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AMENDED SPECIFICATION

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PATENT SPECIFICATION

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COMPLETE SPECIFICATION

Detergent Compositions

5 We, THOMAS HEDLEY & CO. LIMITED, a company organised under the laws of Great Britain, of Phoenix Buildings, Collingwood Street, Newcastle-upon-Tyne, 1, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

10 The object of this invention is to provide a granular detergent composition, the detergent being a soap and/or a synthetic detergent, having a distinctive and readily recognizable appearance so that persons will be able easily to identify it.

15 This object is achieved, according to the invention, by a detergent composition in which 2 to 30% by weight of the composition consists of coloured granules substantially uniformly distributed throughout the composition, the colouring of the granules, which are coloured by colouring matter not substantive to fabrics, being vivid so as to be sharply demarked from the background of the remaining granules, the said coloured granules consisting predominantly of granules whose large dimension is between 0.2 and 1.5 millimetres and the said coloured granules having a Munsell chroma of more than 4 and a Munsell value of 4 to 7.

20 The factors involved in obtaining the distinctive speckled appearance are complex.

25 (1) It is essential that only a minor portion of the granules should be coloured so as to be sharply demarked from the background of the bulk of the granules. The quantity of granules so coloured is preferably from 2% to 30% of the total quantity of granules in the product. If less than about 2% of the granules are 30 distinctively coloured the effect will not be striking, nor pleasing, nor readily discernible.

[Price 4s. 6d.]

If, on the other hand, too many of the granules are distinctively coloured, the effect tends to be one of overall colouration (somewhat diluted) and lacks distinctiveness.

45 (2) The granules used to give the distinctive appearance must be vividly coloured. If one mixes granules of the pastel shades to which granules have been uniformly coloured in the past with uncoloured detergent granules, the contrast is small and the effect neither pleasing nor distinctive. On the contrary, when coloured granules are vivid, the effect is striking and the distinctive appearance of the speckled product can be readily appreciated despite movement of the composition as when it is poured from a carton in scullery or laundry use.

50 The distinctively coloured granules must stand out from the background colouration. The attributes of colour are three in number (a) hue, (b) brightness or value, and (c) saturation, purity or chroma. The hue may be chosen as desired throughout the whole range, inclusive of black, but there are distinct limitations on the other attributes. In the Munsell system of colour notation, see the *Munsell Book of Colour*, Munsell Colour Co. Inc., Baltimore, Maryland, 1929, the distinctively coloured granules of this invention should have a Munsell Value of about 4 to 7, and a Munsell Chroma of more than 4.

55 Using another colourimetric system (see *The Journal of the Optical Society of America*, 1940) the apparent reflectance of the granules, page 596, should be about .20 to about .45, and the excitation purity, see tables, p. 609 et seq., while varying somewhat with the hue, should be above about 20%.

60 (3) The distinctively coloured granules must contrast with the remainder of the granules in the product. A wide range of dis-

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5 tinctive appearances can be attained by adding distinctively coloured granules to an uncoloured bulk of detergent granules. Normally the uncoloured granules are white or off-white in appearance. This does not mean, however, that the bulk of the granules cannot itself be coloured, providing the colouration is of a different hue. Preferably, also, the colouration of the bulk of the granules will be of a light or pastel character, i.e., low in Chroma and high in Value by the Munsell scale. By way of example, strongly coloured blue granules in a pastel blue background would not provide such a distinctive speckled appearance as strongly coloured red granules in a pastel blue ground.

10 (4) The distinctively coloured granules need not all be of the same colour. Distinctive effects can be provided with coloured granules of two or more vivid hues in a bulk of granules which is white or light-coloured.

