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(54) **Process for manufacture of coloured detergents speckles.**

(57) A process for manufacturing coloured speckles for use in washing powder involves in the following steps:
(a) forming a molten mixture of surfactant and colourant;
(b) solidifying the mixture, for example by spraying it onto a water-cooled rotating drum (15);
(c) milling the solid material to particles of a suitable size for use as speckles; and
(d) separating the desired particles by elutriation.

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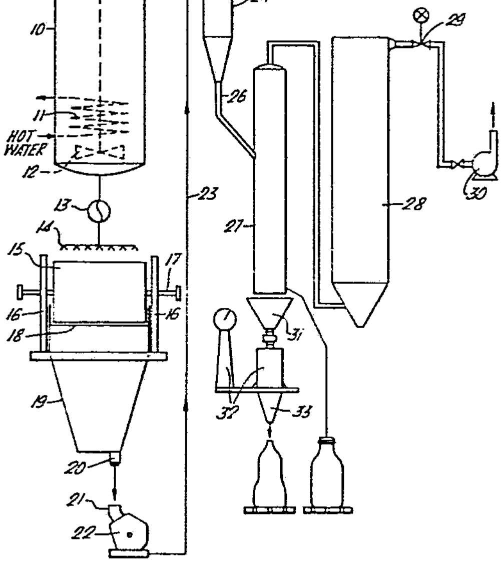

TALLOW ALCOHOL 25EO
POLAR BRILLIANT BLUE GAN
HOT WATER
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PROCESS FOR MANUFACTURE OF COLOURED DETERGENTS SPECKLES

This invention relates to a process for the production of coloured speckles for use in a washing powder and to the speckles obtained by the process.

Coloured speckles are considered by manufacturers to be desirable components of washing powders since there is a substantial body of evidence that customers prefer speckled powders to plain ones. However, to obtain speckles of the correct size and size distribution, so that they have the desired appearance in a white powder, and to obtain them of the desired density so that they do not segregate from the body of the powder, is not at all easy.

Many methods of making speckles have been suggested. The simplest is perhaps that in which a portion of the spray-dried powder, or a component of it such as sodium tripolyphosphate, is sprayed with a colourant solution. That is not really satisfactory because the larger, visible particles become unevenly coloured and because a high proportion of finely-divided coloured material is produced which imparts a slight overall hue to the powder, whereas what is required is a white powder. Spray-drying of coloured crutcher slurries is a second method which has been suggested but although this produces satisfactory speckles, the process itself is inconvenient in that the spray-drying tower needs to be cleaned after each batch of speckles is prepared, interrupting production. Granulation processes have also been proposed, but these are extremely labour intensive and can produce messy working conditions in the factory. Furthermore, unless the processes are carefully controlled, the resulting speckles can be extremely dense and can segregate.

We have now discovered how to manufacture speckles in a wide range of sizes which are non-segregating.

In accordance with the present invention there is provided a process for the production of coloured speckles suitable for incorporation into a washing powder which comprises the steps of

(a) forming a molten mixture comprising a surfactant which is solid at room temperature and a colourant;
(b) forming solid material from the molten mixture;
(c) milling the solid material to form speckles; and
(d) separating the speckles from extraneous material by elutriation.

The process of the invention permits the preparation of speckles which are entirely phosphate-free, which is desirable for regions in which local laws prohibit the sale of washing powder containing phosphate.

The speckles can be prepared from any surfactant which has a melting point sufficiently high for it to be liquefied and solidified again to form a solid material, such as a flake or a bead, at room temperature. Although tallow alcohol ethoxylated with an average of 25 moles of ethylene oxide per mole of alcohol (hereafter referred to as tallow alcohol 25EO) is the preferred material, other surfactants can be used. The melting points of nonionic surfactants are somewhat molecular weight dependent and also dependent on the degree of branching in the hydrophobic part of the molecule. Straight chain primary alcohols in the C_{12}-C_{15} range begin to be sufficiently solid to be used in the process of the invention when they are ethoxylated with about 10 moles of ethylene oxide per mole of alcohol. More highly branched alcohols have lower melting points and very much higher molecular weights must be reached before sufficiently solid materials for use in the invention are encountered.

All of the anionic surfactants which are fusible and solid at room temperature can be used in this invention. Examples of these are sodium dodecylbenzene sulphonate and sodium primary alkyl sulphates.

