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(54) Bleach activator formulations.

(57) This invention relates to a method of improving the performance of water-stable bleach activators of the cyclic anhydride or lactone type by formulating the bleach activators in a finely ground form together with a binder, filler, a dispersing aid and optionally a surfactant into granules and using the granules so formed in a detergent formulation.

EP 0 482 806 A1

This invention relates to a method of improving the performance of water-stable bleach activators and to formulations containing such activators of improved performance.

Bleach activators are well known ingredients of detergent formulations. A widely used activator is tetraacetyl ethylene diamine (also known as TAED) which is described in GB-A-2832021. Bleach activators which have an anhydride structure have recently been used in detergent formulations. Such activators are claimed and described in our published EP-A-331300. These activators are primarily based on the isatoic anhydride (hereafter referred to as "IA" in the specification) type structure. The use of similar anhydrides is also claimed in the later filed EP-A-332050 (Henkel). Similarly activators which have a lactone structure have been claimed and described in our published EP-A-332294.

It is well known to use these activators in powder or granular form, especially when used in powder formulations of detergents. For instance, EP-A-332050 states that IA or its derivatives can be used for activation in pure form, or to improve storage stability it can be added as tablets, granules, or in finely divided coated form. This patent specification adds, however, that special significance is attached to the granulated form which is prepared by agglomeration of powder.

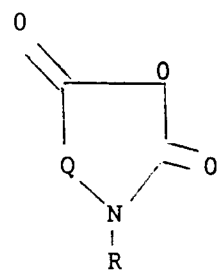
Granulation is a process for binding active material powders together into e.g. agglomerates. This technique usually mitigates problems of dust and materials handling and bestows other advantages such as improvement of flow; prevention of lump or cake formation; ease of metering, dosing and dispensing; enabling formation of uniform mixtures/blends without risk of segregation of the components in the mixture/blend; stabilisation of the active materials against premature degradation; controlling the release profile of the active materials; and enabling coatings to be applied on the active materials.

It has been found that the addition of these activators in conventional granular form can in some instances (e.g. with IA) cause a yellow-brown colouration of the wash liquor when in use. Whilst this discolouration does not always affect the washing performance of the detergent formulation, it is unsightly especially during hand wash. In extreme cases this can also cause brown staining of the clothes washed therewith.

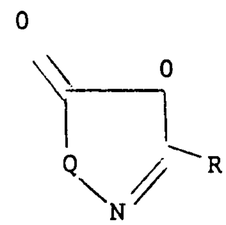
It has now been found that the aforementioned problems can be mitigated and performance improvements can be achieved by rigidly controlling the particle size of the activator within specified limits then granulating the same in an appropriate formulation.

Accordingly, the present invention relates to a granulated bleach activator formulation comprising:

a) a powdered, water-stable bleach activator in which at least 50% of the particles have a particle size below 250 micrometers and which bleach activator comprises at least one compound selected from a cyclic anhydride of the structural formula (I) or a lactone of the structural formula (II) below:



(I)



(II)

wherein Q is a divalent organic grouping such that Q and N together with the carbonyl and oxygen functions form one or more cyclic structures and in (I) R is H, an alkyl, aryl, halogen, a carboxylic or a carbonyl containing function, and in (II) R is a C_2 or higher alkyl, alkaryl, aryl, aralkyl, alkoxyl, haloalkyl, amino, alkylamino, dialkylamino, carboxylic or a carbonyl-containing function,

b) a clay binder and

c) a filler and/or a disintegrating aid,

said granules having a particle size mainly in the range of 200 to 2000 micrometers.

Bleach activators of formula (I) are claimed and described in our published EP-A-331300 and those of formula (II) are claimed and described in our published EP-A-332294.

According to a further embodiment, the present invention relates to a granulated bleach activator formulation comprising on a dry weight basis:

a) at least 60%w/w of a water-stable powdered bleach activator in which at least 50% of the particles have a particle size below 250 micrometers and comprising at least one compound of the structural formula (I) or (II) defined above,

b) from 5-20%w/w of a clay binder,

c) from 0-15%w/w of a disintegrating aid, and

d) from 0-20%w/w of a filler

such that the total amount of the disintegrating aid, the filler and binder present in the granules is at least 9%w/w, said granules having a particle size in the range of 200-2000 micrometers.