15 (5) The coloured granules should be of significant size. If they are too fine and powdery, they blend into the background and present a diffused or pastel appearance. The screen sizes mentioned below are Tyler Standard (W. S. Tyler Co., Cleveland, Ohio). A suitable screen test for coloured granules of reasonable uniformity and good visibility has been found to be

Less than about 1% on 14 meshes per linear inch

About 40% on 48 meshes per linear inch

35 Less than about 10% through 65 meshes per linear inch

The specks will then run from about 0.2 millimetres to about 1.5 millimetres in their large dimension. The preferred size would be all about 1 millimetre in large dimension but, in making granules, it is impossible to have all granules of the same size. For especially good appearance, the fines can be screened out preferably before colouring, using screens of 35 mesh or finer. The distinctive appearance is not furthered by the inclusion of strongly coloured granules which will pass through a 35-mesh screen; but with granules of the screen analysis set forth above, the inclusion of the fine material is not particularly significant as to appearance, and does not represent a significant economic loss.

50 (6) If all the granules in the composition have roughly the same general characteristics of size and density or specific gravity, the mixture is permanent in the sense that the distinctively coloured particles do not tend to segregate upon handling, jogging and the like. This would be true even if all of the granules were of the same size, providing the densities were not too disparate. Where the granules are of graded sizes, as for example, in the screen analysis indicated above, the packing effect helps prevent segregation, and con-

siderable differences in density may be tolerated. It will be usual to have the distinctively coloured granules and the bulk of the granules of approximately the same screen analysis. Compositions which consist of spray-dried granules in physical admixture with powdered builders such as phosphates, for example, may be made successfully in accordance with the invention.

(7) The colouring matter must be appropriately selected. It should be stable to alkali and should not be such that the skin of users will be sensitive to it, nor should it irritate the eyes. It should be tolerated in the feed of test animals to the point where it is not considered toxic. Moreover, of course, the material used to colour the detergent should not appreciably colour clothes washed with the detergent,—in technical terms it must not be substantive to fabrics, at any rate under washing conditions. Since the efforts of dye manufacturers are directed principally at developing dyes that are substantive to fabrics, the number of dyestuffs that may be used to colour detergent granules is limited. However, suitable pigments may be used as an alternative to dyestuffs.

The general characteristics required in the dyes have been set forth above. Certain fluorescent pigments or dyes known generally as Lumogens may be used. Certain basic dyestuffs of which Rhodamine B is an example, give good colourations and are non-staining to fabrics. The group of acid dyes offers a wider choice since on the whole they stain less than do the basic dyes and are sufficiently stable under alkaline conditions. Among acid dyestuffs which may be used, examples are Amaranth Red, Fast Crimson, Azure Blue and Wool Violet as listed in Part V of the 1955 Technical Manual and Year Book of the American Association of Textile Chemists and Colourists. Certain of these dyestuffs may be substantive when used under dyeing conditions but they are in fact non-substantive to the fabrics under washing conditions.

The following Examples are given for the purpose of illustrating the invention, the percentages and parts being by weight:—

EXAMPLE 1.

A spray dried synthetic-soap composition was used which contained about 20% of tallow soap and sodium alkyl benzene sulphonate (sodium dodecylbenzene sulphonate in which the dodecyl radical is derived from poly-propylene) 45% sodium tripolyphosphate, 6.7% of sodium silicate solids, 17% of sodium sulphate, 8% moisture, and 3.3% miscellaneous minor ingredients such as are customarily used. 100 Parts of these granules were accumulated. 10 Parts by weight were separated for colouring. This fraction was screened on a 35 mesh sieve and the fines removed. A 1% aqueous solution of Erio-

glaucine, a blue dye, was made. 0.4 Parts of this solution was sprayed on the coarser fraction of the 10 parts of granules set aside for colouring. The spraying was done in a tumble drum using an air atomizer. An inspection of the sprayed particles, under a low powered microscope shows that about half of the particles were coloured. The treated granules were mixed with the remaining 90 parts of the spray dried product in a revolving cylinder. The appearance was strikingly different from an uncoloured product and also from a product in which all of the granules were coloured by colouring matter added in the crutcher. Instead of "Erioglaucine" there may be used Patent Blue A—Hilton Davis Co.

EXAMPLE 2.

The use of Rhodamine B to colour granules is an excellent example of the utility of this invention. Rhodamine B in water solution, when added to the crutcher before spray drying in amounts of .0008% calculated on the dried product, colours the whole product pastel pink.

