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54 A detergent composition containing a novel anti-redeposition agent.

57 A detergent composition which has improved soil removal anti redeposition properties includes a soil anti-redeposition agent comprising a carboxymethylated soy polysaccharide material in an amount effective to promote soil removal and/or prevent soil redeposition on fabric during washing of the fabric with said composition.

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A DETERGENT COMPOSITION CONTAINING A NOVEL

ANTI-REDEPOSITION AGENT

This invention relates to a detergent composition which has improved soil removal and/or anti-redeposition properties and provides a process for producing the same.

Synthetic detergent compositions have been used commercially for a number of years for the removal of soil from fabric and these compositions generally comprise a number of different types of compounds or additives. Typically, this type of composition may include, although not necessarily be limited to: an organic detergent compound such as a surfactant or surface active agent; a builder component such as a phosphate salt which enhances the cleaning effectiveness of the surfactant by sequestering various metallic ions found in hard water; and finally a soil suspending or anti-redeposition agent to help the surfactant hold the soil particles in suspension and prevent them from being redeposited onto the fabric during washing.

The use of a soil anti-redeposition agent generally improves the whiteness of fabrics washed with the detergent composition since the anti-redeposition agent

suspends the soil in the solution once it has been removed from the fabric and prevents its redeposition onto the washed fabric. If the detergent composition has poor soil suspension properties during washing and the soil is allowed to be redeposited, or settle from the wash onto the washed fabric, the fabric will eventually acquire a gray appearance which represents an extremely undesirable result for a detergent.

A number of materials have been proposed for use as soil anti-redeposition agents but the most widely used material is carboxymethylcellulose, which has been added for a number of years to different types of detergent compositions used for the washing of fabrics to prevent redeposition of soil from the solution once the soil has been removed from the fabric by washing. Other materials which have been proposed as soil anti-redeposition agents include polyvinyl acetate, polyvinyl alcohol, sodium alginate and various modified starches, all of which are generally considered to be less effective than carboxymethylcellulose. Other types of soil anti-redeposition agents which have been described as having improved soil anti-redeposition properties over carboxymethylcellulose include polyvinylpyrrolidone (as described in U.S. 3,000,830) and a combination of carboxymethylcellulose and a gelatin protein (as described in U.S. 3,594,324). While the use of these

materials as soil anti-redeposition agents in detergents has been successful, nevertheless a need still exists for an improved material having better soil anti-redeposition properties and one which is readily adaptable and useful in a wide variety of detergent compositions.

We have now discovered a soil removal and anti-redeposition agent which has improved properties, yet which is convenient to use and readily adaptable to most detergent compositions.

The present invention thus consists in a detergent composition including, as an anti-redeposition agent, a carboxymethylated soy polysaccharide material.

This detergent composition is particularly suited for the washing of fabrics and has improved soil removal and anti-redeposition properties over detergent compositions of the prior art. The detergent composition of the present invention thus comprises at least an organic detergent compound and the carboxymethylated soy polysaccharide material, which is preferably the soy polysaccharide or "spent flake" fraction derived from soy isolate processing, and will normally also include a detergent builder. The carboxymethylated soy polysaccharide material is added

to said detergent composition in an amount effective to improve soil removal and/or prevent soil redeposition on fabric which has been washed with the detergent composition.

5           It has specifically been determined that the carboxymethylated soy polysaccharide material developed in the present invention has better soil anti-redeposition properties in detergent compositions typically used for the washing of fabric than  
10 carboxymethylcellulose which has been added by the prior art to detergent compositions for preventing the redeposition of soil during washing.

          A similar carboxymethylated soy polysaccharide material is disclosed in Japanese Patent Application  
15 Kokai No. 128658/78.

          The material which is employed in the production of the soil anti-redeposition agent used in the detergent composition of the present invention comprises a soy polysaccharide material which itself comprises a mixture  
20 of residual high molecular weight sugars and protein and is normally obtained by the removal of soluble proteins and carbohydrates from soy flakes, meal or flour; the heterogeneous polysaccharides contained therein are principally cell wall structural components of the

soybean cotyledons. This material is typically obtained as a by-product from the manufacture or production of a soy protein isolate and has hitherto been regarded as a waste or, at best, very low value product. The soy  
5 polysaccharide product has also been sometimes characterized as the aqueous alkaline-insoluble residue or "spent flake" fraction from soy isolate processing.