The amount of bleach activator in the granulated formulation is suitably at least 60%w/w, preferably at least 70%w/w.

The particle size of the powdered bleach activator used to produce the granules is below 250 micrometers, suitably below 150 micrometers, preferably from 1-110 micrometers. It has been found that some commercial varieties of activators sold and used in detergent formulations may have a particle size outside this range. In such a case, the activator has to be milled or ground to bring the particle size within the range now specified followed by air classification or sieving to produce a powder of the desired particle size range. Otherwise, the formulations of the invention do not function effectively since the use of larger particles for granulation in the case of e.g. IA cause yellow-brown colouration of the wash liquor and can in some cases cause staining of the clothes washed therewith. If, however, the activator is available in the appropriate particle size it can be used directly to produce the granulated formulations of the present invention.

Specific examples of activators of formulae (I) that can be used to produce the granulated formulations of the present invention include compounds in which Q represents an arylene group, and R is H or a lower alkyl, e.g. methyl such as isatoic anhydride and its derivatives, and those of formula (II) in which Q is again an arylene group and R represents a C_2 or higher alkyl group, an aryl group which may be unsubstituted or further substituted in the aryl nucleus or an alkylamino or a dialkylamino group such as the 2-dimethylamino and 2-aryl, e.g. 2-phenyl derivatives of 3,1-H benoxazin-4-one.

The granulated formulations of the present invention can optionally include conventional bleach activators such as tetralkyl ethylenediamine in addition to the compounds of formula (I) or (II) provided that the particle size of powder comprising such mixed activators used in the formulation to be granulated falls within the ranges now specified.

The binder used is a clay, suitably a bentonite clay, preferably an alkali metal bentonite clay or an alkaline earth metal bentonite clay. Most preferred are the sodium and calcium forms of bentonite clay. The amount of binder used in the formulation is suitably from 5-20%w/w, preferably 5-15%w/w of the dry granules.

The presence of a disintegrating aid in the granulated formulation, though not essential, facilitates the disintegration of the activator present in the formulation into the wash liquor. This is usually achieved by the ability of the disintegrating aid to swell in the presence of excessive amounts of water, as would be encountered during a wash cycle, and thereby help break up the granules to release the activator. In some cases, the disintegrating aid can also be used to partially or wholly replace the filler in the formulation. The amount of disintegrating aid used in the formulation is suitably from 0-15%w/w, preferably from 2-11%w/w on a dry weight basis of the total formulation. Examples of disintegrating aids that can be used include water swellable caboxymethyl cellulose derivatives such as the cross-linked microcrystalline derivative Ac-di-sol (Regd Trade Mark, ex FMC Corp) and polyacrylates.

The presence of a filler in the granulated formulation, though not essential, improves the binding properties of the clay binder. The filler is suitably present in an amount from 0-20%w/w, preferably from 1-12%w/w. Examples of fillers that can be used include various conventional inorganic fillers such as sodium sulphate or cellulosic fillers such as e.g. Avicel (Regd Trade Mark, ex FMC Corp).

The formulations of the present invention may contain in addition surfactants such as e.g. Tween 81 (Regd Trade Mark) which is a polyoxyethylene sorbitan monooleate ester or an alkali metal salt of a polycarboxylic acid, Dispex G40 (Regd. Trade Mark, ex Allied Colloids). Such surfactants, which can also aid disintegration, can be present in an amount from 0.01-5%w/w, preferably upto 3%w/w.

When 2-aryl 3,1 (4H)-benzoxazin-4-ones are used, it may be necessary to grind the activator to a very fine powder before being granulated. The particle size of the activator powder being granulated should be as small as is possible, preferably below 10 micrometers. Use of some surfactant e.g. at least 0.5% w/w, preferably at least 2% w/w of such surfactant appears beneficial in this case in order to achieve optimum results. The preferred surfactant used for 2-aryl 3,1 (4H)-benzoxazin-4-ones is preferably an alkali metal salt, e.g. sodium salt of a polymeric carboxylic acid, e.g. Dispex-G40 (Regd Trade Mark, sold by Allied Colloids, UK), in aqueous solution. The surfactant enables dispersion of the fine activator powder in the wash liquor.