.0008%, ten times the percentage added to the crutcher, based on the weight of the granules to be coloured, of Rhodamine B in water solution at 0.5% strength, was sprayed on 10 parts out of 100 parts of uncoloured granules. The spraying was carried out as indicated in Example 1. When the sprayed product was united with the 90 parts of unsprayed product, the product was speckled with brilliant red particles.

Thus, there is produced with the same amount of the same dye, two differently appearing products—one entirely pastel pink, the other brightly speckled.

EXAMPLE 3.

Equal parts of the blue speckled soap of Example 1 and the red speckled soap of Example 2 were mixed. An unusual and distinctive red, blue and white appearance was achieved.

It is not necessary that the coloured granules be from the same batch or run as the uncoloured granules.

A further feature of the present invention resides in a particular process and apparatus for producing the distinctive detergent composition, which avoids various disadvantages involved in separately making granules of different characteristics and then mixing them together.

The process comprises continuously diverting a portion of a stream of granular detergent, colouring the diverted portion and returning it to the main stream so as to be blended therewith.

The colouring of the granules of the diverted stream is preferably done by spraying with the colouring matter in a liquid vehicle. The colouring matter may be in solution in the vehicle or dispersed or suspended

therein. It is preferred to use aqueous solutions of dyestuffs.

In the preferred performance of the process the diverted stream which has been coloured by spraying is caused to rejoin the main stream of the detergent granules which is conveyed, e.g., in an air lift, usually from a spray drying tower or from storage, in such a fashion as to cause a drying of the colour sprayed material as well as its thorough distribution in the main body of the granules.

The process, and apparatus for performing the process, will now be described by way of example with reference to the accompanying diagrammatic elevational drawing.

Referring now to the drawing, a granular product is shown as delivered to a vertical air lift conduit 3, by means of a belt conveyor 1. The granular product may be produced in any commercial fashion, but in most instances will be a spray-dried product. The belt conveyor 1 may deliver the granular product directly from a spray tower or from storage.

Within the air lift conduit 3, there will be maintained a stream of air of sufficient velocity to carry the product upwardly, with the exception that any substantially heavier impurities or exceptionally large lumps of granules will fall by gravity through a bell-shaped opening at the lower end of the air lift and be caught in a buggy 2 by means of which they may be returned to the crutcher if desired.

At the top of the air lift there is a gravity separator 4 which removes all of the product from the air stream excepting the extreme fines. These pass out a stack or conduit 5 to filters or other separating means. The fines so separated may also be returned to the crutcher feeding the spray tower.

The product removed by the gravity separator 4 passes through a conduit 6 or otherwise is conveyed to a vibrating screen 7, the primary function of which is to remove granules too large to dissolve readily when the product is used. The granules going through the screen will be of acceptable size; and in a carefully controlled procedure will normally have a screen analysis quite similar to that set forth above. The over-size material separated by the screen 7 is caught in a hopper 9 and transferred by conduit 13 to a buggy 14 to be returned to the crutcher.

The product passing through the screen 7 is caught by a hopper 8 and delivered to a conduit 10. Conduit 10 has two branches as shown. One marked 10a delivers the product to a product belt 12 for carrying it to packaging devices or the like. The other branch, 10b, may be used for delivering the product to storage buggies 11 as may be desired. A valve 15 controls the flow of the product from the conduit 10 to either of its branches.

A diverter conduit 16 is connected to the conduit 10 in such fashion as to divert continuously a certain percentage of the granules flowing through the conduit 10. The percentage so diverted may be treated as herein-
5 after set forth. The conduit 16 continuously delivers the diverted granules to holder 16a feeding a variable speed belt which in turn delivers the granules to a rotating drum 18
10 in which the granules are to be sprayed for colouration.

A source 19 of sprayable colouring material such for example as a dye solution, and provided with a pump 20, is connected by means
15 of a conduit 21 to a series of spray heads 22 within a rotating drum 18.

