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(54) Procedure for colouring non-adsorbent minerals and the product thus obtained

(57) Procedure for colouring granulated non-adsorbent minerals and the product thus obtained, of the type used in the field of chemical products and processes leading to coloured specks being obtained which are used in the detergent industry, characterised in that it allows coloured non-adsorbent minerals of a diameter between 300 and 2,000 μm to be obtained from a granular base of that same size, a binding agent and a colouring agent or pigment, used in the detergent industry as "speckles", an English term which defines the multicoloured specks present in powdered detergent.

The invention presented contributes the main advantage of allowing coloured, non-adsorbent minerals to be used with a reproducible method in stable production, all at a significantly lower economic and environmental cost.

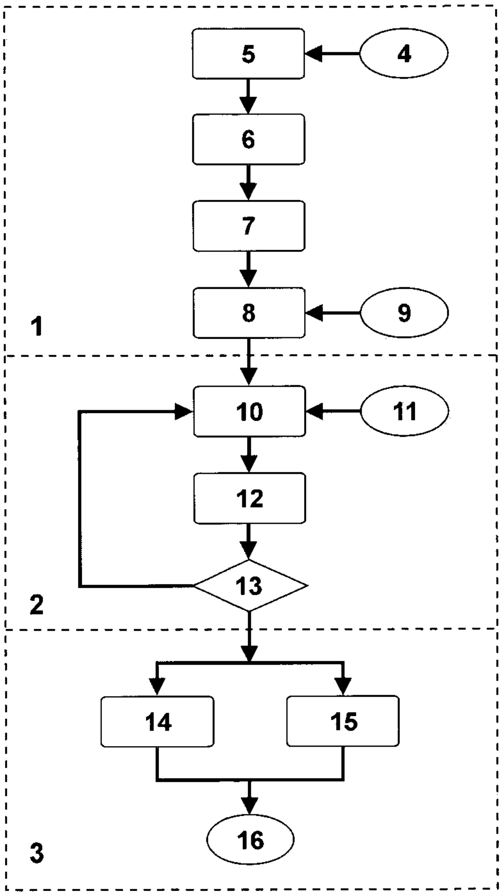

Fig. 1

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Description

[0001] This descriptive report refers, as its title indicates, to a procedure for colouring granulated non-adsorbent minerals and the product thus obtained of the type used in the field of chemical products and processes leading to coloured speckles used in the detergent industry being obtained, characterised in that it allows coloured granules, also known by the English tem "speckles", to be obtained which are particularly used as coloured speckles present in powdered detergents. These coloured granules are obtained from non-adsorbent mineral particles with a diameter of between 300 and 2,000 μm using a granular base of the same size, a binding agent and a colouring agent or pigment, all by means of a characteristic production procedure.

[0002] Many varied types of chemical products and processes leading to colour speckles, used in the powder detergent industry, being obtained are widely known and used on an industrial basis at present. These coloured speckles have been produced since the 70's when the first known references to the state of the art are found, such as, for example, US Patents 3850833, US 4097418, US 4162228, through the introduction of pigment and-or colouring agents into a preferably hygroscopic inorganic salt. In the same way the addition of a binding agent, which allows the colouring agent to be fixed to the non-adsorbent salt, as we can find in Patents US 3850833, EP0060728, and EP1305387, is considered. The introduction of coloured speckles in detergent continues to be a fact to the present day, as is shown by Patent EP1627909, and so the realisation of this invention was put forward.

[0003] One of the most relevant characteristics required from these coloured speckles is their visibility in the detergent bed, so that the average size of a grain has to be between 300 and 2000 μm. As most of the processes to obtain inorganic salts referred to above have the disadvantage of not directly generating this size of grain, it is necessary use particle agglomeration processes, mainly by means of those known as spray driers or high energy mixers, as may be found in Patents US 4162228, US 5605883 and US 4671886. In these processes pigment or a colouring agent and a binding agent is added to the inorganic salt which allows this colouring agent to be fixed to the inorganic salt from giving consistency and hardness to the granule formed. It may be done in a dry process (adding a small amount of water) or a wet process by flash evaporation. These processes have the big problem of being expensive both because of the high consumption of raw materials necessary and because of the high energy requirements involved, and have an enormous effect on the final cost of the product and on the economic profitability of the process.

[0004] The possibility of using other absorbed materials such as bentonite and zeolites as coloured speckles, is also known and used, as described in Patent US 5065883 and European Patent 935343 "*Granular detergent composition containing zeolite MAP"*. These are capable of fixing the colouring agent without the need for a binding agent. These cases, and because of the high specific surface of these materials, have the great problem of there being a large consumption of pigments, which apart from logically making the product expensive, can cause excessive pigment or colouring agent residue to be deposited on clothes when washed.

[0005] The additional use of some inorganic salts as binders, such as sodium sulphate, for example, mixed with surface active products in the manufacture of detergents in aqueous manufacturing processes, as contained, for example in European Patents 89109494 "*Granular adsorbent with improved supply behaviour",* 88303852 *"Procedure for preparing a granulated detergent composition",* 86307864 *"A procedure for preparing a detergent composition* " and 97904833 *"Process for obtaining a low density detergent composition by agglomeration with a double inorganic salt",* but have the disadvantage of a complex and economically very costly method having to be followed to colour these inorganic salts for use as coloured particles in a detergent, which makes profitable use thereof for the manufacture of detergent unviable.