In order to produce a granulated formulation of optimum handling characteristics the formulation suitably contains at least one, preferably both the dispersing aid and the filler. However, whichever combination is used, it is preferable that the total amount of the disintegrating aid, the filler and binder used is at least 9%w/w of the total granulated formulation on a dry weight basis.

The granulated formulations are suitably produced by an extrusion/spheronisation technique.

Extrusion/spheronisation technique is used to convert an active material in powdered form into regular sized, dense spheres. The purpose of this technique is usually to mitigate the problems of dust and materials handling, to allow complete and uniform mixing to be achieved with mixtures of powders without risk of segregation and to enable uniform coatings to be applied.

The technique consists essentially of (i) mixing a fine powder of the active material with a powdered binder and water to form a crumbly dough, (ii) extruding the dough to form thread-like material from the dough and (iii) placing the extrudate in a spheroniser so as to subject the extrudate to circular and tumbling motion whereby the extrudate is broken up into short pieces which then take the shape of granules.

By choice of appropriate formulations, the powders can be extruded into a smooth extrudate which (a) is not friable, (b) does not crumble into a dusty powder in the spheroniser and (c) holds the moisture within the mixture thereby reducing stickiness and the risk of agglomeration.

The extrusion/spheronisation technique is primarily used to produce spheres which: are dense and have a tight particle size distribution; have a relatively smooth surface; enable complete and uniform mixing of the components without risk of segregation; facilitate application of coatings thereon; and protect the active material during storage and transportation.

The compounds of formulae (I) and (II) as herein defined are water-stable, i.e. they do not substantially disintegrate or substantially undergo any undesirable chemical and/or physical change in the presence of or when exposed to small amounts of water and hence they can be subjected to extrusion/spheronisation in aqueous media. An example of such an undesirable change would be the premature decomposition of

(i) a cyclic anhydride structure of formula (I) into an open chain carboxylic compound, or

(ii) a cyclic lactone structure of a compound of formula (II) to expose free carboxyl and/or hydroxyl groups.

A feature of the invention is that the granulated formulation has a high activator content which enables the bleaching function to be carried out more efficiently in comparison with formulations produced conventionally by extrusion and spheronisation where the activator content is usually much lower and the binder content is relatively high.

The following description will illustrate the granulation of a formulation comprising powdered, water-stable bleach activators using an extrusion/spheronisation technique in an aqueous system.

Thus, according to yet another embodiment the present invention is a process for producing a granulated formulation from a water-stable, powdered bleach activator, a binder, a disintegrating aid and/or a filler as appropriate and as hereinbefore described, said process comprising subjecting a dough of suitable consistency made from the components of the formulation and an aqueous medium to the extrusion/spheronisation technique.

As mentioned previously, the activator, binder and other components of the formulation are thoroughly mixed with the aqueous medium, e.g. water, to form a dough. In respect of producing the formulation some general guidance can be useful.

For instance, if insufficient binder is used, the pressure required to extrude the mixture might be too high or the extrudate can disintegrate in the spheroniser to a dusty powder; if the liquid level is too low, the mixture can become difficult to extrude; if the liquid level is too high, this may result in stickiness and agglomeration of the mixture in the spheroniser.

The dough is then extruded by conventional techniques. Extrusion involves forcing the dough through a suitable orifice in order to produce continuously a body of uniform cross-section. The exact technique for forcing the dough and forming the orifice will vary with the type of extruder. For example, one type of extruder that can be used is that which uses a screw to feed the dough to a perforated screen and the dough is extruded through these perforations. In a variant to this system, the dough is fed into a rotating drum in contact with a rotating perforated screen. Other devices that may be used include a piston type extruder in which a movable arm squeezes the dough onto a perforated screen. Yet another device that can be used is extrusion of a dough by the pressure of a ram through a die.

Fundamentally, the material to be extruded must be in plastic condition. This condition may be achieved by choice of a suitable formulation. The size of the orifice would be determined by the ultimate size of the spheres desired and would normally be the approximate final diameter of the spherical product. It would be understood that the particle size of the ingredients forming the dough would be substantially less than the size of the orifice and the particles would ideally be of a diameter which is less than half the diameter of the orifice. Sufficient compaction should be achieved during the extrusion phase to ensure adequate 'green' strength in the extrudate. However, the compaction should not be so great that the subsequent spheronisation and, where applicable, disintegration in use are hampered.