The colouring action occurring in the drum 18 is not necessarily thorough in the sense that all of the granules passing through the
20 drum will be equally coloured or coloured with the same intensity. While it is possible to secure complete colouration of all of the granules by the use of a relatively large quantity of the colouring solution, such an
25 operation is not preferred because it results in the treatment of the granules with a relatively large proportion of the water vehicle of the dyeing solution. This complicates the drying problem. It is ordinarily preferred to
30 so conduct the spraying treatment that a minimum quantity of vehicle is used although this may result in colouring less than the full number of granules passing through the drum 18. Colouring of individual granules is not
35 improved by the use of an excess of the dye. Where less than all of the granules are coloured in the drum 18, compensation may be made by diverting, as at 16, a larger percentage of the total output of granules.
40 For example, if 10% of the granules is diverted through the conduit 16, and half of these granules are coloured strongly in the drum 18, the result will be the addition of about 5% of vividly coloured granules to the
45 main stream of the product.

Coloured granules are delivered from the drum 18 to the hopper of a conduit 23 by means of which they return to the air lift 3.
50 If the percentage of fines has been increased by the handling due to colouration, these fines will be removed by the gravity separator 4. The turbulence in the air lift 3 will result in an essential mixing of the coloured granules with the non-coloured granules. Further, the
55 gases in the air lift will result in a sufficient drying of the coloured granules so that the moisture content of the completed product will be substantially unaffected.

It will be observed that a small fraction
60 of the granules will pass through the spray drum more than once. If the diverting means 16 withdraws 10% of the granules from the stream, on the second time around, 10% of these granules will be diverted through the
65 spray drum. Thus, 1% of the coloured

granules will be exposed to the colouring spray twice. It has not been observed that this produces any difference in the appearance of the coloured granules or in the appearance of
70 the completed product.

The following further Example is given to illustrate the process.

EXAMPLE 4.

Using the arrangements shown in the drawing, the diverter 16 was set so as to
75 divert 10% of the granules passing through the conduit 10. An 0.8% water solution of Erioglaucine was placed in the colour solution tank 19, and the speed of the pump 20 together with the speed of the variable de-
80 livery belt 17 were so regulated that there was sprayed on the granules going through the drum 22 an amount of the dye solution equal to 4% of the weight of the granules. The granules were discharged into the main
85 stream going through the air lift 3, by means of the conveyor 23. The product at the discharge end of the product belt had a distinctive blue speckled appearance and was found to be permanent and not subject to
90 segregation or modification of the general appearance after handling and storage.

If it is desired to remove an additional percentage of fines from the granules being
95 coloured in the drum 18, this may be accomplished by incorporating a screen in the colouring line ahead of the drum. The use of a screen of 35 meshes to the inch at this point will serve to remove substantially all materials
100 of such fineness that they do not contribute usefully to the speckled appearance of the product.

The use of the apparatus is not confined to the manufacture of a product in which a minor
105 percentage of highly coloured granules is mixed with a larger bulk of uncoloured granules. The bulk of the granules may itself be coloured as by crutcher colouring in known ways. A distinctive speckled appearance will
110 still be obtained with a small percentage of highly coloured granules provided the colouration of the bulk of the granules is of a contrasting hue and preferably of a light or pastel shade.

In instances where it is desired to produce
115 a product characterized by a relatively small percentage of multi-coloured granules in a larger bulk of lightly coloured or non-coloured granules, it is possible to provide one or more additional colouring lines, each
120 having means for diverting a portion of the main stream passing through conduit 10 and each delivering its product to the lower end of the air lift 3 for purposes hereinabove set forth. Thus, by way of example, a highly
125 attractive multi-coloured product may be made by mixing granules coloured blue and granules coloured red with a bulk of uncoloured or lightly coloured granules.