In order to illustrate the manner in which the soy polysaccharide material is obtained from a typical  
10 process for the production of a soy isolate, the soybean starting material, such as soy flakes, meal or flour, is dispersed in an aqueous medium and the pH of the medium is adjusted to a value of at least 6.0, more commonly at least 7.0. The flakes, meal or flour is then washed  
15 with an aqueous alkaline solution and subjected to centrifugation. The soluble carbohydrate and protein components are dissolved in the aqueous extract and the solid residue comprises the bulk of the soy polysaccharide material in the soy flakes. This solid  
20 residue is sometimes referred to as the "spent flakes" residue and would be typically discarded as a waste product in a soy isolate process. The alkali-insoluble residue or solids is then customarily subjected to a second extraction with an aqueous alkali solution to  
25 exhaustively extract residual protein, after which all aqueous extracts containing soluble carbohydrates and

protein are combined for further processing and subsequent acid reprecipitation of the protein isolate. The residual product from the extraction of soluble carbohydrates and protein may then be cooked, dried or  
5 further processed, for example, to remove extraneous material if desired. This soy polysaccharide product or fraction, after it has been dried, is the preferred starting material for making the soil anti-redeposition agent of the present invention.

10 The above description represents only a typical process for obtaining a soy polysaccharide material and the present invention is not intended to be limited by any specific process or manner of obtaining the soy polysaccharide material or spent flake fraction which  
15 can be used as a starting material in the present invention. The production of the soy polysaccharide material represents a conventional process known to those skilled in the art for the separation of protein material from soy flour, flakes or meal and other soy  
20 products.

The soy polysaccharide material or spent flake fraction has a typical analysis after drying of: total carbohydrate content about 80% by weight; protein content about 12 to 16% by weight; and ash content about  
25 4% by weight. This soy polysaccharide material

generally comprises the many high molecular weight carbohydrate polymers contained in soybeans and, as previously noted, is normally used as a collective term to describe the fibrous carbohydrate material obtained from the cell wall structural components of soybeans, including soy flakes or meal.

The soy polysaccharide material or spent flake fraction is preferably dried and ground prior to formation of the soil anti-redeposition agent which is used in the detergent composition of the present invention. Although the exact particle size to which the soy polysaccharide material is ground is not critical to the practice of the present invention, it is preferred for purposes of forming the carboxymethylated soy polysaccharide material that the soy polysaccharide material be ground to a particle size such that substantially all of it will pass a 100 mesh screen (Tyler). Furthermore, although the exact moisture level for the soy polysaccharide fraction is not critical to carrying out the carboxymethylation reaction, it is preferred that the soy polysaccharide fraction or material should, as previously noted, be dried, preferably to a moisture content which does not exceed about 12% by weight and preferably is between 6 and 10% by weight of the material.

The present invention is not intended to be limited by the exact manner in which carboxymethylation of the soy polysaccharide material is carried out since a variety of conditions, reactants and equipment are well known to those skilled in the art.

A preferred method of producing the carboxymethylated soy polysaccharide material is to add the ground, dried, soy polysaccharide fraction to an aliphatic alcohol, preferably a  $C_1-C_6$  alcohol, more preferably a  $C_1-C_4$  alcohol, such as methanol, ethanol, isopropanol or butanol, which acts as a reaction diluent, and preheat the mixture to a temperature of from 30 to 60°C, with mixing. The weight ratio of alcohol to polysaccharide material is preferably from 3:1 to 12:1 and more preferably about 7:1.

Sufficient alkali, such as sodium or potassium hydroxide, is then preferably added to provide a mole ratio of alkali to cellulose (single glucose unit) which is preferably from 1.0:1 to 3.0:1. This is followed by the addition of a carboxymethylating agent, such as monochloroacetic acid or suitable salt thereof, preferably in a mole ratio to cellulose of from 0.4:1 to 0.8:1.