[0006] In the same way, in the technical field, various types of colouring agents and pigments are known, such as those described in European Patents 93200451 "Silicate *pigment mixed with doped zirconium, a method for its preparation and products containing this pigment or a pigment prepared in this way"* or 95810553 "*Organic pigments of the stir-in type*", but they have the disadvantage of not being specifically applicable to colouring non-adsorbent mineral granules. There are also some colouring procedures known, such as the one described in European Patent 9902520 *"Procedure for obtaining coloured washing and cleaning agents"* which are also not specifically applicable to these products in an industrially reliable and economically viable way.

[0007] In order to solve the current difficulties regarding the problem of colouring non-adsorbent minerals for granular use in powder detergents the procedure of colouring granulated non-adsorbent minerals and a product thus obtained forming the object of this invention has been devised, whereby a non-adsorbent mineral, preferably an inorganic salt or substrate is combined with a colouring agent and a binding agent, following a characteristic sequence of phases.

[0008] The process is based on a non-adsorbent salt, preferably a monocrystal inorganic salt with a grain size of between 300 and 2,000 μm, preferably between 600 and 1,500 μm, of a hygroscopic nature in order to be able to fix the colouring agent, so these are mainly inorganic salts, such as sodium carbonate, sodium tripolyphosphate, sodium chloride, sodium sulphate among others.

[0009] The colouring agent is responsible for providing the desired colour to the inorganic salt and any pigment or colouring agent allowed in the detergent industry or any preparation thereof, which must have a granulometry between

0.01 and 15 μm, preferably between 0.01 and and 5 μm] is used. As an example of some of the commonest, we may cite: Pigment Blue 15, Pigment Green 7, Pigment Yellow 3, Pigment Red 57, Yellow Food Colouring 3, Food Blue 5, Acid Green 1, Acid Yellow 4, among others.

[0010] The binding agent is necessary to obtain a film, which fixes the colouring agent to the inorganic salt substrate, as it is a non-adsorbent material. This acts by generating a surface layer which fixes the colouring agent to the substrate and which is compatible with formulations in the detergent. As an example of binding agents which may be used and which are found in literature, we may cite among others, sodium silicate, different types of polymers, carboxymethyl cellulose (CMC), etc

[0011] The colouring procedure comprises a sequence of three main phases, which in their turn are divided into different intermediate steps.

- **Phase 1** - Preparation of loading of inorganic salt and addition of colouring agent, heating the product above its hydration temperature to then introduce the pigment or colouring agent in the form of a preparation.

- **Phase 2 -** Addition of binding agent and subsequent drying, carried out in various subsequent steps in order to facilitate the homogeneity thereof and avoid incrustation

- **Phase 3 -** Final cooling of the product

[0012] This colouring procedure is carried out in a industrial rotary mixer of the endless screw or mixing blade variety, with a jacket or encasing double wall equipped with a peripheral water heating circuit and also equipped with a hot air injection system inside. The appropriate temperature control and regulatory devices must be available both for water circulating in the jacket and for the hot air, to allow precise control of the working temperatures to prevent the formation and agglomeration of hydrated compounds.

[0013] This colouring procedure for granulated non-adsorbent minerals and the product thus obtained which is presented provides many advantages over procedures available at present, the most important being one which allows the use of a different product, based on coloured inorganic salts, to the one actually used and an alternative method to the production of these speckles with significant technical, environmental and economic advantages over methods currently used and hence, product advantages.

[0014] The enormous technical advantages of this process must be stressed, as it is a method that can be reproduced on an industrial scale and ensures stable production. On the other hand, current processes are complex and have a high number of rejections that have to be reused, making the system even more complex.

[0015] We should not forget the enormous environmental advantages of this invention, as the lower costs in energy and raw materials are reflected in fewer contaminating gases into the atmosphere and lower fuel, electricity consumption, etc.

[0016] We should likewise stress the added advantage implied by the fact that the product obtained advantageously replaces sodium tripolysulphate, one of the commonest products used in this application and which is one of those responsible for the eutrophication of water in rivers and lakes and for the reduction of O_2 dissolved in water, thus contributing to the minimisation of the contaminating impact of detergents.

[0017] Finally, the undeniable economic advantages obtained by avoiding high consumption of energy and raw materials should be highlighted, as the cost is significantly lower than other compounds in the field and therefore allows much higher industrial profitability.

[0018] In order to better understand the purpose of this invention a practical, preferential embodiment of the procedure for colouring granulated, non-adsorbent minerals and the product thus obtained is given. In this drawing figure -1- shows a block diagram of the sequence of the colouring process.

[0019] Figure -2- gives a graphic example of the evolution of temperature inside the mixer.

[0020] Figure -3- shows a simplified view of the main elements, which the mixing device used in the invention must comprise.

[0021] As can be seen in the attached drawing, the procedure for colouring granulated, non-adsorbent minerals and the product thus obtained which forms the object of this invention, consists of a characteristic sequence of phases (1,2,3) in which a non-adsorbent mineral (4) or substrate is mixed with a colouring agent (9) and a binding agent (11).

[0022] The process is based on a non-adsorbent mineral (4), preferably a monocrystal inorganic salt with a grain size of between 300 and 2,000 μm, preferably between 600 and 1500 μm, so it is not necessary to carry out an agglomeration process for it to achieve the desired size and, given the monocrystalline nature of the product obtained, it is hard enough to withstand the effect of friction during mixing with the rest of the components. It should be of a hygroscopic nature so that the colouring agent (9) can be fixed (9), so that sodium carbonate, sodium tripolysulphate, sodium chloride, sodium sulphate among others may be mainly used.

