-polyvinyl alcohol laundry product of Example I is post-added to the following detergent formulation in amount such that the final detergent contains 5% by weight of the laundry product.

Ingredients:	Parts
Linear tridecylbenzene sulfonate, sodium salt	21
Sodium tripolyphosphate	30
Borax	1
Sodium silicate ($Na_2O:SiO_2$; 1:2.35)	7
Optical brightener	0.5
Perfume	0.2
Water	10
Sodium sulfate, q.s. to 100 parts.	

Upon adding this detergent in an aqueous wash in amount of 1.5 grams per liter at 50° C., the detergent pours substantially free of dust and effectively removes stains such as gravy and blood from solid textile material.

Although this invention has been described with reference to specific examples, it will be apparent to one skilled in the art that various modifications may be made thereto which fall within its scope.

What is claimed is:

1. The process of preparing laundry product granules consisting essentially of a water-soluble binding agent selected from the group consisting of polyvinyl alcohol and sodium carboxymethyl cellulose and hydratable sodium tripolyphosphate builder salt and bound to and homogeneously distributed on the surface of said granules a powdered proteolytic enzyme having an activity of about 1.5 Anson units per gram of the enzyme at a pH of 8–9 comprising blending about 7.5 parts by weight of said powdered proteolytic enzyme with about 87 parts by weight of said hydratable sodium tripolyphosphate builder salt and contacting with said blend about 5.5 parts by weight of 10% by weight aqueous solution of said binding agent, while agitating and spraying said solution onto said blend.

2. The process of preparing the laundry product granules claimed in claim 1 wherein said binding agent is polyvinyl alcohol.

3. The process of preparing the laundry product granules claimed in claim 1 wherein said binding agent is sodium carboxymethyl cellulose.

4. The process of preparing a detergent composition which comprises incorporating the laundry product granules produced in accordance with claim 2 into a spray dried detergent composition consisting essentially of about 21% by weight of linear tridecylbenzene sulfonate sodium salt, about 30% by weight of sodium tripolyphosphate, about 1% by weight of borax, about 7% by weight of sodium silicate having a ratio of $Na_2O:SiO_2$ of about 1:2.35, about 0.5% by weight of optical brightener, about 0.2% by weight of perfume, about 10% by weight of water and the remainder sodium sulfate, said granules being present in amount of about 5% by weight of said detergent.

5. The laundry product granules produced in accordance with the process claimed in claim 1.

6. The detergent composition produced in accordance with the process claimed in claim 4.

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LEON D. ROSDOL, Primary Examiner

D. L. ALBRECHT, Assistant Examiner

U.S. Cl. X.R.

252—89, 132, 135, Dig. 12; 264—117

Dedication

3,781,228.—*Robert William McDonnell*, Teaneck, and *Maung Hla Win*, Berkeley Heights, N.J. LAUNDRY PRODUCT CONTAINING ENZYME. Patent dated Dec. 25, 1973. Dedication filed Sept. 23, 1973, by the assignee, *Colgate-Palmolive Company*.

Hereby dedicates to the Public the entire remaining term of said patent.

[*Official Gazette February 26, 1974.*]

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[*Official Gazette November 12, 1974.*]