Synthesis and Characterization of Sodium Tripolyphosphate

N. D Suwarnakar
Student
Department of Chemical Engineering
College of Engineering and Technology Babhulgaon (Jh), Akola, Dist. Akola, Maharashtra, India

Dr. S. N Nemade
Associate Professor
Department of Chemical Engineering
College of Engineering and Technology Babhulgaon (Jh), Akola, Dist. Akola, Maharashtra, India

Dr. P. V Thorat
Associate Professor & Head of Dept.
Department of Chemical Engineering
College of Engineering and Technology Babhulgaon (Jh), Akola, Dist. Akola, Maharashtra, India

Dr. S. G Gaikwad
Scientist
Department of Chemical Engineering & Process Development
Council of Scientific & Industrial Research India

Abstract

Novel Process for the synthesis of sodium tripolyphosphate and its characterization has been investigated in this work. Reaction experiments were carried out in constant stirred batch reactor at atmospheric temperature. Product from the different reactions are analyzed to check its purity and composition and compared. Also the thermodynamic parameters and kinetic data of the reactions is studied in this work. Material flow diagram and material balance for each reaction is studied to select the appropriate and economical reaction to produce the sodium tripolyphosphate. The results show that the reaction of phosphoric acid and sodium hydroxide is economical and produces highly pure sodium tripolyphosphate with minimum amount of waste. Hence finally produced product from economical process is compared with standard results and characterization has been done.

Keywords: Novel Process, Kinetics, Sodium Tripolyphosphate, Flow Diagram

I. Introduction

Chemical production, including the production of condensed phosphates, constitutes a significant burden on the natural environment. One of the condensed phosphates is sodium tripolyphosphate – an essential ingredient in various types of cleaning substances and a food additive [1]. Sodium tripolyphosphate ($Na_5P_3O_{10}$; STPP) is a crystalline inorganic salt that can exist in two anhydrous crystalline forms (phase I and phase II) or a hydrous

Form ($Na_5P_3O_{10}$. $6H_2O$) [1–4] To determine which of the technological solutions for the STPP manufacturing is the most ecologically beneficial, they have been compared with the application of the cumulative calculation method. Sodium tripolyphosphate, commonly used in cleaning substances, performs the function of an active builder and exerts a considerable influence on the processes of washing and cleaning. The raw material for the STPP production is a solution of sodium orthophosphates with the Na_2O/P_2O_5 molar ratio 1.67. Phosphoric acid neutralization commonly obtains it with sodium carbonate. At the commercial scale, STPP is produced from sodium orthophosphate solutions with one or two-step method. At the first variant drying and calcining is carried out in one apparatus, the rotary kiln with the product recirculation most commonly. Such technology is applied by Progil, Piesteritz and Saint-Gobain. At the two-step, option drying takes place in a spray drier while the calcining in the rotary kiln. This method, also called spry-kiln or a classic method is used at Alwernia S.A. Chemical Company, Knapsack and FMC [2]. A new way of STPP manufacturing has recently been developed as well. It is calcining the mixture of crystallized $NaH_2PO_4{\cdot}2H_2O$ and $Na_2HPO_4{\cdot}7H_2O$ also called wet method [3].The products obtained with those three methods have comparable qualities. The main difference is connected with bulk density which has the lowest value for the STPP produced by two-step variant.

This work presents results of research on STPP production with one stage method and using recycling of produced STPP calcined in laboratory rotary kiln. The goal of the investigation was a determination of the appropriate composition of raw materials charges allowed obtaining of STPP with the best quality.

The improvement of energy system efficiency is covering a variety of actions and approaches. These operations, in general, can be described by the tasks of changing the manufacture procedures and improvement of control, unit operations and system integration. One of the methods, which play a significant role in the effort to improve the energy efficiency of industrial plants is Process Integration PI can be defined very widely as ‘‘design, operation, and management of industrial processes with system-oriented and integrated methods, models, and tools’’. Such definition insinuates that proper use of PI methods includes, on the one hand, the fundamental knowledge of thermodynamics, chemical reactions, process control, unit operations, and on the contrary, an understanding and experience in optimizations methods and technical objectives.

Energy savings rarely motivate major projects in the chemical industry. If the energy is not taken into consideration before late in the design project, the possibilities of its savings will be reduced. The restrictions in choices (originate from the fact that other parts of the projects with influence on the energy consumption have been fixed) do not enable a procedure, which handles the PI task in a general manner. Instead of that, in the case of chemical industry, when reduction of the energy consumption is investigated the optimal objectives for PI are often a subjective decision.

