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ZDZISŁAW JAWORSKI*, IGNACY SARZYŃSKI**, MICHAŁ DYLĄG***

A CONCEPT OF PILOT PLANT FOR INNOVATIVE PRODUCTION OF AMMONIUM FERTILIZERS

KONCEPCJA INSTALACJI PILOTAŻOWEJ DO INNOWACYJNEJ PRODUKCJI NAWOZÓW AMONOWYCH

A b s t r a c t

Basic features of a novel concept for production of ammonium fertilizers was presented in the paper. The key elements of a pilot plant and their roles in testing the concept were briefly described. Important aims of implementation of the pilot plant were also outlined. Six main units constituting the pilot installation were presented along with four basic stages of the project. The project targets were divided into six work packages and 15 individual tasks to be finalized within two years.

Keywords: novel production method, ammonium fertilizers, pilot plant

S t r e s z c z e n i e

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Słowa kluczowe: nowa metoda produkcji, nawozy amonowe, instalacja pilotażowa

* Prof. PhD. Eng. inż. Zdzisław Jaworski, Institute of Chemical Engineering and Environmental Protections Processes, West Pomeranian University of Technology, Szczecin.

** MSc. Eng. Ignacy Szarzyński, Retired design engineer.

*** Prof. PhD. Eng. Michał Dyląg, Institute of Advanced Manufacturing Technologies, Cracow.

1. Introduction

Inorganic fertilizers typically contain three basic elements, which are important macro-nutrients for crops. Those elements are nitrogen N, phosphorus P and potassium K. Especially valuable are those fertilizers that contain high amount of active components and little ballast – inactive compounds. One of the fertilizers rich in active components is DAP, which mainly consists of diammonium phosphate, $(NH_4)_2PO_4$. The standard industrial grade of DAP is 18-46-0 counted as N-P_2O_5-K_2O weight percentage. The fertilizer is usually manufactured by ammonization of ortophosphoric acid derived in wet-process of extraction from phosphorus containing ores. Several technologies of industrial DAP production are used in Europe ranging from classical Dorr-Oliver method to modern processes of Uhde GmbH or Norsk Hydro [1].

The formation reaction of the diammonium phosphate occurs in two stages presented in eqs. (1) and (2).

$$NH_3 + H_3PO_4 = NH_4H_2PO_4 \tag{1}$$

$$NH_3 + NH_4H_2PO_4 = (NH_4)_2HPO_4 \tag{2}$$

The total enthalpy of the exothermal reactions (1) and (2) equals to $\Delta h = -193$ kJ/mol [1].

The published methods of DAP manufacturing employ various reactors fed with phosphoric acid and a necessary excess of ammonia to achieve full di-ammonium salt. That excess is usually recovered in an absorption unit. However, according to current Best Available Technologies (BAT) the losses of ammonia in typical DAP installations in Europe range from 5 to 30 mgNH_3/ Nm3 in exit gases [2]. The high reaction enthalpy is utilised for autothermal evaporation of water included in extracted phosphoric acid. The developed steam contains the excess of ammonia and the vapour stream is usually delivered to absorption unit.

2. Essence of the method

The innovative method for manufacturing of DAP and DAS (diammonium sulphate) consists in a two-stage, counter-current contacting of the basic substrates, i.e. ammonia and a relevant acid, be it phosphoric or sulphuric acid or a mixture of both. The flow scheme of a production plant is presented in Fig. 1 [3]. Results of a survey of patents and other open literature indicate novel features of the two-stage production method.

The innovative method of manufacturing the di-ammonium salt fertilizers distinguishes by the following characteristics from the existing methods: (i) significantly lower acidity than mono-ammonium salts and a high content of nitrogen in the fertilizer, (ii) possibility of utilization of the exothermic reaction heat for generating steam of about 0.6 kg/kg of fertilizer, (iii) decreasing losses of ammonia and water, (iv) lowering energy demand for product granulation, (v) essentially wasteless production. The features signify a considerable progress as compared with the classical methods, however it requires application of pressure equipment that operates at elevated temperature and in corrosive environment. In particular, the reaction liquid/suspension mixture from Stage 1 has to be compressed by pump, P, from the pressure level of Stage 1 to that of Stage 2. The main product exiting Stage 2 can be either directly sprayed over a granulation bed or solidified by cooling the product suspension.

