Contents lists available at ScienceDirect

Chemical Engineering Research and Design

journal homepage: www.elsevier.com/locate/cherd

Two-stage method of obtaining high bulk density sodium tripolyphosphate: Design and mechanism of process

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ABSTRACT

The paper presents a two-step method for obtaining sodium tripolyphosphate (STPP) with a bulk density of about 0.90 kg/dm^3 after the first step, using sodium phosphates after spray drying and water as the raw materials. STPP with a bulk density of 0.95–1.00 kg/dm^3 was generated in the second stage, using STPP from the first step and water as the raw materials. The paper presents statistical analyses to define the process parameters which significantly affect sodium tripolyphosphate bulk density. The determination of the profile approximation and utility function enabled the optimization of process parameters for obtaining a product with a bulk density of 0.95–1.00 kg/dm^3. Mechanisms of increasing bulk density was indicated by studies on the microstructure of the product and phase transformation during the process. The data were empirically verified and satisfactory results were found.

Keywords: Chemical processes; Microstructure; Morphology; Powder technology; Process control

1. Introduction

The detergent market has introduced strict quality requirements for raw materials used in the production of cleaning agents. Detergents have evolved in terms of their content, form and dosage in washing and cleaning processes. The production of innovative household chemical products such as washing tablets and "compact" washing powders is particularly concentrated on the parameters of the raw materials and processes resulting in the production of high bulk density concentrates and products which guarantee relevant cleaning efficiency in low amounts (Showell et al., 2005; Showell, 1997; Tsoler, 2004; Tsoler and Waldhoff, 2004; Zoller and Sosis, 2008). High bulk density sodium tripolyphosphate is particularly useful when applied in both types of detergents.

STPP is widely used in cleaning agents due to its numerous advantageous properties, such as buffering properties, calcium and magnesium ion sequestration, dirt particle deflocculation and dispersion, as well as lipid emulsification (Arai et al., 1965; Köhler, 2001, 2006; Yangxin et al., 2008). Salt hexahydrate ($Na_5P_3O_{10}\cdot 6H_2O$) is a stable form of sodium tripolyphosphate. Anhydrous STPP appears in two crystalline phases – Phase 1 and Phase 2. Phase 1 is formed at temperatures exceeding 450 °C, and Phase 2 at temperatures reaching ca. 400 °C (Corbridge, 1960; Davies and Corbridge, 1958; Dyroff, 1965; Kijkowska et al., 2007).

A high rate of the dehydration process is characteristic for Phase 1. This can be problematic during the detergent production process because the very high rate of STPP hydration causes caking (Kijkowska et al., 2008; Kowalski et al., 2002). New technological solutions in the production of tablets and compact washing powders has enabled the application of sodium tripolyphosphate with a higher content of Phase 1. This significantly reduces hydration time, optimizes the efficiency of the production process and increases the hydrated STPP content in the product.

Sodium tripolyphosphate is mainly obtained in a two-stage process of dehydration in a laboratory spray drier and rotary

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Received 5 April 2013; Received in revised form 7 August 2013; Accepted 17 October 2013

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0263-8762/$ – see front matter
http://dx.doi.org/10.1016/j.cherd.2013.10.016

tubular calciner (Kijkowska et al., 2008; Kowalski et al., 2002; Shrodter et al., 2003). During spray drying, the mixture of orthophosphates obtained as the result of phosphoric acid neutralization with soda ash is condensed into pyrophosphates. The condensation process proceeds according to the following reaction:

$$2NaH_2PO_4 + 4Na_2HPO_4 = Na_2HP_2O_7 + 2Na_4P_2O_7 + 3H_2O/1/$$

The Na_2O/P_2O_5 (TM) molar ratio for the neutralization process is 1.67.

The resulting sodium pyrophosphates mixture is condensed during the calcination process and forms sodium tripolyphosphate:

$$Na_2H_2P_2O_7 + 2Na_4P_2O_7 = 2Na_5P_3O_{10} + H_2O/2/$$

The calcination temperature is the controlling parameter for sodium tripolyphosphate crystalline phases in the end product. Phase I and Phase II sodium tripolyphosphate frequently occur in the product at the same time. The product is also contaminated with tetrasodium pyrophosphate and sodium metaphosphate (Kowalski et al., 2002).

The product properties can be widely modified due to the application of independent drying and calcination reactors. Drying parameters relevantly affect the size of product granules, their shape and mechanical resistance. Spray drying also causes the characteristic "shell" structure of sodium phosphate granules. As a result, the calcination product has a low bulk density of 0.55–0.75 kg/dm^3. The easiest way to increase the bulk density of the sodium tripolyphosphate is its grinding. The ground product has a bulk density of ~0.85 kg/dm^3. However, the process entails a negative effect of receiving the product in dust form, which limits its uses (Banach et al., 2009a, 2009b; Kowalski et al., 2002).

