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(57) Abstract

Process for the preparation of detergent granules which process comprises as a first step agglomeration of a powdered detergent component by agitating the component and optional additives and contacting the agitated powder with a binder solution and as a second step drying the resultant agglomerate to form granules wherein the first step is carried out at a temperature below 40 °C and the second step is carried out at a temperature between 30 °C and 60 °C and at a relative humidity of less than 50 %.

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GRANULATION PROCESS

The present invention relates to an improved process for granulation particularly the granulation of inorganic compounds such as light soda ash, with sodium silicate solutions, the granules so prepared and detergent compositions comprising such granules.

Many products such as instant coffee and washing-machine powder are granulated for the convenience of customers and users. Compared to powders, granules tend to have reduced dustiness, improved flow properties and less of a tendency for caking or separation from mixtures on standing or handling. Granules may also offer other benefits such as rapid dissolution in water and facilitate accurate measurement of the correct dose. Granules also provide a constancy of component ratios in complex mixtures.

Granules can be built up in a number of ways, and a large range of equipment for granulation is commercially available. In some cases all of the granule is composed of desired product while in others, inert carriers are used to facilitate granulation or to enhance granule properties, such as solubility. Base dry granules which are porous can be used to absorb liquids to yield free flowing products that appear dry.

Production of granulated material such as surface-active agents and detergents is well known in the art and, for example, German Patent Publication No. 3910569, and Japanese Patent Publications numbered 59157198 and 59018114 describe the production of granular detergent compositions.

Granules of the prior art are often produced from components supplied as a fine dusty powder and this may lead to production of granules which suffer the disadvantage of being accompanied by dust. The presence of dust may make the granules messy to handle, difficult

accurately to meter and handle, contact with the dust may cause respiratory tract irritation in some people.

Several patents describe methods for preparing a granular detergent material from a dusty detergent component or powder. US patents 3,761,549 and 3,588,950 describe a process of making non-dusty detergent granules by means of gas fluidisation in a rotating drum. According to US Patent 2,730,507, non-dusting soap granules may be formed by mixing soap dust with fatty acid and an alkaline material which saponifies the acid.

It has now been found that granules which are accompanied by a reduced amount of dust can be produced by an improved granulation process. The current invention provides a process for the preparation of granules which process comprises as a first step, agglomeration of a powdered component by agitating said component and optional additives and contacting them with a binder solution and as a second step drying the resultant agglomerate to form granules wherein said first step is carried out at a temperature below 40°C and said second step is carried out at a temperature between 30°C and 60°C and at a relative humidity of less than 50%.

In a preferred aspect the invention provides a process for the preparation of a detergent granules which process comprises as a first step agglomeration of a powdered detergent component by agitating said component and optional additives and contacting the agitated powder with a binder solution and as a second step drying the resultant agglomerate to form granules wherein said first step is carried out at a temperature below 40°C and said second step is carried out at a temperature between 30°C and 60°C and at a relative humidity of less than 50%.

In further embodiments the invention also provides granules prepared according to the process of the invention.

Where used herein the term "powdered component" comprises one or more particulate inorganic compounds of which at least 25% of the particles are smaller than 500 microns. Examples of inorganic compounds include, but are not limited to, sodium carbonate, sodium sulphate, zeolites, sodium tripolyphosphate, surfactants, alkali earth metal salts and alkali metal salts. Preferred powdered components for use in the current invention include sodium carbonate and particularly sodium carbonate in the form referred to as "light soda ash".

In a preferred embodiment of the process of the current invention the powdered component to be granulated is a dry powder comprising at least forty percent by weight of the anhydrous form of an inorganic compound which can take up water of crystallisation. In a particularly preferred embodiment the total powdered component contains essentially no free water and less than five percent of all forms of water. This water may be present in free form and/or as water of crystallisation, and/or held as "zeolitic" water. It is also particularly preferred that the powdered component is in a finely divided, free-flowing, lump free form. The properties of the granulated product formed by the process of the present invention may be adversely affected if "wet" product is used.

Furthermore, it is particularly preferred that the powdered component does not comprise any particles which are larger than the final desired granule size. A powdered component containing even small numbers of particles of size equal to or larger than the final desired granule size is undesirable as it may minimise many of the desirable benefits of the invention.