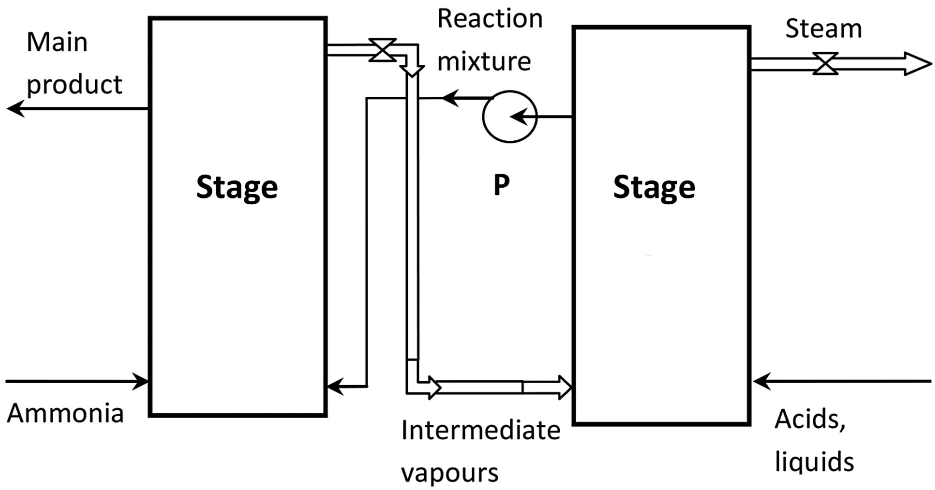

Fig. 1. Sketch of the stream flows in the two-stage ammonization process

Rys. 1. Schemat przepływu strumieni w dwustopniowym procesie amonizacji

Our research team at West-Pomeranian University of Technology accomplished in 2011 a research and development project [4] that was funded by The National Centre for Research and Development. The project was dedicated to advance the innovative concept by testing in laboratory the basic thermodynamic assumptions of the method and to approximately define operating conditions for the reactors of Stage 1 and Stage 2. Analytical tools such as differential thermal analysis, X-ray diffraction and FTIR spectroscopy were used to analyse ammonium salts obtained by ammonization of acids at elevated temperature and pressure. The experiments were complemented by model analysis of thermodynamic equilibrium in 3-phase systems NH_3-H_3PO_4-H_2O and NH_3-H_2SO_4-H_2O. The results of the R&D project fully confirmed thermodynamic correctness of the concept and indicated a need of establishing favourable levels of working conditions by testing the efficacy of such a system in the continuous mode of operation, preferably in a pilot plant. This gave rise to the idea of a next R&D project associated with a continuously operated pilot plant. A mathematical model of the material and energy balances was developed for pilot plants of different productivity. Other preparation steps of the project are described in the following section.

3. Description of the proposed project

Designing, assembly and tests of pilot plant

Three principal direct aims of the project were set up:

1) estimate the ammonization efficiency and operational characteristics of 2–4 preselected geometrical configurations of both 1st and 2nd Stage reactors,
2) establish workable procedures of automatic control of the two counter-current reactors,
3) determine effects of quality of the technical media on incrustation and corrosion of the two reactors.

It was also planned that the programme of designing, building and running of the pilot plant should result in the level of knowledge that enables us to draw up the basic engineering documentation for a full scale installation.

Therefore, development of a concept and a time schedule for the pilot plant was undertaken. Following literature records [5, 6] and other helpful information [7] the project was planned for four basic stages:
1) pre-basic engineering,
2) basic engineering,
3) commissioning and building,
4) start-up and test runs.

In principle, those four stages were planned for completion in 24 months. The pilot plant was intended to use technical grade media and operate continuously with the substrates drawn from their storage containers independent of external sources, which are prone to uncontrolled disturbances. Such a plant is commonly realised as a parallel line to the full-scale industrial installation with the pilot product being utilised in the installation and located in a mobile container [8]. A general block scheme of key components of the studied pilot plant is presented in Fig. 2.