The surfactant preferably has a melting point of above 35°C and more preferably between 40°C and 80°C.

The nature of the colourant is not critical, provided that it does not interact significantly with any of the components of the powder. The speckles can be prepared in any colour desired although darker colours make more visual impact than light ones. Blue is a particularly preferred colour for speckles since the colourant can then serve as a blue-whitener in the formulation. Formulations containing the blue-whitener Polar Brilliant Blue GAW have been described, and this material is particularly preferred as a colourant for use in the invention.

Preferred levels of colourant in the speckles are from 0.01 to 0.20% by weight, preferably 0.05 to 0.15% by weight.

The step (b) in the process in accordance with the invention may be carried out in any manner which leads to the formation of solid material, either in the form of granules or in the form of a continuous or discontinuous film. The preferred manner of performing this step is to spray a molten mixture of colourant and surfactant onto a rotating drum through which coolant passes. Solidified material is then scraped off the surface of the drum by a knife, normally situated diamctrically opposite to the point where the spraying takes place. In this way coloured flakes of surfactant are produced.

In the next stage of the process the flakes are milled to produce speckles of the desired size. We believe that the optimum diameter for speckles made by the process of this invention is below 1500 microns, preferably between 500 and 1500 microns. Although the solid flake material can be milled in any manner, a particularly preferred manner of carrying out this step is to use a hammer mill. This type of mill is described in more detail below, but it consists essentially of a comminuting device and a sieving device. The sieve size can be altered to suit the process in hand, although only the upper size limit can be controlled. There is no control over the amount of fines material which is produced.

The final stage of the process is a separation stage. In this stage speckles of the desired size produced by milling of the coloured, spray-cooled flake material are separated from extraneous material, principally more finely-divided material than is required, by an elutriation technique.

Elutriation is a known technique for classifying particulate material which has been found especially suitable for use in the present process. It is described generally on page 899 of the book "Chemical Engineering" referred to later. Basically, the technique consists in blowing a stream of air up a column and introducing unclassified solid material into the column via a side tube. The air stream carries the more buoyant (and therefore smaller) particles away, leaving the heavier ones behind. In our application of the technique the velocity of the air stream is adjustable so that the fraction of extraneous solid material which is carried away can be controlled.

In our apparatus the elutriation column is transparent so that a visual check can be kept upon the transport phenomena inside.

The invention will be further described by reference to the accompanying drawing which illustrates schematically an apparatus for carrying out a process of making washing powder speckles.

The apparatus consists of a closed cylindrical vessel (10) fitted with heating coils (11) and a stirrer (12). The upper end of the vessel (10) is provided with two inlets, one for nonionic surfactant and one for dyestuff, these being shown schematically. The lower end of the vessel is provided with an outlet, also shown schematically, for coloured molten nonionic surfactant and the outlet leads to a pump (13) connected to a head (14). The head is arranged above a drum (15). Supports (16) carry a hollow shaft (17) on the major axis of the drum by means of bearings (not shown), arranged so that the shaft and drum are rotatable together. The shaft is hollow and has the double function of carrying the drum and also conducting cooling water to the inside of it. A knife edge (18) is arranged substantially tangentially to the drum so that the edge almost touches the surface thereof and both the drum and the knife edge are positioned vertically above a large funnel (19) provided with an outlet (20). The outlet communicates with the feed (21) of a hammer mill (22) which is an important component of the apparatus of the invention.

A hammer mill, as is known to those skilled in the art, consists of a screen, above which a high speed rotating disc is provided, to which are fixed a number of hammer bars which are swung outwards by centrifugal force. Two designs of such mills are illustrated on pages 883 and 884 of "Chemical Engineering", volume 2, by J M Coulson and J F Richardson, published by Pergammon Press, Oxford, in 1962.

The output from the hammer mill shown schematically is connected by line (23) to a cyclone separator (24) which has a gas/vapour outlet (25) and a solids outlet (26), the latter communicating with an elutriation column (27) which is transparent. The elutriation column is connected through filter column (28) and manometer valve (29) to a pump (30).

Immediately below elutriation column (27) is a funnel (31) leading to a weighing pan (32), the underside of which has a discharge (33) to a storage container (34). Alternatively a discharge from the elutriation column (27), shown schematically, by-passes the weighing pan (32) and leads directly to an alternative storage container (35).