The PI task for the improving energy efficiency of sodium tripolyphosphate manufacture, one of the most complex processes in the chemical industry, possesses many qualifications described in previous paragraphs. In the literature review, the author has found only one paper concerning energy analysis of sodium tripolyphosphate manufacture [1], unfortunately with different production route and without the use of computer-aided methods. In spite of this difference, that work was of great help.

This paper will be dedicated to a more detailed description of the process and plant configuration, as well as the explanation of PI methods and software tools, used for the energy efficiency improvement. A detailed description of the mathematical models and the research results will be presented in future papers.

II. Experimental part

This work presents a comparison of the production processes of STPP by the one-step method, with the use of the rotary kiln, the two-step method, and the wet method. The analyzed industrial variants of STPP production are technologies which generate no sewage or waste, hence only the emissions of dust and gases were evaluated.

A. Methods of Production

Sodium tripolyphosphate is produced by following reactions:

$3H_3PO_4 + 5\ NaOH \rightarrow Na_5P_3O_{10}+5H_2O$

$6H_3PO_4+5Na_2CO_3\rightarrow 2Na_5P_3O_{10}+5CO_2+9H_2O$

B. Experimental Materials

The reactants phosphoric acid and sodium hydroxide used were of A.R. grade (99.8%) and were obtained from national chemical laboratory, Pune. The chemicals were used without further purification.

Table – 1
Range of operating conditions

Reaction temperature, K	*330 K, 340 K, 350 K*
Sodium carbonate: phosphoric acid ratio	*5:6, 5:3, 3:1 and 2:1*

The synthesis of sodium tripolyphosphate using phosphoric acid and sodium hydroxide, was carried out in a glass reactor of 500 ml volume. Water bath is used to control the temperature.

C. Experimental Setup and Procedure

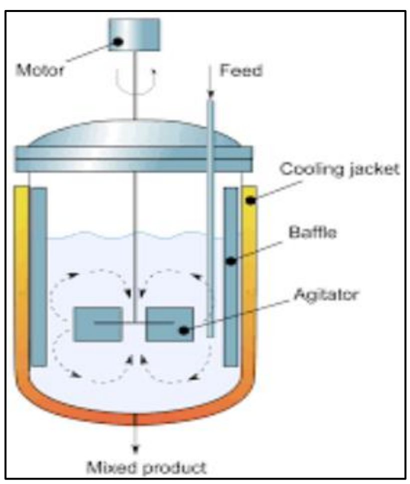

Fig. 1: Batch reactor for synthesis of sodium tripolyphosphate.

In all the experiments, a known amount of phosphoric acid and sodium hydroxide was charged to the reactor and keep it for certain time. As soon as reaction starts heat will evolved due to exothermic nature. To control over the temperature water bath is used. Using thermometer temperature is maintained as less as possible. A sample was removed for analysis. This was taken as the starting point of homogeneous (uncatalyzed) reaction. Samples were withdrawn at regular time intervals for analysis. The volume of sample withdrawn from the reactor during a run was negligible compared with the total volume of the system.

In second reaction, a known amount of phosphoric acid and sodium carbonate was charged to the reactor and keep it for certain time. As soon as reaction starts heat will evolved due to exothermic nature. To control over the temperature water bath is used.

D. Analysis

- Using SEM
- Using X-Ray diffraction method the concentration of the product is found with angle and intensity.

Table – 2
Calcination of different sample at different temperature prepared from reaction of phosphoric acid and NaOH

Sr. No	*Sample no*	*Temperature*	*Time*
1	*Sample 1*	*350^0C*	*2hr*
2	*Sample 2*	*400^0C*	*2hr*
3	*Sample 3*	*450^0C*	*2hr*
4	*Sample 4*	*500^0C*	*2hr*

Table – 3
Calcination of different sample at different temperature prepared from reaction of phosphoric acid and Na2CO3

Sr. No	*Sample no*	*Temperature*	*Time*
1	*Sample 1*	*350^0C*	*2hr*
2	*Sample 2*	*400^0C*	*2hr*
3	*Sample 3*	*450^0C*	*2hr*
4	*Sample 4*	*500^0C*	*2hr*
5	*Sample 5*	*550^0C*	*2hr*