Granulation processes usually produce granules of size which is not uniform but is distributed around a target median size. Granules formed by the process of the present invention are typically of median size from 0.5 to 3 mm.

Where used herein the term "particle size" means the preferred median size as determined using standard ASTM sieve measurement techniques.

Where used herein the term "binder solution" refers to any substance capable of bonding the dusty powdered component to form and maintain agglomerates thereof and to enable them to coat the agglomerating particles. Optimally the binder solution is compatible with the powdered component plus other additives and it also possess a "setting property" either alone or by interaction with the powdered component. It is especially preferable that the binder solution comprises aqueous solutions of sugars, polyacrylate polymers or neutral sodium silicate (waterglass) or water. Small amounts of surfactant material may be added to these binder solutions in order to improve their wetting properties.

Generally, binder solutions are used at aqueous concentrations at which the liquid is viscous and "sticky". Typically, waterglasses are solutions of from about 25% to about 50% by weight, typically around 34% by weight. Properties of the granulated product may be adversely affected if too viscous a binder solution is used. This solution may be diluted if necessary to give a solution of viscosity which is preferably less than or equal to 250 cP at 20°C.

In a preferred embodiment, sodium silicate solution is the binder solution. In a particularly preferred embodiment of the current invention, the binder solution used comprises a sodium silicate solution having a silicate to alkali weight ratio (as SiO_2/Na_2O) between 2.0 and 3.5. Other liquids or water soluble components can be added to this solution provided that they do not increase the viscosity of the liquid, or cause chemical changes to the sodium silicate, such a precipitation of silica. Examples of unacceptable additives include un-neutralised acids, salts containing free fluoride ion and, gelling agents.

In a preferred embodiment of the present invention a sodium silicate solution having a low viscosity is used as binder solution. Without wishing to be bound by theory, it is believed that low viscosity sodium silicate binder solutions allow the silicate to more effectively penetrate the powders. If high viscosity sodium silicate binder solution is utilised, it is believed that less silicate is incorporated into the powders and granule properties such as solubility may be diminished.

Where used herein the term "additives" includes components such as granulising particles and dyes added to the powdered component.

Where used herein the term granulising particles means particles which are capable of promoting granule formulation. Such components are extensively described in Australian Patent No.615239 (26674/88) and include neutral and alkaline salts of alkali metal cations and organic or inorganic anions such as alkali metal orthophosphates and tripolyphosphates.

The aforementioned list of ingredients for use in the process of the current invention is not exhaustive and many other alternatives will be apparent to the worker skilled in the art.

In the first step of the two step granulation process of the present invention, the powder is granulated by contacting it with the binder solution, in the second step the granules formed are dried under controlled conditions. The process of the current invention may include other steps which are commonly practiced in granulation processes such as separation of undersized product and recycling of this product to the granulation process.

In a preferred embodiment of the first (or granulation) step of the process of the present invention, the binder solution is broken up into fine droplets before contacting

the powder. The binder solution can be broken up into fine droplets by any suitable method. Nozzles are commonly used in the art for reducing solutions into sprays but many other methods for forming fine droplets will be apparent to those skilled in the art.

The fine droplets of binder solution contact the powder whilst it is being agitated. Preferably the agitation is such that contact of the spray with already formed granules is minimised. Agitation of the powder can be carried out by any suitable method but pan granulators are particularly preferred.

The first step of the present invention is preferably carried out at a stable temperature of less than 40°C and preferably less than 30°C. Use of higher temperatures may reduce the amount of silicate absorbed and may reduce the solubility of the product in water.

In the second (or drying) step of the two step process of the present invention the granules are dried at low temperature and low humidity, preferably at a temperature of less than 50°C. Drying can be carried out by any suitable method but use of a vacuum dryer or fluidised bed dryer has been found to be particularly suitable. If a fluidised bed drier is used, suitably inlet air temperatures are adjusted to give a bed temperature of a maximum of 50°C (and preferably 45°C). In practice inlet air temperatures of a maximum of 80°C, and preferably 70°C, have been found to be suitable. It is particularly preferred that drying conditions are controlled so that water evaporated from some of the drying granules does not condense other granules as this may cause caking and loss of desirable granule properties.

