United States Patent

[11] 3,607,763

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[21] Appl. No. **878,989**
[22] Filed **Dec. 5, 1969**
[45] Patented **Sept. 21, 1971**

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Continuation-in-part of application Ser. No. 587,364, Sept. 26, 1966, now abandoned.

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[54] **PROCESS FOR THE PREPARATION OF LAUNDERING COMPOSITIONS**
10 Claims, No Drawings

[52] **U.S. Cl.** **252/137,** 252/152
[51] **Int. Cl.** **C11d 1/62,** C11d 1/86, C11d 3/06
[50] **Field of Search** 252/137, 152, 99

[56] **References Cited**
UNITED STATES PATENTS

3,325,414	6/1967	Inamorato	252/152 X
3,360,470	12/1967	Wixon	252/152 X

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ABSTRACT: A process for the preparation of a spray-dried, built, detergent-softener composition containing an ethoxylated nonionic detergent in combination with a di-higher alkyl quaternary ammonium textile softener is described which comprises the steps of (1) admixing the softener, nonionic detergent, and water to form an aqueous mixture containing a homogeneous dispersion of the softener; (2) incorporating an inorganic sodium or potassium phosphate builder in the aqueous mixture of step 1 to form a mixture containing more than 50 percent solids by weight, the temperature of the mixture being at least about 180° F. at the time the phosphate is introduced therein, and (3) spray drying the mixture of step 2.

PROCESS FOR THE PREPARATION OF LAUNDERING COMPOSITIONS

This is a continuation of Ser. No. 587,364, filed Sept. 26, 1966, now abandoned.

A PROCESS FOR THE PREPARATION OF LAUNDERING COMPOSITIONS

This invention relates to the preparation of laundering compositions and more particularly to a process for preparing spray-dried granular laundering compositions.

The application of Harold Eugene Wixon filed Aug. 2, 1966 bearing Ser. No. 576,493 entitled LAUNDERING COMPOSITIONS, now U.S. Pat. No. 3,360,470, which is incorporated by reference herein, concerns itself with a novel composition comprising a quaternary ammonium textile softener and an alkali metal carboxymethylcellulose. The function of the softening agent is to render fabrics and textiles "soft" with respect to handle, hand, touch, or feel. However, due to the presence of colored colloidal particles in most water supplies, these cationic softening agents, while "softening," have a tendency to yellow fabrics and, therefore an antiyellowing agent, i.e., the alkali metal carboxymethylcellulose, is introduced in sufficient amount to inhibit the yellowing effect. This particular softener composition can be used together with a detergent composition in the wash cycle rather than having it introduced in the rinse cycle, which is generally the case with fabric softeners.

The practice has been to form the components of the softener composition into granules and mix these granules with detergent composition granules to produce a commercial package. This is usually accomplished by using a "three-step" process, which merely means that the softener granules and the detergent granules are prepared separately by crutching and spray drying and the two kinds of granules are subsequently blended to produce the product. The "three-step" process as the term is used in the industrial sense is not given a literal interpretation. Actually, the first two steps are complete processes within themselves for crutching and spray-drying a particular composition, in this case a softener or detergent composition. The third step is concerned with the blending of the two components.

Although the "three-step" process produced a satisfactory product, it was uneconomical in that either two sets of crutching and spray-drying equipment were needed if preparation of the softener and detergent granules was to take place simultaneously or, in the alternative, the preparation time had to be extended if the same equipment was to be used first for one type of granule and then for the other type of granule. In both cases, of course, a blending machine was still necessary to mix the granules. It can be readily seen that the elimination of one of the crutching-spray drying procedures and the blending step not only would save equipment but would save time solids eliminate some material waste.

A "one-step" process was then attempted in order to eliminate the need for extra equipment and cut the process time by preparing the softener and detergent in a form whereby each granule contained by proportionate share of softener and detergent. It was felt that this process would also reduce segregation and contribute to a higher uniformity of product.

In order to carry out the "one-step" process, softener and detergent components as disclosed in U.S. Pat. No. 3,360,470 referred to above, were mixed in a crutcher under the assumption that after crutching, the mixture of components could then be spray-dried in a conventional manner; however, it was found that when more than 47 percent solids (by weight based on the total weight of the ingredients introduced into the crutcher) were present in the crutcher substantially irreversible gelation occurred to the extent that spray drying could not be effected. The "one-step" process was, therefore, unfeasible in an industry where generally more than 47 percent solids and preferably about 59 or 60–65 percent solids must be spray-dried in order to produce an economical product. In

view of the problem of gelation, it became apparent that it was more economical to prepare a softener-detergent product from the foregoing "three-step" process than to use a "one-step" process with 47 percent or less solids, especially in view of the fact that the gelation was found to be substantially irreversible even though water dilution and heating were applied to break the gel.