When used in a process in accordance with the invention, granules of tallow alcohol 25EO are placed in the cylindrical vessel (10) and hot water is passed through the heating coils (11). The stirrer is turned on and when a sufficiently high temperature has been reached to liquefy the nonionic suractant, a dyestuff, such as Polar Brilliant Blue GAW, is injected into the molten mass, either in powdered form or pre-dispersed in a liquid nonionic surfactant. The mixture is stirred until homogeneous and is then pumped out of vessel (12) by pump (13) and discharges through head (14). The liquid mixture falls onto the surface of the rotating drum (15) which is cooled below the melting point of the coloured tallow alcohol 25EO by cold water passed into the drum through the hollow shaft (17). The liquid mixture consequently solidifies into a film which is scraped off the surface of the drum by the knife-edge (16) diagonally opposite the head.

The solid material which is scraped off falls into the funnel (19) and passes out through outlet (20) through the inlet (21) of the hammer mill (22). In the mill the solid material is milled to a maximum average diameter of 1400 μ and air lifted through line (23) to the cyclone separator. The solid particles separated by the cyclone are fed into the elutriation column (26). Air is sucked into the column between its base and the funnel (31) by pump (30). The pressure and hence the velocity of the air can be measured and controlled by the manometer/valve (29).

When the rising column of air meets the stream of milled particles entering the elutriation column (27) some of the particles are carried with the stream and some fall down the column into the hopper (31). The faster the air rises the greater the proportion of particles which are carried up the column. By controlling the pressure developed by pump (30) using manometer/valve (29) it is possible to adjust the proportion of particles which are carried up the column (27) and hence the size of those which fall into the hopper (31). In the present process the pressure is adjusted so that particles in the size range 500 to 1400 microns fall into the hopper and the smaller particles are carried up the column with the stream of air. These smaller particles are removed from the air stream in filter column (28).

The particles in the size range 500 to 1400 microns are weighed in the weighing pan (32) and stored until required in storage container (34). Alternatively they can be fed directly into the storage container (35).

When incorporated into a colourless spray-dried washing powder in an amount of up to 5% by weight, the speckles produced by the process of the invention are clearly visible in the powder and do not segregate from it.

CLAIMS:

1. A process for the production of coloured speckles suitable for incorporation into a washing powder which comprises the steps of
   (a) forming a molten mixture comprising a surfactant which is solid at room temperature and a colourant;
   (b) forming solid material from the molten mixture;
   (c) milling the solid material to form speckles; and
   (d) separating the speckles from extraneous material by elutriation.