A spheroniser usually consists of a corrugated disc spinning about a vertical axis and the disc is housed in a cylindrical housing with vertical walls along the edges of the disc. When an extrudate is dropped on the spinning disc, the extrudate tends to break into smaller pieces and is simultaneously flung towards the walls of the housing by centrifugal force. This action and the dragging action of the corrugations cause the extrudate pieces to spheronise either by impact or by coalescence with other pieces to form the spheres.

A typical spheroniser is that sold by GB Caleva Ltd. The extrudate is placed on a 20.3 cm disc and spun eg at about 1000 rpm for a few minutes to result in a spherical bound product. The amount of the extrudate placed on the disc will depend on the size of the disc. Too much or too little of the extrudate will not give optimum spheres. For a disc which is 25 cm in diameter, the amount of extrudate placed thereon is suitably from 200-1000 g. This product is then dried at a temperature from 20-60 °C, preferably around 45 °C to drive off the surplus aqueous components leaving behind robust spheres.

The formulations used to produce the dense spheres of active materials bound in a binder can also optionally include other ingredients depending upon the end use of the spheres.

The present invention is further illustrated with reference to the following Examples:

The extrusion of the dough was carried out by loading the dough into an extruder comprising a metallic barrel provided with a ram and a die. The metallic barrel had an internal diameter of 1.5 cm and a length of 25 cm. The dough was packed into this barrel (50 g at a time) then forced through a 1 mm orifice in the die. The length of the orifice was 2 mm. The ram used to extrude the dough force remained essentially steady at 5000N for the full extent of travelled at 100 mm/min and the force applied was about 5000N. The extrusion was performed at ambient temperature.

The above experiment was successfully repeated in a commercial twin-screw extruder manufactured by Russell-Finex. (Model DFSC 60).

The extrudate was then placed in a 20.3 cm diameter spheroniser (ex GB Caleva Ltd) and spun approximately at 1000 rpm. After about 5 minutes, the extrudate had formed into good spheres being substantially from 500-2000 micrometers in diameter. The spheres produced were robust enough to withstand continuous spheronisation of at least 10 minutes without sticking together or breaking down into dusty particles.

Example 1

The following granular formulations were prepared and contained an active (IA), a disintegrant (Acdisol) and a binder and/or filler (ASB 60 clay and/or Avicel PH 101 respectively). The components were thoroughly blended together, extruded and spheronised.

The processing details e.g. timings etc. and apparatus details of extruder and spheroniser used are described above.

	Composition by Weight / g					
Formulation No.	1	2	3	4	5	6
IA	160	160	160	160	480	2000
Acdisol	4	10	4	4	12	50
ASB 60 clay	36	-	18	18	54	225
Avicel PH101	-	30	18	18	54	225
Water	59	56	60	65	186	775
	Composition as Percentage (Wet formulation)					
IA	61.8	62.5	61.6	60.4	61.0	61.0
Acdisol	1.5	3.9	1.5	1.5	1.5	1.5
ASB 60 clay	13.9	-	6.9	6.8	6.9	6.9
Avicel PH101	-	11.7	6.9	6.8	6.9	6.9
Water	22.8	21.9	23.1	24.5	23.7	23.7
	Composition as Percentage After Drying					
IA	80.0	80.0	80.0	80.0	80.0	80.0
Acdisol	2.0	5.0	2.0	2.0	2.0	2.0
ASB 60 clay	18.0	-	9.0	9.0	9.0	9.0
Avicel PH101	-	15.0	9.0	9.0	9.0	9.0
Water	-	-	-	-	-	-

Observations:

Formulation No.1. Extrudate 'worm-like' and continuous. The mixture appeared to be too 'plastic' and the spheres tend to agglomerate on spheronisation; Acdisol too low, no Avicel.

Formulation No.2. Mixture too dry to extrude, indicating poor binding due to absence of clay.

Formulation No.3. Extrudate too hard to form good spheres. Non-spherical and 'dumbell' shaped granules produced. Too dry.

Formulation No.4. Extrudate appeared too wet. Spheres of various sizes produced; perhaps due to excessive water.