It is, therefore, an object of this invention to provide an economical "one-step" process for the preparation of spray-dried softener-detergent granules based on an amount of crutcher solids which is more than 47 percent by weight and preferably about 59 to about 65 percent by weight.

A further object is to provide a process whereby the spray-dried softener-detergent granules are such that the softener and detergent compositions are combined in each granule in a homogeneous relationship.

Other objects and advantages will become apparent from the following description of the invention.

In accordance with the invention, a "one-step" process has been found for preparing a spray-dried granular softener-detergent composition wherein each granule is composed of a homogeneous mixture of softener-detergent components comprising:

a. admixing water, a water-soluble nonionic detergent, and a quaternary ammonium salt at a temperature of at least 180° F., wherein the quaternary salt is homogeneously dispersed therein and the mixture contains more than 47 percent solids by weight;

b. introducing an alkali metal phosphate builder into the mixture; and

c. spray-drying the mixture.

It is preferred that at or about the time of introduction of the alkali metal phosphate, during step (*b*) an inorganic water soluble diluent to provide bulk and an alkali carboxymethylcellulose as an antiyellowing agent, be added. So long as the temperature of the mixture is at least 180° F., and the quaternary salt is dispersed, the order of addition of the alkali metal phosphate and the inorganic water soluble diluent (electrolytes) is immaterial. The alkali metal carboxymethylcellulose can be added at any point of the process.

The spray-dried product of this invention has been found exceptionally satisfactory in the following parameters: solubility, free flow, low dust content, storage, narrow distribution of particle sizes, bulk density, residual moisture content, and color. The granules, which, as previously mentioned, contain a homogeneous mixture of softener-detergent composition, are hollow and have sufficient strength to resist the stress and strain of handling which take place throughout the processing, packaging, and delivery stages of their commercial life. The final cup weight of the commercial product is preferably from about 60 to 125 grams, flowability about 70 percent to about 80 percent, and particle size about 16 to about 60 mesh. Cup weight is based on a cup which holds 237 grams of water.

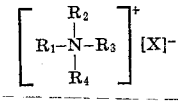
A more detailed description of the process of this invention follows:

The first part of the process is carried out in conventional crutching apparatus wherein initially water, a water-soluble nonionic detergent, and a quaternary ammonium salt are introduced simultaneously or in any order which is desirable to the operator. The preferred order is that stated.

All of the conventional water-soluble nonionic detergents can be used in the process of this invention, but are preferably in liquid or paste form. Generally, such nonionics have a hydrophobic group containing at least 8 to 30 carbon atoms. One particular class of such detergents is that formed by the oxyalkylation of fatty acids, alcohols, phenols, mercaptans, thiophenols, amines, and with ethylene oxide, propylene oxide, and other related alkylene oxides. Such materials usually have at least 5 mols of alkylene oxide, and preferably 5 to 30 mols of alkylene oxide, depending upon the particular hydrophobic group desired. Representative of these materials are those formed by condensation of ethylene oxide with alkyl phenols or alcohols. Particularly preferred herein are conden-

sates formed by the reaction of one mol of nonyl phenol or a mixture of C_{12}–C_{18} saturated, straight-chain, aliphatic alcohols with 8 to 12mols of ethylene oxide, the condensates containing an average of about 8 to 10 ethylene oxide groups per molecule. Some specific examples of this type of nonionic detergent are as follows: nonyl phenol-ethylene oxide condensates having an average of 9.5 ethylene oxide groups per molecule; a mixture of saturated aliphatic alcohols having from 14 carbons to 18 carbons in their chains and an average of 8.5 mols of ethylene oxide per molecule; tallow alcohol-ethylene oxide condensate having an average of 9 mols of ethylene oxide per molecule; and a 1:1 mixture of a C_{12} and a C_{14} saturated aliphatic alcohol having an average of 8.5 mols of ethylene oxide per molecule. Other alkylphenol condensates are those of diamylphenol, p-tert-octylphenol, 2,4-dicyclohexylphenol, m-pentadecylphenol, and benzyl-o-hydroxybiphenyl. Other condensates with alkylene oxide are those of tall oil, branched chain C_{14} to C_{17} aliphatic alcohols, lanolin, beeswax, bis-phenols, oxidized paraffin wax, napthenic acids, and fatty acyl alkanolamides. Mixtures of various water soluble nonionic detergents are contemplated.