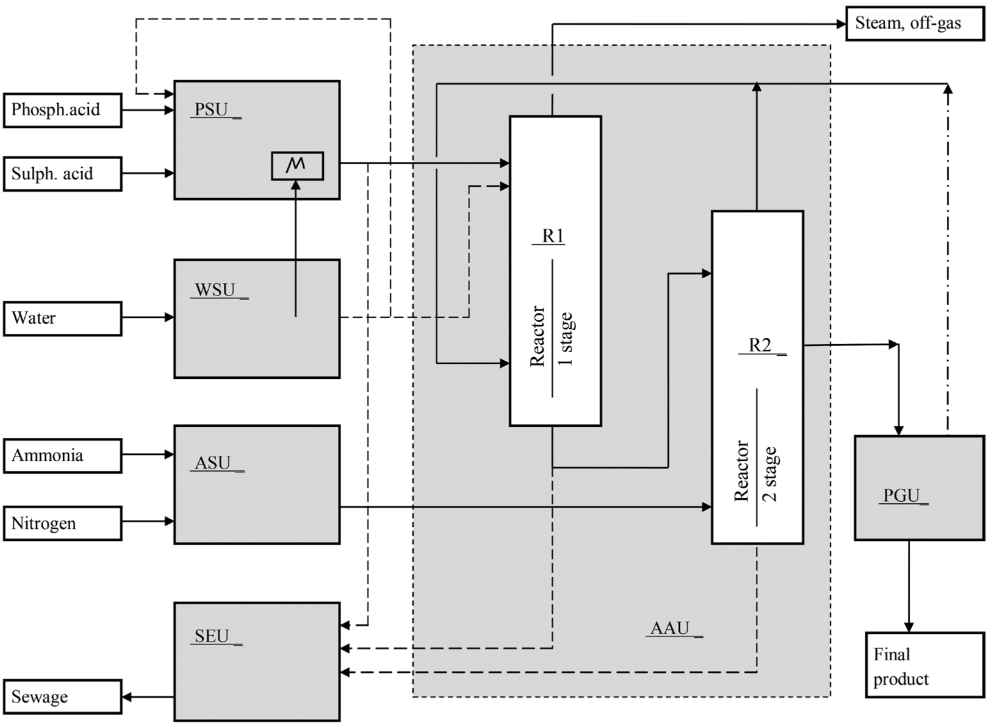

Fig. 2. Schematic block representation of the pilot plant

Rys. 2. Schemat blokowy instalacji pilotażowej

The pilot installation is composed of the following main parts: PSU − acid supply unit, WSU − water supply unit, ASU − ammonia supply unit, SEU − sewage evacuation unit, AAU − acid ammonization unit, PGU − product granulation unit. The sewage unit was intended to operate only during cleaning periods.

The standard set of substrates delivered to the pilot plant should comprise: liquid ammonia, phosphoric (and/or sulphuric) acid, water and compressed nitrogen as auxiliary agent to discharge ammonia. Two reactors; R1 of Stage 1 and R2 of Stage 2 are the key elements of the plant. The main product leaving the plant is in form of a concentrated suspension or molten salt and the by-product is low-pressure water steam slightly contaminated with ammonia. Water in the plant is used for two purposes: as dilutor of acids in the R1 reactor and as cooling agent for a direct granulation unit, if applied. Electric energy is used to drive motors in the pilot plant and for its automatic control.

Engineering realisation of the pilot stage of the development of the new concept of manufacturing the ammonium fertilizers was designed in six work packages, P0 through P5, summarised in Table 1.

Each package was split in 2–4 specific tasks and every task was assigned a definite time span with a starting month and finalizing month of the running project. In addition, a milestone of specific deliverable, denoted by "●" in Table 1, was ascribed to each of the 5 finished packages, P1 to P5, to facilitate keeping up the schedule. Three intermediate reports, I, and the final one, F, were also planned as archive documentation of the major project steps, P2, P3, P4 and P5.

Table 1

Packages and tasks of the project

Package number, name (months)	Task number, name		Start month	End month
P0, Project management (1–24)	T01, Project & Group 1 management		1	24
	T02, Management of Group 2		1	24
P1, Pre-basic engineering (1–3)	T11, Organising and technical preliminaries		1	2
	T12, Internal and external agreements		2	3 ●
P2, Basic engineering of pilot plant (4–10)	T21, Process design		4	9
	T22, Cost analysis		8	9
	T23, Internal and external permits	I	8	10 ●
P3, Detail engineering of pilot plant (6–20)	T31, Ordering and supply of plant elements		6	14
	T32, Construction design and plant assembly		12	18
	T33, Mechanical and technological start-up		17	20
	T34, As-built documentation	I	18	20 ●
P4, Tests of plant at factory (20–23)	T41, Tests & optimisation runs using technical media		20	23
	T42, Report on pilot plant tests	I	22	23 ●
P5, Final report (21–24)	T51, Pre-basic engineering for full scale		21	24
	T52, Final project report	F	22	24 ●

4. Concluding remarks

Five characteristic features of the novel, two-stage manufacturing method for ammonium fertilizers were outlined. Basic targets of the prepared R&D project involving a pilot plant were presented as a continuation of an earlier project. The pilot plant was defined in functional block terms with the main 2-reactor unit for pressure ammonization of acids and with five auxiliary units. Four engineering stages – work packages of the project were set out to form the full project programme together with two additional packages. The programme was split into 15 detailed tasks to be accomplished within two years with 5 milestones and 4 reports predicted.

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