Formulation No.5. A good extrudate and regular sized spheres produced.

Formulation No.6. A good extrudate and regular sized spheres produced.

The above observations indicate that to achieve a high active content (80% IA dry matter) together with up to 5% dry matter of dispersant (Acdisol) a mixture of the binder ASB 60 clay and filler Avicel is required to achieve the right balance of plasticity/friability in the extrudate. With a 50:50 ratio of the binder and filler the level of water used appears to be critical for good spheronisation, the preferred ratios of the components being given by formulation No.5. above. Formulation No.6. shows that this ratio can be scaled up successfully.

Example 2

The following formulations were prepared by using a hand held extruder, which was simply a plate which had an aperture 6mm long and 1mm in diameter through which the dough prepared as previously was manually extruded, air drying the extrudate and cutting it into prills of 1-2mm lengths;

	Composition by Weight / g				
Formulation No.	7	8	9	10	11
IA	20	20	20	20	800
Acdisol	2.75	-	2.75	-	100
Avicel	-	2.75	-	2.75	-
ASB 60 clay	2.25	2.25	2.25	2.25	100
Tween 81	-	-	0.5	0.5	20
Water	10	10	10	7	600
	Composition as Percentage (Wet formulation)				
IA	57.1	57.1	56.3	61.6	49.4
Acdisol	7.9	-	7.8	-	6.2
Avicel	-	7.9	-	8.5	-
ASB 60 clay	6.4	6.4	6.3	6.9	6.2
Tween 81	-	-	1.4	1.5	1.2
Water	28.6	28.6	28.2	21.5	37.0

	Composition as Percentage After Drying				
IA	80.0	80.0	78.4	78.4	78
Acdisol	11.0	-	10.8	-	10
Avicel	-	11.0	-	10.8	-
ASB 60 clay	9.0	9.0	8.8	8.8	10
Tween 81	-	-	2.0	2.0	2
Water	-	-	-	-	

Observations on dropping extrudate prills onto tap water in a beaker;

Formulation No.7. Not readily wetted, float on surface. Gradually sink and break up over 1-2 minutes.
Formulation No.8. Not readily wetted, float on surface. No significant disintegration after 2 minutes.
Formulation No.9. Readily wetted, sink instantaneously. Significant disintegration within 15-20 seconds.
Formulation No.10. Readily wetted, sink after several seconds and start to slowly disintegrate.
Formulation 11. This is a scaled-up (1620g) version of Formulation 9 and was carried out on a Russell/Finex extruder and spheroniser.

The above observations show that Avicel PH101 can be replaced by an increased quantity of Acdisol to improve the rate of prill disintegration. The presence of a surfactant greatly increases the rate of disintegration.

Formulation No.9 containing Acdisol and surfactant (Tween 81) showed rapid disintegration.

Example 3

Detergent powders were then prepared which contained:-

82.5%w/w	ECE test base (a detergent base according to BS 1006:CO6:1981)
15.0%w/w	Sodium perborate tetrahydrate
2.5%w/w	Bleach activator, selected from:-

either IA as spheres of formulation No.6.
or IA as prills of formulation No.9.
or TAED

Wash tests were carried out on the detergent powders by dosing them at 6 g/l into 40 ° C water in Terg-o-tometer pots and washing tests cloths stained with red wine and test cloths stained with tea for 20 minutes. The colour of the cloths was measured both before and after washing and the percentage stain removal calculated. The powders gave the following results:

	Percentage Stain removal		
Detergent powder containing 2.5%:-	IA as No.6.spheres	IA as as No.9.prills	TAED
Red wine stain	81	84	74
Tea stain	28	32	23

It is clear from these results that the readily disintegratable prills (formulation No.9.) achieve better performance than the spheres which do not disintegrate readily (formulation No.6).

Example 4

This example demonstrates a formula suitable for extrusion/spheronisation containing a 50:50 blend of the bleach activators TAED and IA. The same techniques as used in example 2 were applied, i.e. hand extrusion.