The quaternary ammonium slat softener can be exemplified by the following general formula:



wherein R_1 and R_2 are alkyl groups each containing from 1 to 3 carbon atoms; R_3 and R_4 are aliphatic groups each containing from 12 to 22 carbon atoms; and X is selected from the group consisting of chlorine, bromine, and methyl sulfate. These compounds are readily dispersible in water. Specific examples are as follows: distearyl dimethyl quaternary ammonium chloride; distearyl dimethyl quaternary ammonium bromide; distearyl dimethyl quaternary ammonium methylsulfate; dicoco dimethyl quaternary ammonium chloride; dimethyl arachidyl behenyl quaternary ammonium chloride; dialkyl dimethyl quaternary ammonium chloride, the alkyl groups of which comprise a mixture consisting essentially of 24 parts hexadecyl 75 parts octadecyl and 1 part octadecenyl groups; the latter quaternary ammonium chloride is also known as dimethyl dihydrogenated ditallow ammonium chloride and is particularly preferred. Mixture of two or more cationic softener agents can be employed if desired. (The term "coco" refers to fatty acid groups formed in coconut oil fatty acids. Such acids contain from about 8 to about 18 carbon atoms per molecule, predominating in C_{12}–C_{14} acids). Other examples are dimethyl ditallow hydrazinium chloride and dimethyl ditallow quaternary ammonium methyl sulfate.

The proportions of the foregoing components which can be used are as follows: water—about 33 percent to about 43 percent and preferably about 35 percent to about 41 percent; nonionic detergent—about 3.5 percent to about 20 percent and preferably about 4.5 percent to about 8.5 percent; quaternary ammonium salt—about 1 percent to about 6 percent and preferably about 2.5 to about 4 percent. These percentages are by weight and based on the total weight of the initial materials as introduced into the crutcher. The proportions of the other components will be set forth below.

It is important that the quaternary ammonium salt be homogeneously dispersed in the mixture before the phosphate builder is added. This is accomplished by heating or agitation caused by merely admixing the water, nonionic, and quaternary salt or by external means. Both agitation and heating are discussed below. The homogeneous dispersion of the quaternary ammonium salt bears a similarity to a melt or solution of the salt in the water and nonionic.

Agitation, which is preferred and practical, can be initiated before, during, or after the water, nonionic detergent, and quaternary ammonium salt are introduced into the crutcher. The speed of agitation can range from about 100 revolutions per minute (r.p.m.) to about 500 r.p.m. and preferably from about 150 r.p.m. to about 400 r.p.m. and can be varied during the course of the process. The given rates are based on the used of conventional equipment. Actually, any reasonable movement or agitation of the mixture will suffice to carry out the process of this invention so long as it commences before or at the same time as the addition of the second group of components, although high shear agitation is not practical.

Before introduction of the phosphate, the materials in the crutcher should be a temperature of at least about 180° F. It is preferred to initially heat the first group of materials (water, nonionic, and quaternary salt) about 130° F. to about 160° F; however, there is no prohibition against raising the temperature at any time before the second group of materials is added. The particular mode of heating is not important and can be accomplished with whatever heating apparatus is available so long as the temperature can be ascertained. Once the temperature of the mixture has been raised to at least 180° F. and the quaternary salt is homogeneously dispersed, the second group of components can be introduced into the crutcher. There is actually no upper limit for the temperature of heating the crutcher materials other than keeping the temperature below the boiling point of water (212° F.). However, the most practical temperatures are from about 180° F. to about 200° F. and preferably from about 180° F. to about 190° F. If the temperature is less than 180° F. and particularly about 175° F. or less, gelation occurs after one or more of the second group of components is introduced to obtain more than 47 percent solids. It should be noted that between 175° F. and below about 180° F. there is a gray area in which gelation is not sharply apparent, but is sufficient to hamper economical spray-drying. By heating to at least about 180° F., this partial gelation is also avoided.