In the event that a product is being made
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- in which granules of soap or detergent are mechanically mixed with finely divided builders, such for example as phosphates, it is preferred to manufacture the soap or detergent granules, divert and colour a portion thereof, and mix the coloured granules with the uncoloured granules, all prior to admixture of the product with the pulverulent builders.
- 10 The treatment of the granules in the drum 18 is not limited to a treatment with colouring substances. Other treatment substances may be used in addition, including, but without limitation, anti-caking agents, sudsing control agents and substantive fluorescent dyes which control the apparent whiteness of fabrics washed with the composition. When such modifications are practised, the highly coloured granules or particles become an active component of the composition; and the process and apparatus furnish a convenient way of controlling and varying the percentage of important minor-quantity ingredients in the composition.
- 25 **WHAT WE CLAIM IS:—**
1. A speckled granular detergent composition in which 2 to 30% by weight of the composition consists of coloured granules substantially uniformly distributed throughout the composition, the colouring of the granules, which are coloured by colouring matter not substantive to fabrics, being vivid so as to be sharply demarked from the background of the remaining granules, the said coloured granules consisting predominantly of granules whose large dimension is between 0.2 and 1.5 millimetres and the said coloured granules having a Munsell chroma of more than 4 and a Munsell value of 4 to 7.
 2. A detergent composition according to Claim 1 in which all the said high chroma coloured granules are of the same colour.
 3. A detergent composition according to Claim 1 or 2 in which the said high chroma coloured granules are of two or more different colours.
 4. A detergent composition according to any of preceding claims in which the granules (other than the said high chroma coloured granules) have the natural colour of a spray dried detergent to which no colouring matter has been added.
 5. A detergent composition according to Claims 1, 2 and 3 in which the granules other than the said high chroma coloured granules have a light or pastel colouration of a different hue low in chroma and high in value by the Munsell Scale.
 6. A detergent composition according to any of preceding claims in which the said high chroma coloured granules are coloured with Erioglaucine, Rhodamine B, Amaranth Red, Fast Crimson, Wool Violet, Azure Blue or a Lumogen.
 7. A granular detergent composition according to any of Claims 1 to 6 in which all the granules in the composition have roughly the same general characteristics as to size and density.
 8. Process for producing a granular detergent composition according to any of Claims 1 to 7 comprising forming a continuously flowing stream of granular detergent composition, diverting a fraction of said continuously flowing stream, applying colouring matter to the diverted fraction, and recombining and substantially uniformly mixing the coloured diverted fraction with the main stream.
 9. Process according to Claim 8 in which more than one fraction of the main stream is diverted and differently coloured and then the plurality of diverted fractions are recombined with the main stream.
 10. Process according to Claim 8 or 9 in which colouring matter is applied to a diverted fraction by spraying with colouring matter in a liquid vehicle.
 11. Process according to Claim 10 in which the spraying is effected with an aqueous solution or dispersion of a dyestuff or pigment.
 12. Process according to any of Claims 8 to 11 in which the coloured diverted fraction, or fractions, is mixed with the main stream in an air lift.
 13. Process of making a speckled granular detergent composition according to any of the Claims 1 to 7, which comprises subjecting detergent granules to an air lift and during the continuous movement of the said granules separating fines from them and subjecting them to a screening operation prior to final delivery, continuously separating from the granules as so treated, a portion thereof controlled as to quantity, and spraying the separated granules with an aqueous solution or dispersion of colouring material capable of imparting to the granules a colour having a Munsell chroma of more than 4 and a Munsell value of 4 to 7, and subjecting the so-coloured granules to the said air lift along with fresh granules passing therethrough, whereby to dry the so treated granules and air them thoroughly with the said fresh granules the quantity of so treated granules being 2% to 30% of the total quantity of the mixture.
 14. Apparatus for carrying out the process according to any of Claims 8 to 13 comprising an air lift adapted to receive detergent granules, said air lift having in connection therewith means for separating fines from said granules and screen means for separating over-sized particles therefrom, means for diverting a percentage of the granules passing through said screen, means for spraying the diverted granules with colouring material in solution, and a means for returning the so treated granules to the said air lift, whereby to dry them and mix them thoroughly with the granules passing through said air lift.



in which granules of soap or detergent are mechanically mixed with finely divided builders, such for example as phosphates, it is preferred to manufacture the soap or detergent granules, divert and colour a

5 portion thereof, and mix the coloured granules with the uncoloured granules, all prior to admixture of the product with the pulverulent builders.
10 The treatment of the granules in the drum 18 is not limited to a treatment with colouring substances. Other treatment substances may be used in addition, including, but without limitation, anti-caking agents, sudsing
15 control agents and substantive fluorescent dyes which control the apparent whiteness of fabrics washed with the composition. When such modifications are practised, the highly coloured granules or particles become an
20 active component of the composition; and the process and apparatus furnish a convenient way of controlling and varying the percentage of important minor-quantity ingredients in the composition.