[0023] The colouring agent (9) is responsible for providing the desired colour to the non-adsorbent mineral (4) and for this any pigment or colouring agent allowed in the detergent industry or any preparation thereof, which must have a granulometry of between 0.01 and 15 μm, preferably between 0.01 and 5 μm, is used, thus maintaining its visibility and facilitating the homogeneity of the colour in the subsequent process and avoiding stains in the product because of a larger size. As an example of the commonest ones that may be used in the process we may cite: Pigment Blue 15, Pigment Green 7, Pigment Yellow 3, Pigment Red 57, Food yellow 3, Food Blue 5, Acid Green 1, Acid Yellow 4, among others.

[0024] The binding agent (11) is necessary to obtain a film, which fixes the colouring agent (9) to the inorganic salt substrate. Moreover, being non-adsorbent materials, the pigment or colouring agent must be fixed to the grain. The binding agent (11) acts by generating a surface layer, which fixes the colouring agent (9) to the inorganic salt substrate (4), allowing it at the same time to be compatible with the formulations in the detergent. As an example of binding agents (11) which may be used in this process, we may cite among others, sodium silicate, different types of polymers, carboxymethyl cellulose (CMC), etc ., preferably soluble sodium silicate because of its readiness to form vitreous layers with the above features when its diluting water dries up.

[0025] The colouring procedure characteristic of the invention comprises a sequence of three main phases, which in their turn are divided into different intermediate steps.

- **Phase 1 (1) -** Preparation of the loading (5) of the inorganic salt (4) and addition of colouring agent (9), heating the product above its hydration temperature to then introduce the pigment or colouring agent (9) in the form of a preparation.

- **Phase 2 (2) -** Addition (10) of binding agent (11) and subsequent drying (12), carried out in various subsequent steps (13) in order to facilitate the homogeneity thereof and avoid incrustations.

- **Phase 3 (3) -** Final cooling of the product (16)

[0026] We now describe in detail the different phases and intermediate steps which the process comprises.

- Phase 1 (1)

[0027] The phase starts with the initial step of loading (5) the mixer (17) with the non-adsorbent mineral (4), where the step of heating the product (6) above its hydration temperature is carried out, if it has one. To do this, the jacket (18) is heated at the same time above this temperature and hot air is introduced into the mixer (17).

[0028] Once that temperature has been reached, the phase continues with the step of lowering the temperature (7) of the jacket (18) below the temperature of the product followed by the step of introducing (8) the colouring agent (9) in the form of a preparation.

[0029] It is important that the jacket (18) be below the temperature of the product in order to avoid incrustations. This may be achieved in two ways: by keeping it constant just above the hydration temperature or by maintaining a constant difference with the temperature of the product.

[0030] The colouring agent (9) will be added in suspension with water and, if necessary, with some kind of dispersant. The concentration of colouring agent (9) in the preparation will be preferably between 15 %, to avoid using a lot of water which will evaporate afterwards, and 60 %, to allow a good mix and close contact between the substrate (4) and the colouring agent (9), as the medium which puts them in contact and allows them to join is water, although they are fixed by the binding agent (11) in the subsequent drying process.

[0031] It should be carried out in a way that allows a good mix. Thus, the addition to the mass will be made with sufficient time for mixing, preferably between 10 seconds and 5 minutes, depending on the mixing capacity of the equipment.

[0032] The proportion of colouring agent (9) to be used in relation to the non-adsorbent mineral (4) varies between 0.001% and 10%, preferably between 0.01% and 1%.

[0033] The introduction of the colouring agent (9) should be carried out preferably by means of spray nozzles or a shower to make mixing easier.

- Phase 2 (2)

[0034] In this phase the step of adding (10) the binding agent (11) is carried out several times (13) to facilitate homogeneity and avoid incrustations. A drying step (12) is then carried out by introducing hot air into the mixer (17) to dry after each step of adding (10) a binding agent (11).

[0035] The method of introducing this hot air, its rate of flow and temperature will result in less time and, therefore, greater production capacity, resulting in fewer operational costs. The temperature of the drying air must be preferably between 100 and 300° C, depending on the physical characteristics of the substrate (4), colouring agents (9) and binding agent (11) chosen.

[0036] The rate of flow of hot air to be introduced should be such that it is sufficient to dry and keep the bed spongy if necessary, but avoiding elutriates (solid product carried away by the air) being produced. As high granulometry is involved, excessive amounts of elutriates are not generated and this solved by installing a sleeve filter or an air washer at the outlet for the exhaust air.

[0037] In the temperature curve in the example given in Figure 2, the number of times (13) that the cycle of adding (10) and subsequent drying (12) steps is 5 by way of example, thus optimising the process in accordance with the type of mixer and product.

[0038] In this temperature curve in Figure 2 we can observe how in each step of adding (10) the binding agent (11) a drop in temperature occurs when the coloured substrate (4) mixes with the binding agent (11) at a lower temperature. As the water of the binding agent (11) goes on evaporating an increase in temperature occurs. Once the binding agent (11) has dried, it is time to carry out the second addition (10). The time for making that next addition (10) depends on the equipment used, but always coincides in all cases with a change of slope, i.e. the water does not continue evaporating, and so the temperature of the solid matter goes up much more abruptly. Thus, the temperatures on addition (10) depend on the equipment, the number (13) of additions intended, the substrate (4) load, the temperature of the cooling jacket (18) and the characteristics of the drying air. The increase in temperatures between additions (10) should preferably be between 2 and 10° C. In this way a dry product will be obtained, preferably with humidity of less than 5% in weight.

[0039] The method of adding (10) the binding agent (11) should enhance uniform distribution throughout the product without generating any kind of agglomerate (either of product or dry residue of the binding agent itself) and avoid giving rise to incrustations. Thus, adding is carried out over various steps emulating an on-going addition (10) and drying (12) process. That is to say, an addition (10) is made and we wait for this product to be dry enough for the next addition to be made.