The entire reaction mixture is preferably continuously agitated at a temperature of from 30 to 60°C, preferably for a period of from 1 to 8 hours, in order to provide substantial carboxymethylation of the soy polysaccharide material. Following formation of the carboxymethylated soy polysaccharide material, the solids and reactants may be removed by washing with an alcohol (such as those exemplified above). The carboxymethylated soy material may be concentrated by filtration or centrifugation. The concentrated carboxymethylated soy material is then preferably dried in a conventional manner to form a product suitable for mixing with a detergent composition.

Since the reaction is usually and preferably effected in the presence of an alkali, the product will, in most cases, be the salt of the carboxymethylated soy polysaccharide material. This salt is preferably an alkali metal salt, particularly the sodium or potassium salt, although many other salts are possible. The sodium salt is generally most convenient.

The detergent composition of the present invention is then formulated in the same way as conventional detergent compositions but employing the carboxymethylated soy material as the anti-redeposition agent in place of carboxymethylcellulose or other conventional

agent. The detergent composition of the invention employs at least an organic, normally synthetic, detergent substance or surfactant which can be an anionic, nonionic, ampholytic or zwitterionic  
5 surfactant. A single one of these or two or more in combination may be employed to produce a detergent composition of the present invention.

The anionic organic detergent compounds or anionic surface active agents can include detergent compounds  
10 which contain an organic hydrophobic group and an anionic solubilizing group. Typical examples of anionic solubilizing groups are the sulphonate, sulphate, carboxylate and phosphate groups. Examples of suitable anionic detergents which could be used in the invention  
15 include the water-soluble salts of higher fatty acids or resin acids, such as may be derived from fats, oils and waxes of animal or vegetable origin, and the sulphated and sulphonated synthetic detergents. Also included within the class of suitable organic detergent compounds  
20 are the synthetic anionic detergents such as the higher alkyl aryl sulphonates (e.g. the higher alkyl benzene sulphonates) as well as the sulphates of higher alcohols, such as sodium lauryl sulphate.

Nonionic synthetic detergent compounds do not ionize  
25 in solution and the whole molecule acts as the cleaning

agent. Amongst the compounds which can be used in the present invention are those which can be broadly defined as compounds produced by the condensation of alkyl oxide groups (which are hydrophilic in nature) with an organic hydrophobic compound (which may be aliphatic or alkyl-  
5 aromatic in nature). The most widely used class of nonionic synthetic detergents includes those which are formed by condensing ethylene oxide or propylene oxide with a hydrophobic base. However, other suitable  
10 nonionic organic synthetic detergent compounds include the polyethylene oxide condensates of alkylphenols, as well as condensation products of materials such as ethylene oxide and the product resulting from the reaction of propylene oxide and ethylene diamine, the  
15 condensation product of aliphatic alcohols with ethylene oxide, long chain tertiary amine oxides and long chain alkyl phosphates.

Ampholytic synthetic detergent compounds are mainly derivatives of aliphatic secondary or tertiary amines.  
20 An example of a specific compound within this general grouping is sodium 3-dodecylaminopropionate. Ampholytic surfactants have both positive and negative centres and can assume either a positive or negative charge depending on the pH of the solution.

25 Zwitterionic synthetic detergent compounds behave

somewhat like nonionic surfactants and most are derivatives of aliphatic, araliphatic and aromatic quaternary ammonium, phosphonium and sulphonium compounds. Examples of specific compounds falling  
5 within this definition are N-hexadecyl-N,N-dimethyl-N-propylammonium sulphonate and benzyl and phenyl analogues thereof, such compounds being especially preferred for their detergent characteristics in relatively cool water.

10 The above general groupings of organic detergent compounds may be used singly or in combination in the practice of the present invention. These represent specific illustrations of many of the numerous organic detergent compounds or surfactants which can find  
15 application within the scope of the present invention.

The present invention is further not intended to be limited to the physical form of the detergent composition since it can be made into any of several commercially desirable physical forms including powder,  
20 granular, flake, liquid and tablet forms.