	Composition by weight / g
Formulation No.	11
IA	10.0
TAED	10.0
Acdisol	2.75
ASB 60 clay	2.25
Tween 81	0.5
Water	13.0
	Composition as Percentage (Wet formulation)
IA	26.0
TAED	26.0
Acdisol	7.1
ASB 60 clay	5.8
Tween 81	1.3
Water	33.8

	Composition as Percentage After Drying
IA	39.2
TAED	39.2
Acdisol	10.8
ASB 60 clay	8.8
Tween 81	2.0
Water	-

Observations:

The extra water in this formulation made the extrudate more compact. The dried prills disintegrated in cold water within 30 seconds.

Example 5:

Crystals of 2-phenyl 3,1 (4H)-benzoxazin-4-one (hereafter "2PB4") (as received, average particle size 100-500 micrometers) were ground in a pestle and mortar to an average particle size of 50-100 micrometers and then in a Raymond Laboratory hammer mill (manufactured by International Combustion Ltd) to an average particle size distribution of less than 30 micrometers.

Powder detergent formulations were prepared using the procedure in Example 1 above, and tested with the formulations and washing conditions shown below. The quantities of the various components used are also shown below. A wet mix was prepared using water and the surfactant shown. This wet mix was then mixed together with the dry components and formed into a dough, which was then extruded on a Caleva model 10 extruder, using a 1mm screen and running at a speed setting of 100. The extrudate was then spheronised on a Calvea model 120 spheroniser, running at 2700 rpm (setting 13). The resulting spheres were dried on a Strea-1 fluid-bed drier for 15 minutes, with an inlet air temperature of 70 ° C. The following granules were prepared:

Mix	Components	Amounts Used	
Dry	2PB4	82g	83g
	ASB60 Clay	13g	14g
	Avicel Filler	3g	3g
Wet	Surfactant Dispex G40	2g	0g
	Water	23g	25g

Also tested were the compartative effects of using commercial TAED, 2PB4 crystals which were milled but were not granulated and a blank sample containing no bleach activator at all.

All the tests were conducted using the following conditions:

Washing machine used: Miele, Programme 4 (Front - loading Automatic)

Temperature:	40 ° C
ECE Base:	70% w/w
Bleach:	15% sodium perborate tetrahydrate
Bleach Activator:	2.5% w/w
Sodium sulphate	Balance to 100%
Dosage of Formulation:	120g (ca. 12g/litre)
Load:	2.5Kg (50% cotton, 50% synthetic fabrics)
Cloths used:	EMPA 114 Red Wine stain & WFK BC-1 tea stain EMPA 115 Immedial Black

	stain
Measurements:	Reflectance measurements were taken with ICS-Texicon Micromatch spectrometer.

The results show the performance of the various types of activators on a percent stain removal basis (%SR) using the formula:

$$\%SR = \frac{L(final) - L(initial)}{L(standard) - L(final)} \times 100$$

L(final) =	final reflectance value
L(initial) =	initial reflectance value
L(standard) =	standard reflectance value

the L value being the reflectance valur as defined in COmmittee Internationale D'Eclarage LAB system (CIELAB)

These results are tabulated below:

Washing Machine trials on various grades of 2PB4 and Comparative Activators:				
Cloth stain	2PB4 as received	2PB4 (fine ground)	TAED	Blank
Red Wine	52	64	63	55
Tea	14	26	25	16
Immedial Black	8	17	9	6

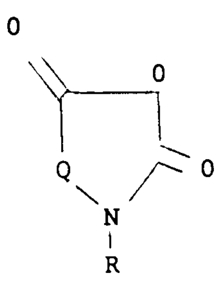
Washing Machine trials on 2PB4 granules according to the invention:				
Cloth stain	2PB4 Hammer Milled		TAED	Blank
	Without Surfactant	With Surfactant		
Red Wine	62	68	67	57
Tea	29	34	26	15
Immedial Black	20	25	8	6

The above results show that the granulated bleach activators are superior to those which have not been ground and granulated.

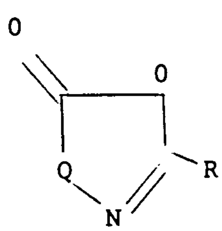
Claims

1. A granulated bleach activator formulation comprising:

a. a powdered, water-stable bleach activator in which at least 50% of the particles have a particle size below 250 micrometers and which bleach activator comprises at least one compound selected from a cyclic anhydride of the structural formula (I) or a lactone of the structural formula (II) below:



(I)



(II)

wherein Q is a divalent organic grouping such that Q and N together with the carbonyl and oxygen functions form one or more cyclic structures and in (I) R is H, an alkyl, aryl, halogen, a carboxylic or a carbonyl containing function, and in (II) R is a C_2 or higher alkyl, alkaryl, aryl, aralkyl, alkoxyl, haloalkyl, alkylamino, dialkylamino, carboxylic or a carbonyl-containing function,

b. a clay binder and

c. a filler and/or a disintegrating aid,

said granules having a particle size in the range of 200 to 2000 micrometers.