25 WHAT WE CLAIM IS:—

1. A speckled granular detergent composition in which 2 to 30% by weight of the composition consists of coloured granules substantially uniformly distributed throughout
30 the composition, the colouring of the granules, which are coloured by colouring matter not substantive to fabrics, being vivid so as to be sharply demarked from the background of the remaining granules, the said
35 coloured granules consisting predominantly of granules whose large dimension is between 0.2 and 1.5 millimetres and the said coloured granules having a Munsell chroma of more than 4 and a Munsell value of 4 to 7.

40 2. A detergent composition according to Claim 1 in which all the said high chroma coloured granules are of the same colour.

3. A detergent composition according to Claim 1 or 2 in which the said high chroma
45 coloured granules are of two or more different colours.

4. A detergent composition according to any of preceding claims in which the granules (other than the said high chroma
50 coloured granules) have the natural colour of a spray dried detergent to which no colouring matter has been added.

5. A detergent composition according to Claims 1, 2 and 3 in which the granules
55 other than the said high chroma coloured granules have a light or pastel colouration of a different hue low in chroma and high in value by the Munsell Scale.

6. A detergent composition according to any of preceding claims in which the said
60 high chroma coloured granules are coloured with Erioglaucine, Rhodamine B, Amaranth Red, Fast Crimson, Wool Violet, Azure Blue or a Lumogen.

65 7. A granular detergent composition

according to any of Claims 1 to 6 in which all the granules in the composition have roughly the same general characteristics as to size and density.

8. Process for producing a granular
70 detergent composition according to any of Claims 1 to 7 comprising forming a continuously flowing stream of granular detergent composition, diverting a fraction of said continuously flowing stream, applying colouring
75 matter to the diverted fraction, and recombining and substantially uniformly mixing the coloured diverted fraction with the main stream.

9. Process according to Claim 8 in which
80 more than one fraction of the main stream is diverted and differently coloured and then the plurality of diverted fractions are recombined with the main stream.

10. Process according to Claim 8 or 9 in
85 which colouring matter is applied to a diverted fraction by spraying with colouring matter in a liquid vehicle.

11. Process according to Claim 10 in which
90 the spraying is effected with an aqueous solution or dispersion of a dyestuff or pigment.

12. Process according to any of Claims 8 to 11 in which the coloured diverted fraction, or fractions, is mixed with the main stream
95 in an air lift.

13. Process of making a speckled granular detergent composition according to any of the Claims 1 to 7, which comprises subjecting
100 detergent granules to an air lift and during the continuous movement of the said granules separating fines from them and subjecting them to a screening operation prior to final delivery, continuously separating from the granules as so treated, a portion thereof controlled as to quantity, and spraying the
105 separated granules with an aqueous solution or dispersion of colouring material capable of imparting to the granules a colour having a Munsell chroma of more than 4 and a Munsell value of 4 to 7, and subjecting the so-coloured
110 granules to the said air lift along with fresh granules passing therethrough, whereby to dry the so treated granules and air them thoroughly with the said fresh granules the quantity of so treated granules being 2% to
115 30% of the total quantity of the mixture.

14. Apparatus for carrying out the process according to any of Claims 8 to 13 comprising an air lift adapted to receive detergent granules, said air lift having in connection
120 therewith means for separating fines from said granules and screen means for separating over-sized particles therefrom, means for diverting a percentage of the granules passing through said screen, means for spraying the diverted granules with colouring material in
125 solution, and a means for returning the so treated granules to the said air lift, whereby to dry them and mix them thoroughly with the granules passing through said air lift. 130

15. Apparatus for carrying out the process as described in any of the foregoing Examples according to any of Claims 8 to 13 constructed substantially as described with reference to the accompanying drawing.
- 5 16. Process for producing a speckled granular detergent composition substantially
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