The detergent composition of the present invention can further include conventional ingredients to improve other properties of the detergent composition. Included within this grouping of materials are the compounds

described typically as water-soluble builder salts, such as phosphates, which are added to enhance the cleaning power of the detergent composition. Furthermore, various other materials may also be present, for example, materials to improve the detergency of the composition and to modify its foaming properties in whatever manner desired as well as other conventional additives, such as various optical brightening agents and fluorescent whitening agents. Germicidal ingredients may also be added to improve the overall cleaning or disinfecting properties of the detergent composition of the present invention. The present invention is not intended to be limited by the exact contents of the detergent composition of the present invention since numerous materials are well known and well within the knowledge of those skilled in the art in the production of detergents.

Included as an essential ingredient of the detergent composition of the present invention is the carboxymethylated soy polysaccharide fraction as a soil removal and/or anti-redeposition agent. The carboxymethylated soy polysaccharide fraction is added to the detergent composition in an amount effective to improve soil removal and substantially prevent soil redeposition on fabric during its washing with the detergent composition. Typical amounts of carboxymethylated soy

polysaccharide fraction to be used are from 0.5 to 5% (dry weight) of the detergent composition and more preferably from 1 to 2% (dry weight) of the detergent composition. The use of the carboxymethylated soy polysaccharide fraction developed in the present invention has been shown to result in a detergent composition which has improved soil anti-redeposition properties over detergent compositions which contain conventional soil anti-redeposition agents such as carboxymethylcellulose or other soluble polymeric materials.

The following examples are given to illustrate specific embodiments of the present invention and specific improvements achieved therewith.

EXAMPLES

A soy polysaccharide material was obtained by forming an aqueous slurry of 100 lb (45 kg) of defatted soy flakes and adding thereto 1000 lb (453 kg) of water at 90°F (32°C). Sufficient calcium hydroxide was added to adjust the pH of the mixture to a value of 9.7. The soy flakes were extracted for 30 minutes, after which the aqueous solution containing dissolved protein and carbohydrates was separated from the extracted flakes by centrifugation. The extracted flake residue was then redispersed in 600 lb (272 kg) of water at a temperature of 90°F (32°C) and extracted for an additional 15 minutes. The extracted flakes were then removed by centrifugation from the mixture and the aqueous extracts were combined for the subsequent production of a soy protein isolate. The extracted or spent flakes were then dried to a moisture content of 9% by weight at an air temperature of 180°F (82°C) to produce a soy polysaccharide material used in preparing the soil removal and anti-redeposition agent of the present invention.

415 grams of the dried soy polysaccharide material were then added to 3.8 litres of isopropanol and the mixture was heated to 40°C with agitation. 375 grams of a 50% by weight aqueous solution of sodium hydroxide and

125 grams of monochloroacetic acid were added simultaneously to the reaction mixture over a 30 minute period, with constant agitation. The reaction mixture was then continuously agitated and maintained at a temperature of 40°C for an additional 3.5 hours.

The reacted solids were removed from the mixture by filtration and dried at 190°F (88°C). This resulted in 500 - 600 g of carboxymethylated soy polysaccharide material.

The carboxymethylated soy polysaccharide material was then added to the following detergent composition at a level of 1% or 2% by weight of the composition (the amount of sodium sulphate was correspondingly 3% or 2% by weight).

Detergent Composition

<u>Ingredient</u>	<u>% by Weight</u>
Sodium Tripolyphosphate	30%
40% Linear Alkyl Benzene Sulphonate	45%
Sodium Metasilicate	10%
Sodium Carbonate	10%
Sodium Sulphate	3 - 2%
Brightener	0.5%
Carboxymethylated Soy Polysaccharide	1 - 2%

The detergent compositions containing the carboxy-methylated soy polysaccharide material were then evaluated for effectiveness in preventing the redeposition of soil on fabric during washing.

5 We used a Terg-O-Tometer Apparatus manufactured by United States Testing Company, Inc., which is a small scale, multiple unit washing machine that simulates the action of the agitator-type home washer. Swatches of fabrics were washed in the machines to determine a soil  
10 redeposition index.

Two different types of fabric were employed, a 100% cotton cloth and a dacron (trade mark)/cotton cloth with permanent press finish. Each cloth used had dimensions of 4 inches x 4.5 inches (10 x 11.5 cm).

