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**ARINOVA Szabadalmi és Védjegy Iroda,
Budapest**(54) **Eljárás nátrium-karbonát festésére és annak alkalmazása mosószer- és tisztítószer-készítményekben**

Az európai szabadalom ellen, megadásának az Európai Szabadalmi Közlönyben való meghirdetésétől számított kilenc hónapon belül, felszólalást lehet benyújtani az Európai Szabadalmi Hivatalnál. (Európai Szabadalmi Egyezmény 99. cikk(1))

A fordítást a szabadalmat az 1995. évi XXXIII. törvény 84/H. §-a szerint nyújtotta be. A fordítás tartalmi helyességét a Szellemi Tulajdon Nemzeti Hivatala nem vizsgálta.



SZTNH-100006511

Description

The object of the present invention is a method for colouring anhydrous sodium carbonate and its use as an aesthetic and detergent additive in detergent and cleaning agent formulations.

Methods for colouring and the use of dyed detergent components have long been known from prior art.

Coloured products within the detergent and cleaning agent industry have lately become ever more important for the consumer, as they desire coloured products that also fulfil a particular aesthetic requirement and make it easier to differentiate between the products of individual manufacturers in addition to an improved washing effect.

Polyphosphates, in particular coloured sodium tripolyphosphates (NTPPs) are therefore for example used in cleaning agents, for example in washing powders or dishwasher detergents. These compounds have been installed into detergent formulations in the form of non-glossy granulates or round particles.

Hydratable anorganic alkanine salts, selected from the group of tripolyphosphates, pyrophosphates, orthophosphates and mixtures of the same are coloured together with a surfactant, for example sodium dodecyl benzene sulfonate, and the corresponding colouring agent or colour pigment in a mixer in US 4,097,418. Colouring agents or colour pigments that form a paste or slurry with a surfactant are used here. This surfactant/colouring agent slurry is then mixed with the phosphate in the corresponding mixing device. Free-flowing coloured granulates with a specific particle size are created in this way. The suitability of sodium carbonate as a particle to be coloured has been mentioned.

A detergent formulation containing solid coloured particles with a so-called "transparency index" of at least 5% and a "sparkle index" of at least 5% is described in application WO-00/27980. Crystalline hydrated anorganic salts, crystalline organic acids, crystalline surfactants, saccharides or their mixtures can be considered as coloured particles, page 5, line 25 ff. A crystalline coloured trisodium citrate dihydrate is preferably used.

The inventors see the advantage of using crystalline salts in that less colouring agent is required due to the surface characteristics of the crystals. The colouring method can be carried out in an aqueous medium as well as in a non-aqueous medium, with the latter not requiring subsequent drying and thus being preferred.

Colouring is here realised in a mixing unit, wherein sodium citrate is mixed with the colouring agent paste. After drying coloured sodium citrate particles with a colouring agent content of 1000 ppm are created.

A method for producing coloured detergent and cleaning agent particles is described in DE 100 48 875, which is characterised in that the colouring agent is admixed with the ingredients as a solution in a solvent agent mixture of water and at least one organic polar solvent.

These ingredients include, amongst others, silicates, all types of zeolites, phosphates, but also

carbonates and/or hydrogen carbonates. Particles according to DE-39 26 253 A1 are used, see examples, page 10. It was found here that the use of a solvent mixture consisting of water and a polar organic solvent can achieve an even colour introduction into the starting components of the particles to be produced, and therefore an even colouring of the finished particles.

Colouring agents used in this method are preferably those that are oxidatively destroyed during the washing process, as well as mixtures of the same with suitable blue colouring agents, so-called blue tones. The detergent produced with this method should be coloured in such a way that the same will not develop too strong an affinity with textile surfaces during use, and in particular with synthetic fibres, page 2, lines 56 ff.

Although sodium carbonates and/or hydrogen carbonates are mentioned as ingredients to be coloured in DE 100 48 875, the method mentioned here has disadvantages.

This is firstly because the colouring agent is admixed with the ingredients as a solution in a solvent mixture consisting of water and at least one polar solvent in the method. The solvent mixture must however be processed again after use, i.e. separated by distillation or disposed of in a costly way under certain circumstances. A closed reactor must also be used for environmental reasons if using organic solvents. The use of water as a reaction medium can be omitted in plants with explosion protection means. This makes the method less complicated, safer for operators and economical.

A method for producing a granulated and coloured material used as a cleaning agent for dentures is introduced in JP 10 298588. The major characteristic of this method is the use of the suitable high-speed mixer and the suitable grain size of the two components, i.e. the component to be coloured and the colouring agent. Only if these lie within a range of 10 to 50 μ will it be possible that both components adhere to and completely cover each other in the mixer [0011]. According to example 1, a colouring agent, namely C.I. acid with a grain size of 80 μ m, is mixed with water-free sodium carbonate with a grain size of less than 300 μ m at a temperature of 25°C and a humidity of 60% for 4 hours in the mixer. In example 2 the same method is carried out with sodium polyphosphate. Sodium polyphosphate as well as sodium carbonate, granulated and coloured, form parts of the cleaning agent. The method described in JP 10 298588 differs from the method of the invention in that it requires the use of a high-speed mixer and the colouring of the components takes place during a solid, dry phase, as well as in the requirement of the grain size distribution of the two components.

