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PROCESS FOR THE MANUFACTURE OF DETERGENT COMPOSITIONS CONTAINING SODIUM TRIPOLYPHOSPHATE

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ABSTRACT OF THE DISCLOSURE

In the process for the manufacture of detergent compositions comprising mixing a slurry of detergent components and an active amount of sodium tripolyphosphate and spray drying the resulting mixture, the improvement comprising mixing said slurry with dry sodium tripolyphosphate, at least a portion of the tripolyphosphate being in its hexahydrate form and spray drying the resulting mixture.

Sodium tripolyphosphate in detergent slurries is known to result in the viscosity of the slurry being increased. This increase is the result of the sodium tripolyphosphate undergoing hydration to give hexahydrate. In the manufacture of detergent compositions, attempts are being made with the object of transforming fairly quantitatively the amount of sodium tripolyphosphate used into hexahydrate and of obtaining a final product of good flow properties and stable to storage once the slurry has been sprayed while hot.

When phase-II sodium tripolyphosphate is used, the hydration is found to proceed very reluctantly with a minor increase in viscosity. Prolonged stirring periods are therefore necessary when it is intended to hydrate the tripolyphosphate quantitatively. Sodium tripolyphosphates containing phase-I-material undergo more rapid hydration and accordingly form the hexahydrate more rapidly. This means shorter stirring periods for the slurry. The use of sodium tripolyphosphates containing phase-I-material results, however, in a considerable viscosity increase of the slurry. The higher the content of phase-I-material in the tripolyphosphate, the more rapid the viscosity increase of the slurry. It is customary to use sodium tripolyphosphates containing between 15 and 45% phase-I-material. The processability of detergent slurries being a function of their further treatment, the slurries are not allowed to exceed a given maximum viscosity, determined e.g. by the spraying means used for spraying the slurry, which means that the tripolyphosphates are not permitted to contain phase-I-material in a proportion exceeding a certain upper limiting value. In addition thereto, high contents of phase-I-material are found to result in lump formation in the slurry.

The present invention now provides a process for making detergent compositions containing sodium tripolyphosphate by subjecting a slurry including the detergent components to spray-drying, which unexpectedly enables all of the difficulties resulting from a viscosity increase of the slurry, and the agglomeration of sodium tripolyphosphate containing phase-I-material to be diminished or obviated, which comprises using the sodium tripolyphosphate at least partially in the form of its hexahydrate for preparing the detergent slurry. This results in a constant viscosity being obtained practically immediately and in considerably shorter stirring periods.

Sodium tripolyphosphate is known to undergo hydrolysis via the hexahydrate stage. It was therefore an

unexpected result that sodium tripolyphosphate hexahydrate used in a standard detergent slurry was found to have undergone no appreciable hydrolytic decomposition even after a stirring period of 120 minutes at a temperature of 80° C. A further advantage offered by the process of the present invention resides in the viscosity being readily controllable. Still further, it has been found that the hexahydrate causes the viscosity of detergent slurries to decrease as the temperature drops. Heretofore, when anhydrous sodium tripolyphosphate was used, it was necessary, last not least for reasons of viscosity, to prepare the slurry at high temperatures since the viscosity generally decreased as the temperature increased. However, a disadvantage was found to be associated therewith. The temperature of the slurry could not be increased at will because the polyphosphates underwent extremely strong hydrolysis at temperatures above 80° C. In other words, the hexahydrate enables the reaction to be carried out under more economic conditions at low temperatures in the absence of appreciable hydrolytic decomposition, and enables omitting e.g. the use of dissolving intermediaries.

The hexahydrate should be used in a proportion of at least 5% by weight. Preferably, 50 to 100% by weight of the total amount of sodium tripolyphosphate used for making the detergent slurry should be present in the form of the hexahydrate. In addition to sodium tripolyphosphate, the detergent slurry may contain condensed and, if desired, anhydrous phosphates other than sodium tripolyphosphate, e.g. sodium pyrophosphate or sodium metaphosphate. The detergent slurry should preferably contain 10 to 50% by weight sodium tripolyphosphate or sodium tripolyphosphate hexahydrate. In accordance with the present invention, the detergent slurry is prepared at temperatures between 20 and 90° C., preferably 40 and 60° C.

In summary, the present invention offers the following advantages:

(1) The detergent slurry is immediately obtained at a constant viscosity which means no viscosity problems such as encountered heretofore.
(2) Extremely minor hydrolysis during the preparation of the slurry.
(3) Preparation of the slurry at low temperature as the viscosity is lower at lower temperatures than at higher temperatures.

The following examples illustrate the process of the present invention:

EXAMPLE 1a: (CONVENTIONAL PROCESS)

Detergent slurry:
Solid content: 60% by weight
Temperature: 80° C.