2. A granulated bleach activator formulation comprising on a dry weight basis:

a. at least 60% w/w of a water-stable powdered bleach activator in which at least 50% of the particles have a particle size below 250 micrometers and comprising at least one compound of the structural formula (I) or (II) defined as in Claim 1 above,

b. from 5-20% w/w of a clay binder,

c. from 0-15% w/w of a disintegrating aid, and

d. from 0-20% w/w of a filler

such that the total amount of the disintegrating aid, the filler and binder present in the granules is at least 9% w/w, said granules having a particle size in the range of 200-2000 micrometers.

3. A bleach activator formulation according to Claim 1 or 2 wherein the particle size of the powdered bleach activator is below 150 micrometers.

4. A bleach activator according to Claim 3 wherein in the particle size of the powdered bleach activator is from 1-110 micrometers.

5. A bleach activator formulation according to any one of the preceding Claims wherein the bleach activator is a cyclic anhydride of formula (I) in which Q is an arylene group, R is H or a lower alkyl group.

6. A bleach activator formulation according to Claim 5 wherein the bleach activator is isatoic anhydride.

7. A bleach activator according to any one of the preceding Claims 1 to 4 wherein the bleach activator is a lactone of formula (II) in which Q is an arylene group and R represents a C_2 or higher alkyl group, an aryl group with or without further nuclear substituents in said aryl group, an alkylamino group or a dialkylamino group.

8. A bleach activator formulation according to Claim 7 wherein the bleach activator is a lactone in which R is a 2-phenyl group or a 2-dimethylamino group.

9. A bleach activator formulation according to any one of the preceding Claims wherein the binder used is a bentonite clay.

10. A bleach activator according to any one of the preceding Claims wherein the binder is used in an amount from 5-15% w/w.

11. A bleach activator formulation according to any one of the preceding Claims wherein the disintegrating aid is selected from a water-swellable carboxymethyl cellulose derivative, a cross-linked microcrystalline derivative thereof and a polyacrylate salt.

12. A bleach activator formulation according to any one of the preceding Claims wherein the disintegrating aid is used in an amount from 2-11% w/w.

13. A bleach activator formulation according to any one of the preceding Claims wherein the filler is sodium sulphate or cellulosic fillers.

14. A bleach activator formulation according to any one of the preceding Claims wherein the filler is used in an amount from 1-12% w/w.

15. A bleach activator formulation according to any one of the preceding Claims wherein the formulation contains a surfactant in an amount from 0.01-5% w/w based on the dry weight of the total formulation.

16. A process for producing a bleach activator formulation according to any one of the preceding Claims wherein the components of the formulation are kneaded with water into a dough and the dough is subjected to an extrusion/spheronisation process in order to form granules having a particle size in the range from 200-2000 micrometers.

17. A detergent composition comprising granules of a bleach activator formulation according to any one of the preceding Claims 1-15.

European Patent Office

EUROPEAN SEARCH REPORT

Application Number

EP 91 30 9431

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
A	EP-A-0 240 057 (UNILEVER NV) * whole document; particularly page 7, lines 43-46 * – – –	1,3,10,11, 13,17	C 11 D 3/39
A	EP-A-0 053 859 (UNILEVER NV et al.) * abstract; page 6, lines 15-20 * – – –	1	
A	EP-A-0 373 743 (THE CLOROX COMPANY) * abstract; page 15, lines 27-58; page 1, lines 1-38 * – – –	1	
D,A	EP-A-0 332 050 (HENKEL KGAA) – – – – –	1	

TECHNICAL FIELDS SEARCHED (Int. Cl.5)

C 11 D

The present search report has been drawn up for all claims

Place of search	Date of completion of search	Examiner
Berlin	29 January 92	PELLI-WABLAT B

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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