Once the proper temperature has been attained, and the quaternary salt homogeneously dispersed, the alkali metal phosphate, the inorganic water-soluble diluent, and the alkali metal carboxymethylcellulose can be introduced into the crutcher. Just as the first group of components, this second group has no particular order of introduction and each component can be introduced one at a time or the entire group can be added simultaneously, although preferably the order stated is the order followed. Agitation can be continued until a homogeneous mixture is attained.

Examples of the alkali metal phosphate builder salts are the alkali metal tripolyphosphates and pyrophosphates of which the sodium and potassium compounds are most commonly used. These phosphates are well known in the detergent art as builders and can either be used alone or as mixtures of different phosphates. More specific examples of phosphate builder salts are as follows; sodium tripolyphosphate; sodium phosphate, tribasic; sodium phosphate, monobasic; sodium phosphate, dibasic, sodium pyrophosphate; sodium pyrophosphate; acid. The corresponding potassium salts are also examples of satisfactory builders for use herein along with mixtures of the salts or corresponding mixed potassium-sodium salts.

Of the tripolyphosphates, a low phase 1 material is preferred, i.e., a phase 2 tripolyphosphate associated with a maximum of about 8 percent phase 1 tripolyphosphate, the phase 1 crystalline form being a high temperature rise material with hydrates more rapidly than the phase 2 material. The preferred tripolyphosphate form is conventional for spray drying operation and was used in Example I, below.

In order to give bulk to the composition, an inorganic water soluble diluent can be used which is exemplified by the preferred alkali metal (generally Na or K) sulfates such as sodium sulfate. These preferred diluents generally do not provide any substantial detersive activity. Other examples of diluents are sodium chloride and sodium carbonate. Diluents such as the silicates exemplified by sodium metasilicate and sodium silicates wherein the ratio of Na_2O to SiO_2 is from about 1.6:1 to about 3.2:1; borax; clays such as kaolin, the montmorillonites, and bentonites; silica sol; titanium oxide; colloidal aluminum hydroxide; and ammonium carbonate can

also be used; however, it is preferred that the major proportion of the diluent be an alkali metal sulfate, which can be considered an inert material.

The third component of the second group is an alkali metal (generally, Na or K) carboxymethylcellulose, in particular those having about 0.5 to about 0.8 carboxymethyl group per cellulose unit. This component is considered an organic builder and as noted in U.S. Pat. No. 3,360,470 referred to above provides the antiyellowing effect for the quaternary ammonium softener.

The proportions of the second group of components which are by weight based on the total weight of components introduced into the crutcher are as follows: alkali metal phosphate builder salt—about 13 percent to about 45 percent and preferably 27 percent to 33 percent; dilvent—about 7 percent to about 25 percent and preferably 17 percent to about 23 percent; and an alkali metal carboxymethylcellulose—about 0.3 percent to about 4.5 percent and preferably about 0.7 percent to about 2.5 percent.

In addition to the stated proportions, the proportions should be selected in a corresponding ratio so that there will be a sufficient amount of each component to provide more than 47 percent solids in the crutcher composition. It should be noted that even through 59–60to 65 percent solids are generally considered the most desirable for spray-drying, all of the solids' percentages above 47 percent and preferably about 50 percent are important because the bulk density of the composition after spray-drying is dependent upon the percentage of solids resulting from the crutching operation. Although it is not generally the case, there are some instances when it is commercially necessary to achieve a bulk density which will result from a lower solids percentage. However, this does not in any case normally go below the 50 percent solids level. About 65 percent solids is the upper practical limit for spray-drying because the high solids content becomes too difficult to pump; however, higher levels of solids can be crutched within the process of this invention, if desired.

Crutching time is generally from about 5 minutes to an hour or more with about 20 to 40 minutes being most desirable. Time is not critical and it has been found that about 30 minutes provides satisfactory homogeneity. Times greater than an hour provide no advantage and shorter than 5 minutes contribute to waste because of settling.

The spray-drying equipment used is just as conventional as the crutching equipment. It includes spray nozzles, a high pressure pump, an air spray system, a heater for the air, and a dust separator. Two types of systems make use of this equipment. They are known as parallel-current and countercurrent drying and of these two systems the countercurrent system is the most desirable; however, in many cases, both systems are used to take advantage of each.