[0040] The number of additions (13) simulates an on-going process of addition and depends on the percentage of binding agent (11) necessary for each size of grain and substrate (4).

[0041] The percentage addition of binding agent (11) may vary between 2 % and 10 %, the number of additions to fulfil the conditions of the first heading being a total of between 1 and infinity (exactly what an on-going addition process would be). Preferably, a suitable number of additions is between 2 and 10. Below this amount there is the risk of generating incrustations and agglomerations and above, the process is affected energy wise.

[0042] Each one of the different additions (10) should be made in a way that allows a good mix. Thus, the addition to the mass will be made with sufficient mixing time, preferably between 10 seconds and 5 minutes, depending on the mixing capacity of the equipment.

[0043] Introduction of the binding agent (11) should be by means of spray nozzles or a shower to make mixing easier.

[0044] In an alternative planned embodiment the binding agent (11) will be added continuously throughout the drying process (12).

- Phase 3 (3)

[0045] After the final temperature has been reached, a cooling process of the end product (16) must be carried out as otherwise the hygroscopic nature of the inorganic salts (4) being treated would cause condensation effects during storage. A suitable temperature for finishing the process and considering the product as finished (and cold) would be around 30-60° C.

[0046] The cooling process is carried out by means of a step introducing cold air (14) and another simultaneous step introducing cold water (15) into the jacket (18). Cold is considered to be at a temperature around room temperature.

[0047] In each of the phases of the procedure (1,2,3) the rotating speed of the mixing element (17) may be varied by controlling the rotating motor (20) in order to improve each one. These revolutions should not be excessive in order to avoid stirring the granules too violently causing them to break and fine materials to be produced. As a general rule, but not restrictive in scope, the following preferred values for each one of the above stages in a horizontal mixer (18) may be given:

- Phase 1 (1) - High revolutions to improve the mix.
- Phase 2 (2) - Addition (10) of binding agent (11): High revolutions to improve the mix.
- Phase 2 (2) - Drying steps (12): Optimising between high and low revolutions to avoid the granules breaking up and to achieve a good mixing effect between the dry air and the solid matter.
- Phase 3 (3) - Low revolutions to avoid the granules breaking up and to assist the cooling process.

[0048] Thus, revolutions during the process will fluctuate between 1 and 150 rpm in the mixing element, depending on the type of product, equipment and process. The choice of the type of revolutions will directly influence the process time and, therefore the production capacity however, this will not have any great effect on the quality of the product.

[0049] The mixer (17), which may be used for this invention, may be any of those used in industry, preferably the horizontal type with flat blades and having the following components:

- Outside jacket (17) or double insulated wall.
- Internal mixing chamber (19) with mixing blades (34) linked to an external drive mechanism (20).
- Exhaust gas outlet (21). This flow may or must go to an elutriate collection system, of the sleeve filter or air washer variety. If the air flow rate and mixing power is properly chosen, the amount of entrained product should be minimal. Otherwise, the entrained product may be used again, for example by putting it back into the system or mixing it with the end product.
- Introduction (22) of colouring agent (9) and binding agent (11), preferably through spray nozzles.
- Introduction of substrate (23). Loading of each batch of inorganic salt is carried out here (4).
- Unloading (24) end product (16)
- Entry of water (25) to the jacket (18) of the mixer (17). With temperature control (28), which affects the hot water (26) and cold water (27) intakes.
- Entry of hot air (26)
- Entry of cold water (27)
- Exit of water (29) from the jacket (18) of the mixer (17).
- Entry of air (30) to the internal mixing chamber (19).
- Air heating device (32). With adjustable temperature control for airflow in order to achieve a suitable drying temperature. This may be by steam, direct heating by combustion, electrical heating elements, use of waste gases etc.
- Entry of cold air (31)
- Exit of air (33)

[0050] This equipment to be used must also meet the following requirements:

- Good mixing capacity so that all speckles have the same tone and the same amount of binding agent.
- Gentleness with the product to avoid the granules breaking up during the mixing process.
- Preventing the product from agglomerating.
- Drying capacity to obtain a product with preferably less than 5 % humidity.
- External cooling through a jacket

[0051] The colouring process forming the object of the invention should also meet a series of requirements:

The process may be continuous or discontinuous (batch), although a batch process is chosen to facilitate changes of colour in same equipment.

[0052] No hydrated compounds should be formed during the process in order to prevent their agglomeration, therefore an operating temperature has to be set for each product.

[0053] Hot spots on the walls of the equipment should be avoided in order to prevent the binding agent (11) drying onto the wall and causing incrustations. As the wall is going to be colder than the product to be dried, the volume/surface area (V/SA) ratio of the jacket (18) should be optimised in order to avoid loss of energy and incrustations of the product. This volume/surface area ratio should likewise optimise the contact surface of drying air with the product and therefore increase the production capacity for a given volume. This ratio will be different depending on the type of mixer (17).

[0054] There are two ways of keeping the wall colder than the product. Either by maintaining a constant temperature in the jacket (18) or by keeping the jacket (18) slightly colder than the product (1-5 °C). In both cases, always above the hydration temperature in order to start adding the colouring agent (9) or binding agent (11). In the first case, a loss of energy occurs during drying and in the second more incrustations occur in the body of the equipment where the product bed is not located. This will depend on the type of mixer (17), the choice of one method of working or another.

[0055] The volume of product should be such that the formation of incrustations is prevented as, due to abrasion, they do not occur on the actual product bed. Thus, the occupied volume should be such as to allow mixing and minimise incrustations. In most equipment this percentage fluctuates between 50 and 100 % of the total volume of the equipment, which allows movement of the mixture.