	Grams
Alkyl aryl sulfonate	20
Sodium tripolyphosphate (30% phase I)	45
Sodium silicate	6
Magnesium silicate	4
Sodium sulfate	5
Carboxymethyl cellulose	1
Water	54

Viscosity:							
Time in min	0	10	20	30	40	50	60
Knead resistance in mkp	0	0.04	0.06	0.08	0.09	0.09	009

Hydrolysis:			
Minutes	0	30	60
$Na_5P_3O_{10}$, percent	98.9	93.4	92.2

EXAMPLE 1b: (PROCESS OF INVENTION)

Detergent slurry:
Solid content: 60% by weight [1]
Temperature: 80° C.

	Grams
Alkyl aryl sulfonate	20
Sodium triploy phosphate-hexahydrate	57.7
Sodium silicate	6
Magnesium silicate	4
Sodium sulfate	5
Carboxymethyl cellulose	1
Water	41.3

Viscosity:

Time in min	0	10	20	30	40	50	60
Knead resistance in mkp	0.06	0.06	0.06	0.06	0.06	0.06	0.06

Hydrolysis:

Minutes	0	30	60
$Na_5P_3O_{10}$, percent	97.5	97.5	97.0

EXAMPLE 2a: (CONVENTIONAL PROCESS)

Detergent slurry:
Solid content: 60% by weight
Temperature: 70° C.

	Grams
Alkyl aryl sulfonate	20
Sodium tripolyphosphate (30% phase I)	45
Sodium silicate	6
Magnesium silicate	4
Sodium sulfate	5
Carboxymethyl cellulose	1
Water	54

Viscosity:

Time in min	0	10	20	30	40	50	60
Knead resistance in mkp	0	0.04	0.06	0.08	0.09	0.09	0.09

Hydrolysis:

Minutes	0	30	60
$Na_5P_3O_{10}$, percent	98.9	95.0	94.4

EXAMPLE 2b: (PROCESS OF INVENTION)

Detergent slurry:
Solid content: 60% by weight [1]
Temperature: 70° C.

	Grams
Alkyl aryl sulfonate	20
Sodium tripolyphosphate-hexahydrate	57.7
Sodium silicate	6
Magnesium silicate	4
Sodium sulfate	5
Carboxymethyl cellulose	1
Water	41.3

Viscosity:

Knead resistance in mkp	0	0.05	0.05	0.05	0.05	0.05	0.05

Hydrolysis:

Time in minutes	0	30	60
$Na_5P_3O_{10}$, percent	97.5	97.5	97.0

[1] The water of crystallization contained in the hexahydrate is not included in the solid matter content. In the above Examples, the viscosity was determined by means of a Brabender-type plastograph.

EXAMPLE 3a: (CONVENTIONAL PROCESS)

Detergent slurry:
Solid content: 60% by weight
Temperature: 60° C.

	Grams
Alkyl aryl sulfonate	20
Sodium tripolyphosphate (30% phase I)	45
Sodium silicate	6
Magnesium silicate	4
Sodium sulfate	5
Carboxymethyl cellulose	1
Water	54

Viscosity:

Time in min	0	10	20	30	40	50	60
Knead resistance in mkp	0	0.05	0.06	0.09	0.1	0.1	0.1

Hydrolysis:

Minutes	0	30	60
$Na_5P_3O_{10}$, percent	98.9	97.3	97.0

EXAMPLE 3b: (PROCESS OF INVENTION)

Detergent slurry:
Solid content: 60% by weight [1]
Temperature: 60° C.

	Grams
Alkyl aryl sulfonate	20
Sodium tripolyphosphate-hexahydrate	57.7
Sodium silicate	6
Magnesium silicate	4
Sodium sulfate	5
Carboxymethyl cellulose	1
Water	41.3

Viscosity:

Time in min	0	10	20	30	40	50	60
Knead resistance in mkp	0	0.02	0.02	0.02	0.02	0.02	0.02

Hydrolysis:

Minutes	0	30	60
$Na_5P_3O_{10}$, percent	97.5	97.5	97.0

We claim:

1. In the process for the manufacture of detergent compositions containing between 10 and 50% by weight of sodium tripolyphosphate wherein said sodium tripolyphosphate is mixed with an aqueous slurry of detergent components other than said sodium tripolyphosphate and the mixture obtained is spray dried, the improvement which comprises mixing said slurry with dry crystalline sodium tripolyphosphate of which 50 to 100% by weight is in hexahydrate form and spray drying the resulting mixture.

2. A process as claimed in claim 1, wherein the detergent slurry is prepared at a temperature of 20° to 90° C.

3. A process as claimed in claim 2, wherein the detergent slurry is prepared at a temperature of 40° to 60° C.

4. The process of claim 1, wherein the detergent slurry contains at least one member selected from the group consisting of sodium pyrophosphate and sodium metaphosphate in addition to sodium tripolyphosphate.

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,574,121 Dated April 6, 1971

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It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Column 1, between lines 9 and 10 insert

-- Claims priority, application Germany, May 28, 1965, K 56,246 --

Signed and sealed this 7th day of September 1971.

(SEAL)
Attest:

EDWARD M.FLETCHER, JR.
Attesting Officer

ROBERT GOTTSCHALK
Acting Commissioner of Pat