15 For each test, soiled cloths of the above dimensions were washed between 1 and 5 times and the % soil removal and redeposition index were measured after each wash.

Wash time was 15 minutes, with two separate 2 minute rinses, a rinse temperature of 100°F (38°C) and a  
20 machine speed of 125 cycles/minute. The water had a hardness of 150 ppm and the detergent concentration was 0.15% by weight.

The heating bath of the Terg-O-Tometer was filled with water, the heaters were turned on, and the thermostat was adjusted to hold the bath at the required temperature. Solutions of the desired water hardness were prepared. They were heated to a temperature about 5°F (3°C) higher than that required for the test.

The soiled and unsoiled cloths were placed in the detergent solution and washed, the fabric was squeezed by hand after each rinse cycle. After the last rinse, the cloths were dried and ironed, if necessary. The reflectance of each cloth had previously been measured prior to washing using a Photovolt Reflectometer Model 670 equipped with a green filter. Following washing, the reflectance of each cloth was measured again. Soil removal is calculated for each cloth from the following:

$$\frac{A - B}{C - B} \times 100 = \% \text{ Soil Removal}$$

A = Average reflectance of soiled cloth after washing.  
B = Average reflectance of soiled cloth before washing.  
C = Average reflectance of unsoiled cloth without washing.

The higher the percentage of soil removal, the better is the soil removal ability of the product.

The soil redeposition index is calculated for each cloth by the following:

$$\frac{RA}{RB} \times 100 = \text{Redeposition Index}$$

RB

5 RA = Average reflectance of soiled cloth after test.

RB = Average reflectance of unsoiled cloth before test.

This represents the ability of the detergent to hold in suspension the soil that has been removed by washing. The higher the value, the better the product performance.

Employing the above test procedure, the detergent composition set forth above was tested containing 1% by weight carboxymethylated soy polysaccharide and compared against a control detergent which contained 1% by weight of carboxymethylcellulose in place of the carboxymethylated soy polysaccharide.

These results are set forth in Tables 1 and 2.

Table 1

Additive	% Soil Removal			
	Cotton (Avg)		Dacron/Cotton(Avg)	
	1 Wash	Washes 3-5	1 Wash	Washes 3-5
5 Carboxymethyl- cellulose	17.4	17.0	11.7	13.8
Carboxy- methylated Soy Polysaccharide	20.6	24.2	11.7	16.8

10

Table 2

Additive	Redeposition Index			
	Cotton (Avg)		Dacron/Cotton(Avg)	
	1 Wash	Washes 3-5	1 Wash	Washes 3-5
15 Carboxymethyl- cellulose	94.9	91.2	94.2	84.1
Carboxy- methylated Soy Polysaccharide	94.5	89.6	94.2	88.8

It may be seen from the above data that the carboxy-methylated soy polysaccharide was significantly better than the carboxymethylcellulose both for soil removal and in preventing the redeposition of soil.

CLAIMS

1. A detergent composition including, as an anti-redeposition agent, a carboxymethylated soy polysaccharide material.
2. A detergent composition according to Claim 1, wherein said carboxymethylated soy polysaccharide material is present in an amount of from 0.5 to 5.0% by weight of the detergent composition on a dry basis.
3. A detergent composition according to Claim 2, wherein said carboxymethylated soy polysaccharide material is present in an amount of from 1 to 2% by weight of the detergent composition on a dry basis.
4. A detergent composition according to any one of the preceding Claims, wherein said carboxymethylated soy polysaccharide material is obtained by carboxymethylation of a soy polysaccharide material having a moisture content that does not exceed 12% by weight.
5. A detergent composition according to Claim 4, wherein said soy polysaccharide material has a moisture content which is from 6 to 10% by weight of the material.
6. A detergent composition according to any one of the

preceding Claims, wherein the soy polysaccharide material that is carboxymethylated has a total carbohydrate content of about 80% by weight.

7. A detergent composition according to any one of the preceding Claims, wherein the soy polysaccharide material that is carboxymethylated has a protein content of from 12 to 16% by weight.

8. A detergent composition according to any one of the preceding Claims, wherein the soy polysaccharide material that is carboxymethylated has an ash content of about 4% by weight.