There thus existed the task of providing a really simple and environmentally friendly method for producing coloured detergent and cleaning agent components. It is desirable here that such coloured particles are developed for the detergent industry as well as the cleaning agent industry, i.e. for the production of detergents within the textile sector as well as for the production of powdered cleaning agents, for example for automatic dishwashers.

This task is solved by the characteristics of independent claim 1 and the characteristics of the dependent claims.

The coloured product is obtained as a dry powder taken from the mixer. In this method variant

water-soluble colouring agents are used.

According to the invention the water-soluble colouring agents from the compound classes of chinolines, azo dyes, xanthene, indigoids and anthrachinones are used. Colouring agent pigments can however also be used, which are used either as aqueous solutions or as aqueous suspensions depending on their water solubility.

The concentration of the colouring agent solution or the colouring agent suspension is 0.1 to 4.9 wt. %, preferably 0.5 to 2.5 wt. %.

These colouring agents are compatible with the water-free soda with regard to the pH value as well as with regard to their stability and compared to other detergent and cleaning agent ingredients, for example with bleaching agents or optical brighteners.

The soda particles coloured according to the invention can therefore be incorporated without problems into any detergent and cleaning agent recipe.

The water-free sodium carbonate used for colouring according to the invention is commercially available, for example from the company Solvay. It is a special calcinated type with a maximum of 10 wt. % water absorption capacity, which equals $\frac{1}{2}$ mol crystal water content. The carbonate is not crystalline and has no surface sheen, as it is primarily amorphous.

Following the dying process the coloured particles are subjected to sieving and only those that can be found within a particle spectrum of 125 to 1000 μm , preferably 250 to 500 μm , are selected. A subsequent grinding, agglomeration or granulation of therefore not required.

With this very simple process soda particles with a homogeneous colouring are created in a pourable dry form. Heat is released due to the exothermal conduct of the water-free soda during the colouring process. This generated heat is used for drying the product, so that downstream drying become superfluous. The product dried in this way has a maximum crystal water content of 0.5 mol. This type of process management is economical and simple.

The following examples describe the invention in more detail without limiting the same.

Example 1

1300 kg water-free soda with a crystal water content of 0.5 was filled into a Lödige plough blade mixer. An aqueous colouring solution with 4 wt. % Fastusol red was sprayed on evenly by means of a nozzle system. An aqueous pigment suspension, for example of the blue or green pigment or cosmenyl green, can also be used in place of Fastusol red. The reactor chamber is here heated to approx. 50 to 60°C due to the exothermal reaction of the soda with the aqueous colour solution or pigment suspension. The coloured product is obtained already dried, as a free-flowing powder, on the fluid bed and has a maximum crystal water content of 0.5 mol water. It is subsequently sieved. The product has a drying loss of 8.02 %.

Particle size spectrum of the product according to the invention:

Particle size in μm	Content of product according to the invention in %
> 1000	1.5
1000-500	19
500-250	39
250-125	34.5
125-63	5
<63	1

Example 2

2000g water-free sodium carbonate, calcinated in the form of a dry powder, is filled into a mixer as the raw material. 4 g Basovit Green 876 colouring agent is also added to the mixer in a dry form and mixed with the sodium carbonate powder. A Lödige plough blade mixer was used as the mixing aggregate. 196 g water was slowly added during the mixing process. The product is removed after a mixing period of 2 min. The product is homogeneously coloured and is present as a dry free-flowing powder.

Colouring agent	Concentration of colour solution in %	Drying loss at 110°C in %	Crystal water (drying loss)	Colour
Vitasyn orange RGL 90	4.4	8.02	0.5	homogeneous
Fastusol red 53L	4.0	8.02	0.5	homogeneous
Basacid yellow 094	4.4	7.94	0.5	homogeneous
Cosmenyl green GG DLHK	4.0	8.52	0.5	homogeneous
Sandolan brilliant red NB-G-125	2.9	8.6	0.5	homogeneous
Nylosan blue	2.72	8.1	0.5	homogeneous
Cosmenyl blue	4.54	8.9	0.5	homogeneous

ELJÁRÁS NÁTRIUM-KARBONÁT FESTÉSÉRE ÉS ANNAK ALKALMAZÁSA MOSÓSZER- ÉS TISZTÍTÓSZER- KÉSZÍTMÉNYEKBEN

Szabadalmi igénypontok

1. Eljárás nátrium-karbonát festésére (színezésére), **azzal jellemezve, hogy** vízmentes nátrium-karbonát, amelynek képlete Na_2CO_3 , egy folyamatosan működő keverő berendezésben por alakú, vízben oldódó festőanyaggal (színezőanyaggal) van összekeverve, ahol a keverési folyamat közben egy elégséges mennyiségű víz van hozzáadva és ahol az végül el van távolítva a keverő berendezésből egy száraz, szabadon folyó (folyóképes) por alakjában, amely egy 0,5 mól víz maximális kristályvíz-tartalommal rendelkezik.
2. Eljárás az 1. igénypont szerint, **azzal jellemezve, hogy** a festett (színezett) termék szitálva (rostálva) van a festési (színezési) folyamat után oly módon, hogy egy 125 μm -tól 1000 μm -ig terjedő részecskeméret-eloszlás legyen előállítva.
3. Eljárás az 1.-től 2.-ig igénypontok szerint, **azzal jellemezve, hogy** az alkalmazott vízben oldódó festőanyagok (színezőanyagok) a következő vegyület-osztályokból származnak: kinolonok, azofestékek (azoszínezékek), xantének, indigoidok és antrakinonok.



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