While it is important for the product to be relatively dry to reduce caking, it is also important that all water which is not crystallisation or chemically bound water is removed. The rate of dissolution in water of the product

granules may significantly reduced if the granules are over dried.

Preferred embodiments of the present invention will now be further described by way of the following examples.

Example 1

Laboratory Bench Scale Agglomeration of Light Soda Ash

Penrice Light Soda Ash (obtained from Penrice Soda Ltd) powder was agglomerated with VITROSOL NA53 sodium silicate solution to which a compatible surfactant, ALKADET 15, was added at 0.1% to improve wetting and spraying behaviour (ALKADET is registered trade mark of ICI Australia Limited and VITROSOL is a registered trade mark of PQ Australia Proprietary Limited). The composition of the agglomerates are given in Tables 1(a) and 1(b) .

Table 1 (a)

Raw Materials	Composition (w/w%)		
	Binder Solution	Powder	Product
Penrice Light Soda Ash		50.00	54.00
VITROSOL NA53*	40.00		43.20
Water	9.95		2.75
ALKADET 15	0.05		0.05

* (VITROSOL NA53: 29.3% SiO_2, 14.7% Na_2O, 56.0% H_2O)

Table 1 (b)

Chemical Components	g/100g Powder	
	Binder Solution	Powder
Na_2CO_3		100.0
Na_2O	10.0	
SiO_2	19.9	
H_2O	55.0	
Surfactant	0.1	

Agglomeration was performed in a small pan granulator using a hand spray to apply the sodium silicate solution. The pan was loaded with 200g of powder and allowed to rotate so as to produce a falling curtain of powder. Sodium silicate solution 200g) warmed to 40°C was sprayed onto the falling curtain of powder at the 11 o'clock position until the particles reached desired size range.

The wet agglomerated powder was spread out on metal trays at 0.5 to 1.0 cm in depth and dried overnight in an oven at 50°C at a relative humidity of less than 50%. The properties of the granules are given in Table 2. Solubility was measured by the number of inversions at ten second intervals that are required to dissolve 5g of material in a 250 ml cylinder of tap water.

Table 2

Properties	Value
$\%H_2O$** (w/w%)	27
Bulk Density (gcm^{-3})	0.49
Tap Density (gcm^{-3})	0.54
Solubility (no. of inversion)	4

** (Water content determined by Karl-Fischer titration)

Comparative Example I

Laboratory Bench Scale Agglomeration of Light Soda Ash

Light soda ash agglomerated in the manner of Example 1 was dried over a 48 Hour period on trays at room temperature open to the atmosphere and at a relative humidity of greater than 50%. The resulting, granules were similar in appearance to that prepared in Example 1 but contained 16.0% water and were no longer fully soluble in water.

Comparison of the products of Example 1 and Comparative Example I indicates that drying of the agglomerate at a relative humidity greater than 50% leads to a product of inferior solubility compared to the product of Example 1.

Comparative Example II

Laboratory Bench Scale Agglomeration of light Soda Ash: High Humidity Drying

Light soda ash was agglomerated in the manner of Example 1 to produce 1 kg of wet material. The wet agglomerated powder was spread out on metal trays at 0.5 to 1.0 cm depth and dried overnight at 50°C. During the drying the humidity in the oven was raised to about 50% relative humidity which appeared to cause recrystallization of the light soda ash. The granules produced had the properties in Table 3. Comparison of Tables 1 and 3 indicate that high humidity drying of Comparative Example II produces a denser and much less soluble form of granules compared with the granules of Example 1.

Table 3

Properties	Value
$\%H_2O$** (w/w%)	21.6
Bulk Density (gcm^{-3})	0.5
Tap Density (gcm^{-3})	0.58
Solubility (no. of inversion)	>10
Surfactant Absorption (w/w%)	14.4

Example 2

Laboratory Bench Scale Agglomeration of Light Soda Ash & Zeolite A

The technique described in Example 1 for agglomeration was applied to powdered blends containing Penrice Light soda ash and dried Toyobuilder zeolite A. The composition of the powdered detergent components used are given in Tables 4(a) and (b). The technique was successful in forming granules and reducing the dusting character of zeolite A. The properties of the granules are given in Table 5.