Conventional colorants, perfumes, brighteners, and bluing agents can be added if desired. The amounts of these substances introduced into the softener-detergent composition are obviously such that they will not adversely affect the desired properties. The amounts of all of these substances taken together add up to less than 2 percent by weight of the total weight of the crutcher composition and preferably less than 1 percent. The colorants are usually in the form of dyes. Examples of the brighteners are stilbene, triazole, and benzidine sulfone compositions, the stilbene and triazole compositions in combination being preferred. These components are, of course, not an essential part of the softener-detergent composition used in the process of this invention. The colorant and perfume are preferably added during or after spray-drying whereas the brighteners are added to the crutcher mixture. The bluing agent is usually added to the crutcher mixture after the water and it is a preferred component because of its antiyellowing contribution. An example of a bluing agent is ultramarine blue which has been found to be stable to light, alkali, and bleach and is insoluble in water. Ultramarine blue is a well-known blue pigment occurring naturally as the mineral lapis lazuli and can be synthesized by igniting a mixture of kaolin, sodium carbonate or sulfate, sulfur, and carbon. The micropulverized, synthetic ultramarine blues have proved satisfactory.

The proportions of components which can be present in the final product in percent by weight based on the total weight of the final composition are as follows: water—about 5 percent to about 15 percent and preferably about 6.5 percent to about 12.5 percent; nonionic detergent—about 5 percent to about 30 percent and preferably about 7 percent to about 12 percent; quaternary ammonium salt—about 2 percent to about 8 percent and preferably about 4 percent to about 6 percent; alkali metal phosphate builder salts about 20 percent to about 65 percent and preferably about 40 percent to about 48percent; dilvent—about 10 percent to about 37.5 percent and preferably about 25 percent to about 35percent; an alkali metal carboxymethylcellulose—about 0.5 percent to about 5.33 percent and preferably about 1 percent to about 3.5 percent; colorant, perfumes, brighteners, and bluing agents—total weight 0 percent to about 2 percent and preferably 0 percent to about 1 percent; bleaching agent—0 percent to about 10 percent and preferably 0 percent to about 5 percent.

The following examples are illustrative of the invention. Parts and percentages are by weight.

EXAMPLE I

The following crutcher formula (59.5 percent solids) was used in this example and the components were introduced in consecutive order as numbered:

Component	Percent in Crutcher	Percent in Final Spray-dried Product
1. water (exclusive of water mentioned below)	37.640	10.000
2. ultramarine blue	0.064	0.096
3. nonionic detergent (nonyl phenol—ethylene oxide condensate having an average of 9 ethylene oxide groups per molecule)	6.544	9.700
4. softener (dimethyl dihydrogenated ditallow quaternary ammonium chloride)	3.164	4.500
5. anhydrous sodium sulfate	18.140	27.840
6. a 43.5 percent solution of sodium silicate in water; ratio of Na_2 to SiO_2 is 2.35:1	3.378	2.200 (silicate)
7. sodium tripolyphosphate	29.735	44.300
8. a 75 percent dispersion of sodium carboxymethylcellulose (CMC) in water	1.213	1.180 (CMC)
9. a stilbene brightener	0.083	0.184
10. a triazole brightener	0.039	

The procedure was as follows: Steam at atmospheric pressure was directed continuously against the jacket of a crutcher equipped with a paddle mixer after which water was introduced into the crutcher at 145° F. Ultramarine blue was then added with medium agitation (150 r.p.m.), followed by the nonionic detergent without agitation. The quaternary ammonium softener was then introduced, medium agitation (150 r.p.m.) commenced and the temperature raised to 180° F. until the quaternary salt was homogeneously dispersed. Sodium sulfate and sodium silicate were added consecutively and then the tripolyphospate was introduced with an increase in

agitation (350 r.p.m.), after which the CMC and the brighteners were added. The crutching time was 30 minutes.

The slurry was then pumped with a triplex pump into a spray tower where it was met with a countercurrent of hot air. The dried particles dropped to a conveyor and were transported to an air lift, which passed the granules along to a separator. Screening, perfuming, and packaging followed.

The spray tower conditions were as follows:

tower inlet air temperature	510°–520° F.
tower outlet air temperature	185°–269° F.
nozzle size	10
number of nozzles	4
triplex pressure	660 p.s.i.g.
Product cup weight	81–100 grams

The spray-dried product was found to perform well in softening and laundering.