2. A process in accordance with claim 1, wherein the surfactant comprises a nonionic surfactant.

3. A process in accordance with claim 2, wherein the nonionic surfactant comprises tallow alcohol 25EO.

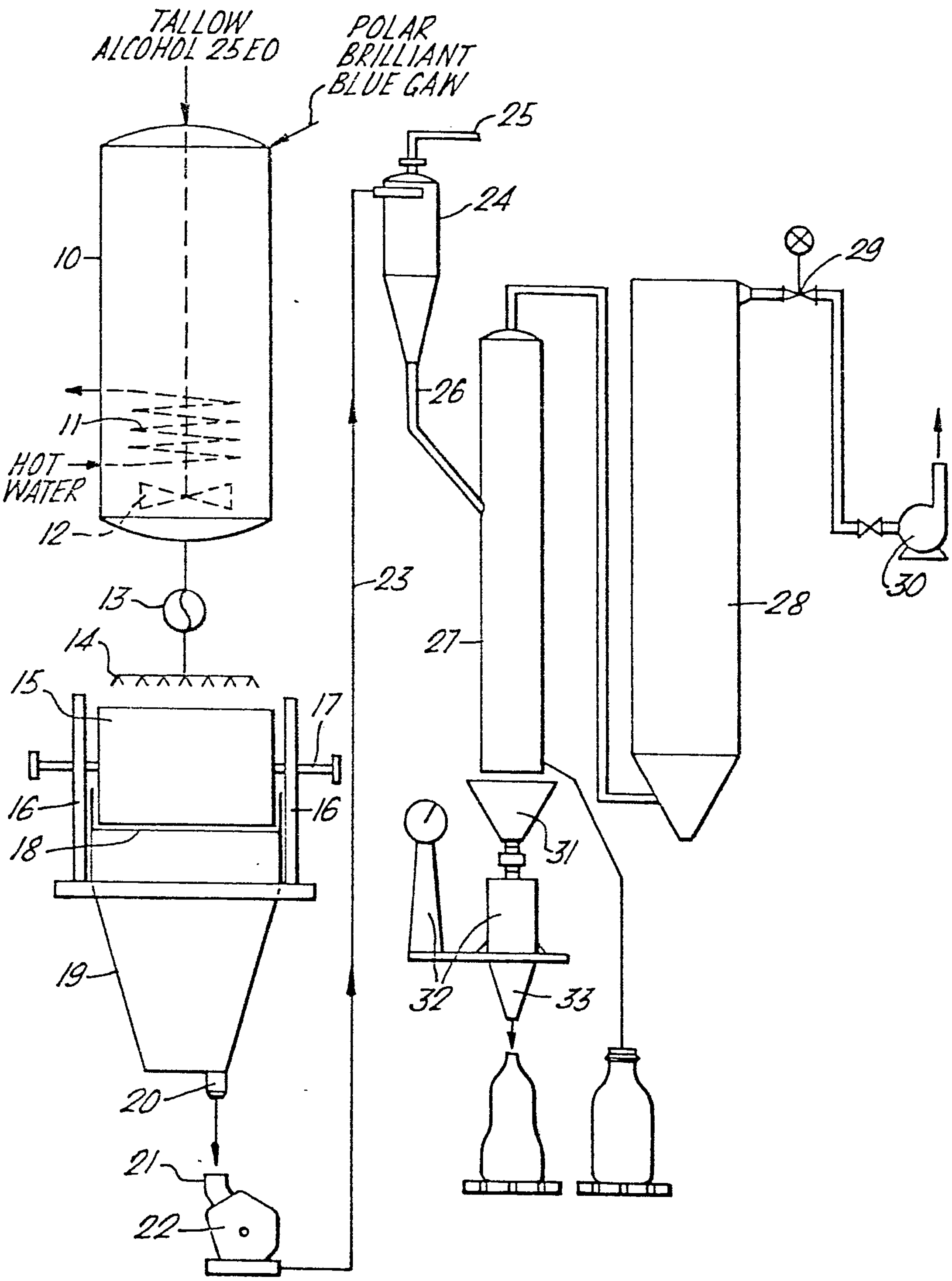
4. A process in accordance with any one of the preceding claims, wherein the mixture of surfactant and colourant comprises from 0.01 to 0.20% colourant.

5. A process in accordance with any one of the preceding claims, wherein the surfactant has a melting point above 60°C.

6. A process in accordance with any one of the preceding claims, wherein the solid material is formed by spray-cooling the molten mixture onto a rotatable drum.

7. A process in accordance with any one of the preceding claims, wherein the solid material is milled to a maximum diameter of 1500 microns.

8. A process in accordance with any one of the preceding claims, wherein the speckles separated from the extraneous material have diameters in the range 500-1500 microns.


TALLOW
ALCOHOL 25 EO
POLAR
BRILLIANT
BLUE GAW
HOT
WATER
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European Patent Office

EUROPEAN SEARCH REPORT

Application number
EP 80 30 1488.5

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim
P	US - A - 4 162 228 (E.C. ROBLES) * claims 1 and 4 * --	2,4,5
A	US - A - 4 082 682 (J.T. INAMORATO et al.) * complete document * --	
A	FR - A1 - 2 318 924 (THE PROCTER & GAMBLE COMPANY) * complete document * ----	

CLASSIFICATION OF THE APPLICATION (Int. Cl.³)

C 11 D 17/06
C 11 D 3/40
C 11 D 1/66

TECHNICAL FIELDS SEARCHED (Int.Cl.³)

C 11 D 1/00
C 11 D 3/00
C 11 D 17/00

CATEGORY OF CITED DOCUMENTS

X: particularly relevant
A: technological background
O: non-written disclosure
P: intermediate document
T: theory or principle underlying the invention
E: conflicting application
D: document cited in the application
L. citation for other reasons

&: member of the same patent family, corresponding document

X The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
Berlin	18-07-1980	SCHULTZE