Table 4 (a)

Raw Materials	Composition (w/w%)				
	Silicate Solution	Powder 1	Powder 2	Prod. 1	Prod. 2
Soda Ash		50	65	27.9	38.5
VITROSOL NA53	80			44.7	47.4
Water	19.9			0.55	6.75
ALKADET 15	0.1			0.05	0.05
TOYOBUILDER ZA		50	35	27.9	20.8

Table 4 (b)

Chemical Components	g/100 g Powder		
	Silicate Solution	Powder 1	Powder 2
Na_2CO_3		50	65
Na_2O	9.1		
SiO_2	18.05		
H_2O	50..0		
Zeolite A		50	35
Surfactant	0.1		

Table 5

Properties	Product 1	Product 2
$\%H_2O$ (w/w%)	24.40	19.8
Bulk Density (gcm^{-3})	0.54	0.42
Tap Density (gcm^{-3})	0.61	0.48
Solubility	N.A.	N.A.
Surfactant Absorption (w/w%)	32.00	22.1

Example 3

Pilot Scale Agglomeration of Light Soda Ash

Penrice Light Soda Ash powder was agglomerated with Vitrosol NA56 sodium silicate solution to which a compatible surfactant, Alkadet was added to improve wetting and spraying behaviour. The composition of the sodium silicate solution and the final solids composition of the granules are given in Table 6.

Agglomeration was performed in a closed vessel fitted internally with a spray arm for application of the sodium silicate solution. The closed vessel was loaded with 20 kg of light soda ash and sprayed with 15 kg of sodium silicate solution per batch. The sodium silicate solution was preheated to 40°C to lower the viscosity to the point where the spray nozzle functions efficiently. The spray was directed towards the back of the vessel where powder would most likely be exposed during the vessel's rotation. Cooling was applied to the exterior of the vessel in order to remove heat generated by the hydration of the light soda ash and from the sprayed sodium silicate solution.

The wet agglomerate was dried under vacuum that is at a relative humidity of close to zero in the closed vessel at a temperature of 50°C after an aging period of 1 to 2 hours.

The agglomerated powder required at least 4 hours drying under rotation before it was sufficiently dried. The properties of the granules formed are given in Table 7.

Table 6

Raw Materials	Composition (w/w%)		
	Silicate Sol.	Powder Detergent Component	Granules
Penrice Lt Soda Ash		100.0	56.2
VITROSOL NA56*	85.0		47.8
Water	14.9		4.05
ALKADET	0.1		0.05

Chemical Components	g/100 g Powder	
	Silicate Solution	Powdered Component g/100g Powder
Na_2CO_3		100
Na_2O	9.5	
SiO_2	21.4	
H_2O	47	
Surfactant	0.1	

* (VITROSOL NA56; 32.3% SiO_2, 14.3% Na_2O, 53.4% H_2O)

Table 7

Properties	Value
$\%H_2O$** (w/w%)	21.5
Bulk Density (gcm^{-3})	0.78
Tap Density (gcm^{-3})	0.85
Solubility (no. of inversions)	8

** (Determined by difference in weight after drying at 290°C for 12 hours).

Comparative Example III

Pilot Scale Agglomeration of Light Soda Ash: No Vessel Cooling

Light soda ash was agglomerated with sodium silicate solution in the manner described in Example without cooling applied to the vessel during agglomeration so that temperatures rose to greater than 40°C. The agglomeration of the light soda ash was unsatisfactory due to the high yields of oversize agglomerates and excessive adhesion of powder to the vessel walls. It is believed that the

warming of the powder above a temperature of 40°C during agglomeration resulted in the unsatisfactory product.

Example 4

Pilot Scale Agglomeration of Light Soda Ash with Fluidised Bed Drying

Light soda ash was agglomerated in the manner described in Example 3 and then dried using a laboratory bench model fluidised bed dryer with a capacity of 750g of wet agglomerate. Wet agglomerate was dried over varying intervals of time and at different temperatures. The properties of the dried agglomerates relevant to the effects of drying are given in Table 8 in which temperature °C refers to inlet air temperature of the fluidised bed drier as distinct from the bed temperature. The preferred conditions giving the shortest drying time and greatest removal of water without adverse effect on the solubility of the agglomerates are given in Example 4G.