EXAMPLE II

The same crutcher steps and conditions as in example I are followed except the order of addition was water, blue, sulfate, silicate, tripolyphosphate, nonionic detergent, CMC and brighteners, and quaternary ammonium salt. Agitation is used for all ingredients except the nonionic detergent. The 180° F. temperature was invoked before the addition of sulfate.

This procedure produces gelation

EXAMPLE III

The same crutcher steps and conditions prevalent in example I are followed except that instead of 180° F., a temperature of 175° F. is used.

This procedure produces a gelation

EXAMPLE IV

The same crutcher steps and conditions are followed as in examples II and III except that in each case proportions are adjusted to reduce the level of crutcher solids to 47 percent.

No gelation occurs.

EXAMPLE V

The same crutcher steps and conditions are followed as in example I except that proportions are adjusted to reduce the level of crutcher solids to 50 percent.

No gelation occurs.

EXAMPLE VI

Example I is repeated except that the proportions are adjusted to obtain 65 percent solids.

No gelation occurs.

EXAMPLE VII

Example I is repeated except that the CMC and brighteners are added just before the tripolyphosphate.

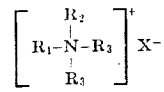
No gelation occurs.

EXAMPLE VIII

The procedure of examples II and III is followed to determine which of the components has an effect on gelation. This is accomplished by elimination of components. It is ascertained that the elimination of either the nonionic detergent, the quaternary ammonium salt or the phosphate from the crutching formula results in no gelation.

We claim:

1. A process for preparing a spray-dried granular softener-detergent composition consisting essentially of (*a*) about 2 percent to about 8 percent by weight of a quaternary ammonium textile softener represented by the general formula



wherein R_1 and R_2 are alkyl groups each containing from 1 to 3 carbon atoms; R_3 and R_4 are alkyl groups each containing from 12 to 22 carbon atoms; and X is selected from the group consisting of chlorine, bromine, and methyl sulfate; (*b*) about 5 percent to 30 percent by weight of a water-soluble nonionic detergent having a hydrophobic group of 8 to 30 carbon atoms condensed with 5 to 30 moles of ethylene oxide; (*c*) about 20 percent to 65 percent of a sodium or potassium inorganic phosphate builder salt; and (*d*) 5 to 15 percent water which comprises the steps of: (1) admixing water, said nonionic detergent, and quaternary ammonium softener to form an aqueous mixture wherein the quaternary softener is homogeneously dispersed; (2) introducing a sodium or potassium inorganic phosphate builder salt into said aqueous mixture whereby a mixture containing more than 50 percent of solids is formed, the temperature of the mixture being at least about 180° F. and below 212° F. at the time said phosphate is introduced therein; and (3) spray drying the mixture of step 2.

2. A process in accordance with claim **1** wherein said water, said nonionic, and said softener are admixed at a temperature in the range of about 130° F. to about 160° F. and the temperature of said mixture is raised to at least about 180° F. prior to the introduction of said phosphate.

3. A process in accordance with claim **1** wherein said water, said nonionic, and said softener are admixed at a temperature of at least 180° F.

4. A process in accordance with claim **1** wherein at least one water-soluble inorganic diluent selected from the group consisting of sodium sulfate, potassium sulfate, sodium carbonate, sodium chloride and sodium silicate is introduced prior to, simultaneously with, or after said introduction of said phosphate, the amount of said diluent being sufficient to provide 10 to 37.5 percent by weight of said diluent in the spray-dried product.

5. A process in accordance with claim **1** wherein a sodium or potassium carboxymethylcellulose salt is introduced prior to, simultaneously with or after said introduction of said phosphate, the amount of said cellulose being sufficient to provide 0.5 to about 5.33 percent by weight of said cellulose in the spray-dried product.

6. A process in accordance with claim **1** wherein the temperature of the mixture of step 1 is about 180° F. to about 190° F. at the time said phosphate is introduced.

7. A process in accordance with claim **1** wherein said phosphate builder salt is sodium or potassium tripolyphosphate.

8. A process in accordance with claim **4** wherein said diluent is sodium or potassium sulfate.

9. A process in accordance with claim **8** wherein said sulfate is added after said phosphate salt and a sodium or potassium carboxymethylcellulose is introduced thereafter, the amount of said cellulose being sufficient to provide 0.5 percent to about 5.33 percent by weight thereof in the spray-dried product.

10. A process in accordance with claim **9** wherein the mixture formed and spray dried contains about 59 percent to about 65 percent solids by weight.