[0056] It is important to point out that the method and order of adding the colouring agent (9) and the binding agent (11) does not matter, although it is preferred to add the colouring agent (9) first and then the binding agent (11).

[0057] Finally, this invention is illustrated by the following example, which in no way is restrictive in its scope.

[0058] A production batch of coloured speckles of different colours is made, based on granular sodium sulphate with a constant jacket temperature, with the following mixer and process characteristics:

Characteristics of the substrate.

Characteristic	Value
Compound	Granular sodium sulphate
Average size	900 μm
Apparent density	1.6 Kg/l
Initial humidity	0.03 %

Characteristics of the binding agent

Characteristic	Value
Compound	Soluble sodium silicate
Percentage of total addition to substrate (in N additions)	5 %

Characteristics of the different colouring agents

Characteristic	Value
Colouring agent$_1$ (Green)	Pigment Green 7 (35 %)
Colouring agent$_2$ (Blue)	Pigment Blue 15 (35 %)
Colouring agent$_3$ (Orange)	Food Yellow 3 (35 %)
Percentage addition of preparation	0.20 %

Characteristics of the mixer

Characteristic	Value
Type of mixer	Horizontal with flat blades
Volume of mixer	3,000 litres
V/SA ratio	0.26 m^3/m^2

Characteristics of process

Characteristic	Value
Substrate load	3,000 Kg. (70 %)
rpm addition of colouring agent	80
rpm addition of binding agent	80
rpm drying	60
rpm cooling	30
Number of additions of binding agent (N)	*4*
Temperatures of addition of binding agent (L)	L_1 = 45° C / L_2 = 51° C L_3 = 57° C / L_4 = 65° C
Unloading temperature	50° C
Temperature of drying air	190° C
Flow rate of drying air	3500 m^3/h (190° C)
Temperature of jacket during drying	Constant at 35° C
Flow rate of water to jacket	30 m^3/h
Total process time	64 minutes

[0059] This same example, carried out in the same way, but maintaining the difference between the jacket temperature and the product temperature constant, gives a process time of 58 minutes, although it generates more incrustations on the equipment.

[0060] The results of the products obtained (three colours) are given below. It should be pointed out that the CIE L*a*b* scale is used to measure the colour and illuminant C is measured with a commercial colorimeter.

Results obtained for the different colours in the example

Green sodium sulphate		Blue sodium sulphate		Orange sodium sulphate	
*L**	50	*L**	40	*L**	45
*a**	-25	*a**	5	*a**	53
*b**	-8	*b**	-34	*b**	40
Humidity	0.11 %	*Humidity*	0.12 %	*Humidity*	0.10 %

[0061] The sulphate thus obtained contains 97.4% pure sodium sulphate, higher than the methods currently used, since, as stated, the consumption of raw materials is less. The remaining properties (granulometry, density, etc.) remain very similar to the starting product as practically no grains have been broken.

[0062] It can also be observed from the table of results that by setting the process characteristics a totally reproducible method is achieved, resulting in a product with constant characteristics.

Claims

1. Procedure for colouring granulated non-adsorbent minerals **characterised in that** it consists of the appropriate combination of a non-adsorbent mineral (4) or substrate, with a colouring agent (9) and a binding agent (11) .

2. Procedure for colouring granulated non-adsorbent minerals in accordance with claim 1, **characterised in that** it consists of the appropriate combination of a non-adsorbent mineral (4) or substrate, with a colouring agent (9) and a binding agent (11), comprising the following stages:

    a) - Phase 1 (1). Loading of non-adsorbent mineral (4) into a mixer (17) and adding of a colouring agent (9).
    b) - Phase 2 (2). Drying process and addition of binding agent (11).

c) - Phase 3 (3). Product cooling process.

3. Procedure for colouring granulated non-adsorbent minerals in accordance with claim 2, wherein Phase 1 (1) comprises the following steps:

a) - Loading (5) of non-adsorbent mineral (4) into mixer (17),
b) - heating of product (6) above hydration temperature,
c) - lowering of temperature (7) of jacket (18) of mixer (17) below temperature of product,
d) - introduction (8) of colouring agent (9) in the form of a preparation.

4. Procedure for colouring granulated non-adsorbent minerals in accordance with claim 3 , wherein the step of heating the product (6) above its hydration temperature is carried out simultaneously by heating the jacket (18) above that temperature and by introducing hot air into the mixer (17).

5. Procedure for colouring granulated non-adsorbent minerals in accordance with claim 3, wherein the colouring (9) is added in suspension with water and, if necessary, with some kind of dispersant, the concentration of colouring agent (9) in the preparation preferably being between 15 % and 60 % with granulometry of between 0.01 and 5 μm.

6. Procedure for colouring granulated non-adsorbent minerals in accordance with claim 2, wherein the proportion in weight of colouring agent (9) in relation to the non-adsorbent mineral (4) is between 0.001 % and 10 %.

7. Procedure for colouring granulated non-adsorbent minerals in accordance with claim 6, wherein the proportion in weight of colouring agent (9) in relation to the non-adsorbent mineral (4) is between 0.01 % and 1 %.

8. Procedure for colouring granulated non-adsorbent minerals in accordance with claim 2, wherein Phase 2 (2) comprises the following steps:

a) - addition (10) of binding agent (11),
b) - drying (12) by introducing hot air into the mixer (17),

this addition cycle (10) followed by drying (12) being carried out several times (13) to facilitate homogeneity and avoid incrustations.