Table 8

Example	Temp. °C	Time (min.)	Solubility no. of inversions	$\%H_2O$ (w/w%)
4A	40	15	17	23.4
4B	40	21	16	22.3
4C	50	15	16	21.7
4D	50	21	15	21.3
4E	60	15	15	21.5
4F	60	21	15	19.9
4G	70	15	15	22.0
CE 4H	70	21	23	17.7
CE 4I	80	15	>25	17.7
CE 4J	80	21	>25	16.2
Wet Agglom.			16	29.7

Comparative Example IV

Pilot Scale Agglomeration of Light Soda Ash: Overdrying in a Fluidised Bed Dryer

The Comparative Examples CE 4H to CE 4J demonstrate the effect of using excessive drying conditions. The dried agglomerate in each case did not readily dissolve in water and left residual flocs.

While the invention has been explained in relation to its preferred embodiments it is to be understood that various modifications thereof will become apparent to those skilled in the art upon reading the specification. Therefore, it is to be understood that the invention disclosed herein is intended to cover such modifications as fall within the scope of the appended claims.

The claims defining the invention are as follows:

1. A process for the preparation of granules which process comprises as a first step agglomeration of a powdered component by agitating said powdered component and optional additives and contacting the agitated powdered component with a binder solution and as a second step, drying the resultant agglomerate to form granules wherein said first step is carried out at a temperature below 40°C and said second step is carried out at a temperature between 30°C and 60°C and at a relative humidity of less than 50%.

2. A process according to Claim 1 wherein said binder solution comprises at least one of the group comprising aqueous solutions of sugars, polyacrylate polymers, sodium silicate or water.

3. A process according to Claim 1 or 2 wherein the powdered component is a detergent.

4. A process according to Claim 1 or 2 wherein said powdered component comprises at least 40% by weight of the anhydrous form of an inorganic compound which can take up water of crystallisation.

5. A process according to Claim 1 or 2 wherein said powdered component comprises no free water and less than 5% of all forms of water.

6. A process according to Claim 1 or 2 wherein said powdered component is sodium carbonate.

7. A process according to Claim 1 wherein said binder solution comprises an aqueous solution of from 25% to 50% by weight of sodium silicate.

8. A process according to Claim 1 wherein said binder solution has a viscosity of less than or equal to 250cp at 20°C.

9. A process according to Claim 1 wherein said binder solution comprises a sodium silicate solution having an SiO_2/Na_2O weight ratio between 2.0 and 3.5.

10. A process according to Claim 1 wherein said binder solution is broken up into fine droplets before contacting the powder as it is agitated by a pan granulator.

11. Granules prepared according to the process of any of the preceding claims.

12. Granules of median size from 0.5 to 3 mm which have been prepared according to the process of any of claims 1 to 10.

13. A process substantially as herein described with reference to the Examples.

14. Granules substantially as herein described with reference to the Examples.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/AU 93/00662

A. CLASSIFICATION OF SUBJECT MATTER
Int. Cl.[5] B01J 2/30, 2/14, C11D 17/06

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC B01J, C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base, and where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to Claim No.
A	AU,A, 54549/90 (THE PROCTER & GAMBLE COMPANY) 1 November 1990 (01.11.90) See page 2, lines 16-38	1-2
A	WO 92/18603 (THE PROCTER & GAMBLE cOMPANY) 29 October 1992 (29.10.92) See pages 19-20	1-12
A	AU,A, 33749/88 (UNILEVER PLC) 2 November 1989 (02.11.89) See example 1	1

(continued)

[X] Further documents are listed in the continuation of Box C.

[X] See patent family annex.

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C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	**Citation of document, with indication, where appropriate of the relevant passages**	**Relevant to Claim No.**
A	EP,A2, 374652 (HENKEL KOMMANDITGESELLSCHAFT AUF AKTIEN) 27 June 1990 (27.06.90)	1
A	WO 92/01036 (HENKEL KOMMANDITGESELLSCHAFT AUF AKTIEN) 23 January 1992 (23.01.92)	1

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent Document Cited in Search Report	Patent Family Member					
WO 9218603	EP	510746	AU	18785/92	CA	2108167
	CN	1067674	FI	934464	MX	9201722
	NO	933642	PT	100359		
AU 33749/89	BR	8902005	CA	1323277	DE	68907438
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	ZA	8903191				
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	CN	1046932	JP	3000800		
						END OF ANNEX