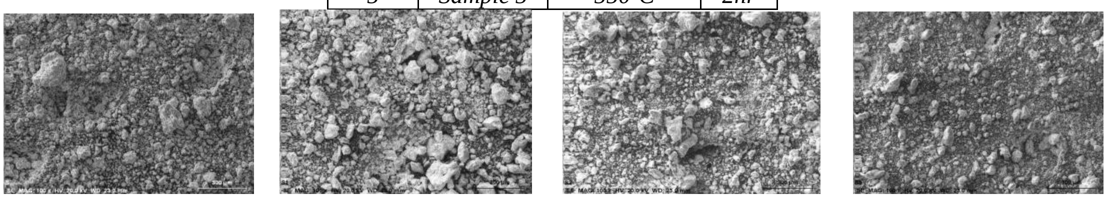

Fig. 2: SEM photos of products from calcinating charges from thermal phosphoric acid and NaOH at the following temperatures: A - 350°C, B - 450°C, C - 550°C

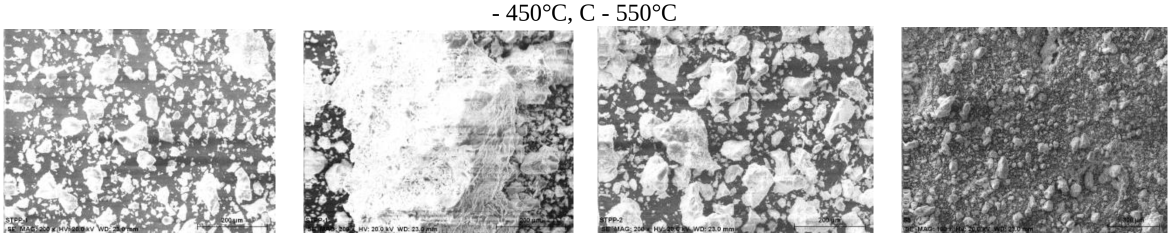

Fig. 3: SEM photos of products from calcinating charges from “Alwernia” WPPA and Na2CO3 at the following temperatures: A - 350°C, B - 450°C, C - 550°C

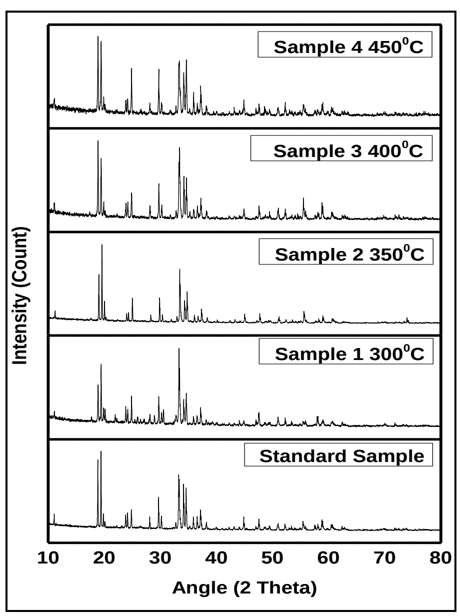

Fig. 4: XRD patterns of $Na_5P_3O_{10}$ powders ignited at different temperatures in comparison to standard $Na_5P_3O_{10}$

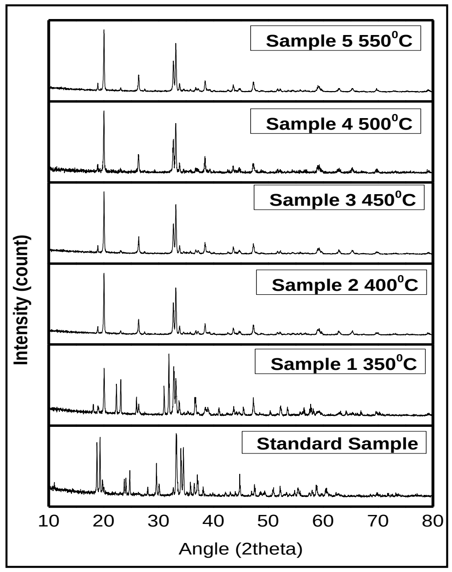

Fig. 5: XRD patterns of $Na_5P_3O_{10}$powders ignited at different temperatures in comparison to standard $Na_5P_3O_{10}$

III. CONCLUSIONS

Earlier studies of the reaction between sodium carbonate and sodium hydroxide with phosphoric acid was considered to be better as it produces sodium tripolyphosphate economically. As there are many ways to synthesis the sodium tripolyphosphate some of them are hazardous some of them produces more waste or by product. Hence, reaction between sodium carbonate and sodium hydroxide with phosphoric acid will synthesis sodium tripolyphosphate with less waste and no by product with the final conversion of 96%. Also, if the mole ratio of sodium carbonate and sodium hydroxide increases it will give higher conversion product. Finally this reaction is economical and should be used for the synthesis of tripolyphosphate with the characterization of the product.

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