9. Procedure for colouring granulated non-adsorbent minerals in accordance with claim 2, wherein the percentage of binding agent (11) added is between 2% and 10% in respect of the non-adsorbent mineral.

10. Procedure for colouring granulated non-adsorbent minerals in accordance with claim 8, wherein the number of times (13) that the addition cycle (10) followed by drying (12) is carried out in Phase 2 (2) is between 2 and 10.

11. Procedure for colouring granulated non-adsorbent minerals in accordance with claim 8, wherein drying (12) is carried out with air at a temperature between 100° C and 300° C.

12. Procedure for colouring granulated non-adsorbent minerals in accordance with claim 8, wherein each addition (10) of binding agent (11) must be separated from the next by an increase in the temperature of the product of between 2 and 10° C in order to ensure correct drying and suitable final humidity.

13. Procedure for colouring granulated non-adsorbent minerals in accordance with claim 2, wherein the binding agent (11) used is soluble sodium silicate.

14. Procedure for colouring granulated non-adsorbent minerals in accordance with claim 13, wherein the amount of sodium silicate added is between 2 and 10 % in weight in respect of the non-adsorbent mineral (4).

15. Procedure for colouring granulated non-adsorbent minerals in accordance with claim 13, wherein the method of adding the sodium silicate is continuous throughout the drying process (12).

16. Procedure for colouring granulated non-adsorbent minerals in accordance with claim 2, wherein Phase 3 (3) comprises the following steps:

a) - introduction of cold air (14) into the mixer (17) and
b) - introduction of cold water (15) into the jacket (18) to cool the end product (16)

17. Procedure for colouring granulated non-adsorbent minerals in accordance with claim 16, wherein the introduction of cold air (14) into the mixer (17) is carried out at a temperature between 10 and 60° C.

18. Procedure for colouring granulated non-adsorbent minerals in accordance with claim 16, wherein the introduction of cold water (15) into the jacket (18) is carried out at temperature of between 5 and 35°C.

19. Procedure for colouring granulated non-adsorbent minerals in accordance with claim 2, wherein the process may be carried out continuously or discontinuously, mixing by discontinuous batches being preferable.

20. Procedure for colouring granulated non-adsorbent minerals in accordance with claim 2, wherein the mixer (17) used has a drying capacity and an external jacket (18) to allow it to be cooled.

21. Procedure for colouring granulated non-adsorbent minerals in accordance with claim 20, wherein the mixer (17) used has a volume /surface area ratio of between 0.15 and 0.40, depending on the production capacity required.

22. Procedure for colouring granulated non-adsorbent minerals in accordance with claim 21, wherein the mixer (17) used has a volume /surface area ratio of between 0.25 and 0.30.

23. Procedure for colouring granulated non-adsorbent minerals in accordance with claim 20, wherein the total volume occupied by the product inside the mixer (17) is between 50% and 100 %.

24. Procedure for colouring granulated non-adsorbent minerals in accordance with claim 23, wherein the total volume occupied by the product inside the mixer (17) is between 70% and 80 %.

25. Procedure for colouring granulated non-adsorbent minerals in accordance with claim 20, wherein the jacket (18) of the mixer (17) should be at a lower temperature than the product to prevent incrustations.

26. Procedure for colouring granulated non-adsorbent minerals in accordance with claim 25, wherein the temperature of the jacket (18) is maintained fixed.

27. Procedure for colouring granulated non-adsorbent minerals in accordance with claim 25, wherein the temperature of the jacket (18) is lower than that of the product at between° y 10° C.

28. Procedure for colouring granulated non-adsorbent minerals in accordance with claim 2, wherein each of the Phases (1, 2 3) is carried out with the mixture at different revolutions, with high revolutions being used in Phase 1 (1) to assist mixing, high revolutions in Phase 2 (2) during addition (12) of the binding agent (11) to assist mixing, medium revolutions in Phase 2 (2) during drying (12) to prevent granules breaking up and to assist the mixing of the product and the drying air, and low revolutions in Phase 3 (3) to assist the cooling process.

29. Coloured product obtained from non-adsorbent minerals (4) in accordance with the procedure described in any of the above claims.

30. Coloured product in accordance with claim 29, **characterised in that** it is based on a non-adsorbent mineral (4) with a granulometry of between 300 and 2,000 μm, a colouring agent (9) and a binding agent (11) as raw materials, obtaining an end product (16) with a final humidity of less than 5 %.

31. Coloured product in accordance with claim 30, wherein the non-adsorbent mineral (4) is preferably an inorganic salt.

32. Coloured product in accordance with claim 31, wherein the inorganic salt is sodium sulphate.

33. Coloured product in accordance with claim 32, wherein sodium sulphate is a monocrystal granular sodium sulphate.

34. Coloured product in accordance with claim 29, wherein the non-adsorbent mineral (4) has a granulometry of between 600 and 1,500 μm.