The compacting process is an often used method of increasing the bulk density of sodium tripolyphosphate. Sodium tripolyphosphate, as a product of calcination, can be directly routed to the compacting process, or pre-moistened with water. Compacted material is subjected to the forces occurring between the rotating rolls which are hydraulically biased with pressure of 13–16 MPa. If the process is conducted without the presence of water, the material is given to further calcination at a temperature of about 350 °C. Addition of water to the sodium tripolyphosphate effects of its hydration and obtaining a product having a low content of anhydrous form of STPP. The obtained STPP requires grinding. Its bulk density is about 1.15 kg/dm^3 (Tafler and Mills, 1967).

In the patent literature, for example, the process based on the additive of orthophosphate solution to STPP which was obtained in the process of calcination, drying the mixture at temperatures up to 120 °C and calcination at a temperature of 200–600 °C is described. The resulting product is granulated but has a bulk density of approximately 0.75 kg/dm^3 (Dear, 1972).

The aim of the study was to develop a method for obtaining STPP of high bulk density, determine the mechanism of physicochemical phenomena affecting its changes, and to determine the process parameters that will control the density and other properties of the resulting product (including the contents of the various phosphate phases and the ratio of crystalline phases of STPP). This results from the need to change existing ways of increasing the bulk density of STPP by using a methodology that would allow to obtain a product with appropriate quality-controlled parameters, a higher concentration of the main component, and would reduce the energy consumption of existing processes.

The possibility of obtaining sodium tripolyphosphate with a chemically controlled bulk density and content of Phase I and II will simplify production technology and enable the formation of a perfect component for innovative cleaning agents. In this study, investigations into the phase transitions occurring during the process of obtaining STPP were performed and the characteristic temperature ranges were defined. The study of phase transitions aimed to confirm the possibility of obtaining sodium tripolyphosphate in the present process and to determine the ability to control the phase composition of the resulting product.

The experiments focused on verifying the significance of the impact of process parameters on sodium tripolyphosphate bulk density, determining the function of the study object and using the developed model to predict the output value, i.e. sodium tripolyphosphate bulk density.

Understanding the mechanism of the increasing process of bulk density allows to select the optimum parameters of the tested process and control the functional properties of the product.

2. Materials and methods

The process of obtaining high bulk density sodium tripolyphosphate was the aim of the study. The experiments were conducted in a two-stage process which enables obtaining STPP with a bulk density equal to 0.95–1.00 kg/dm^3. A mixture of sodium phosphates and water were used as the raw materials in the first stage, and granulated STPP from the first stage and water were used in the second stage.

The applied mixture of sodium phosphates as an intermediate was obtained by sodium phosphate solution spray drying in an STPP industrial installation. The solution of sodium phosphates was obtained by the neutralization of wet process phosphoric acid with soda ash. The molar ratio of Na_2O:P_2O_5 for the neutralization process was ca. 1.67.

X-ray diffraction analysis demonstrated that monosodium and disodium orthophosphate double salt ($NaH_2PO_4 \cdot Na_2HPO_4$) present the basic crystalline phase of the applied raw material. The accompanying phases for the double salt are disodium orthophosphate (Na_2HPO_4), disodium orthophosphate dihydrate ($Na_2HPO_4 \cdot 2H_2O$) and monosodium orthophosphate (NaH_2PO_4).

Sodium tripolyphosphate samples with a pre-determined bulk density were prepared by mixing and grinding reagents of known weight ratios in a mortar. The mass was calcined in an electrically heated chamber furnace. A process flow sheet is presented in Fig. 1. Bulk density was measured for the granulometric fractions of the obtained products mixture (fraction % with size [mm]) > 1.00–10.0%; 20% – 0.85–1.00; 5% – 0.60–0.85; 58% – 0.25–0.60 (Procter and Gamble, 1998).

An SDT 2960 Simultaneous DTA-DTG TA thermoanalyzer was used to conduct thermal analysis of the sodium phosphates. The measurements were performed in an air atmosphere. This equipment allows for simultaneously obtaining TG DTG (Thermogravimetric analysis) and DTA (Differential thermal analysis) curves. The DDTA curve was determined by the graphical method. Thermal analysis is a method to study the phase transformations occurring during the process of producing STPP and to determine the characteristic temperature ranges. An X-ray method using a