35. Use of the coloured product of claim 29 in the detergent industry as coloured speckles.

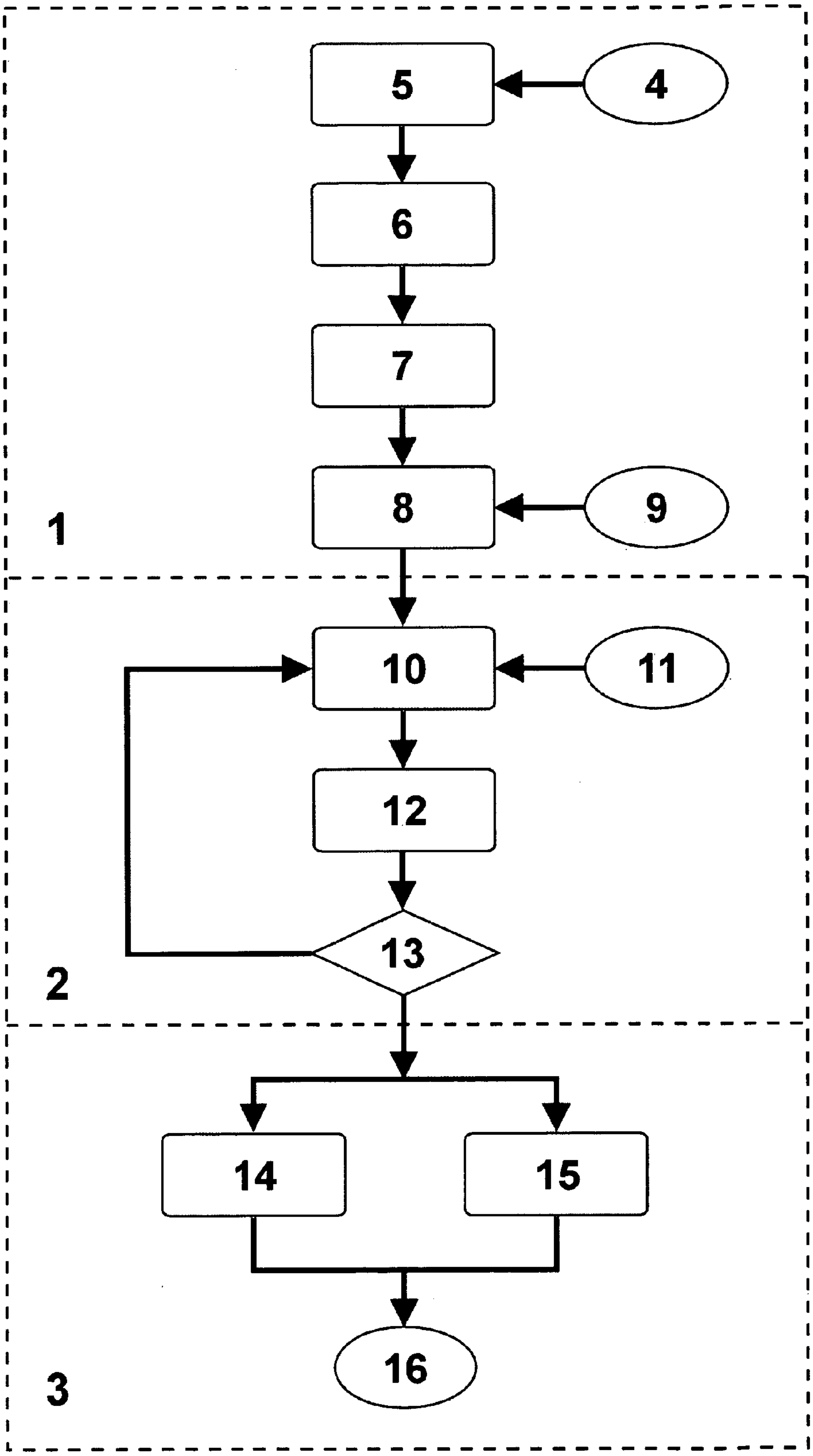

Fig. 1

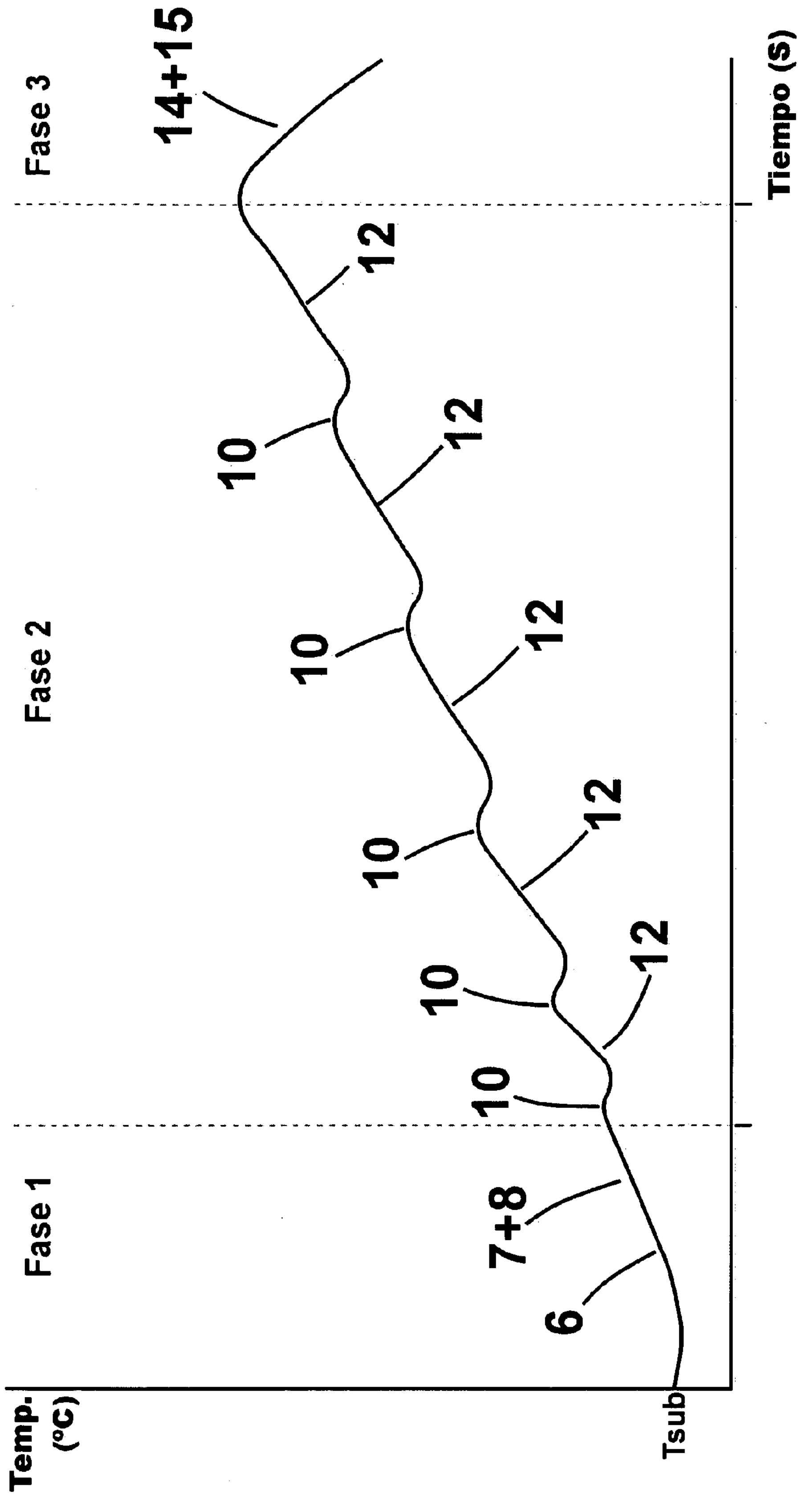
Temp.
(ºC)
Tsub
Fase 1
Fase 2
Fase 3
6
7+8
10
12
10
12
10
12
10
12
10
12
12
14+15
Tiempo (S)

Fig. 2

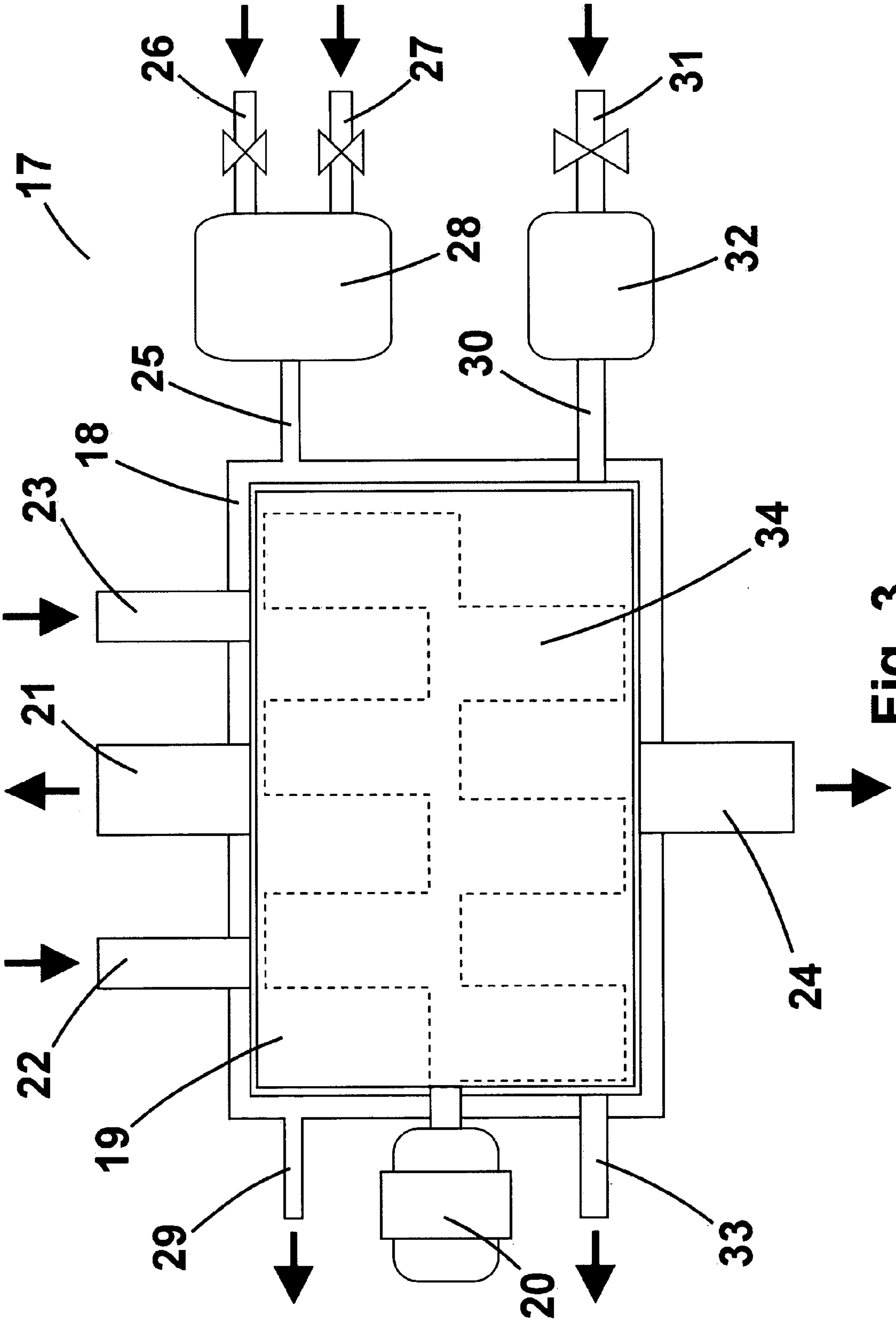

Fig. 3

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The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
The Hague	8 October 2007	Bertran Nadal, Josep

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