Method and plant for continuous production of granular USP nitrogen and phosphate type fertilizers and products on their basis

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This paper presents results of research on a method for production of granular USP (urea-superphosphate) nitrogen and phosphate fertilizers, in a continuous manner.

The use of urea as a component of solid complex fertilizers allows to obtain high total nutrients content in fertilizers and to increase its yield efficiency. Therefore, various attempts have been taken previously to launch production of complex fertilizers containing urea. Projects of this type included attempts to obtain complex fertilizers using superphosphates and urea. Such activities have been conducted by various companies and, up to the 1990s, relied mainly on attempts to use intermediates, i.e. superphosphates and solid urea, for the manufacture of NP and NPK fertilizers (Guelfi et al., 2022). In such technologies the urea induced liquefaction of granular mixtures forces the application of a series of economically adverse and technically troublesome solutions in the individual stages of a fertilizer manufacturing process. These are:

- conducting the granulation and drying at significantly reduced temperatures, which forces using an increased amount of the drying agent and increasing dryer size,
- large recycle, which translates into an increase in consumption of energy to move recycling masses,
- applying special solutions in the granulation unit in order to obtain optimum temperature in connection to the use of the cold recycle,
- the risk of more frequent plant shutdowns for individual equipment cleaning.

A significant improvement in the production of fertilisers containing urea and superphosphate components has been achieved as a result of work coordinated by Grande Paroisse (currently Borealis). The USP production process, according to the first information from Grande Paroisse (Schwob, 1990), is similar to the production of the single superphosphate (Achard, Schwob, Limousin, 1994). It comprises the following steps: preparation of reactant solution (sulphuric acid solution for single superphosphate and urea solution in sulphuric acid for USP), reaction of the obtained solution with phosphate, aging and granulation of the solid product without/with additives (Achard et al., 1991), (Swissa, Ben-Joseph, 2000).

Methods for the production of granular fertilizers of the USP type and granular products containing USP, which require so-called aging, have significant drawbacks. These are:

- storage warehouse for the aging process,
- handling operations consisting in raking the aging intermediate product or the final product in order to prevent caking of the whole mass, and to prevent spontaneous heat-up of the whole mass,
- installation of an air purification system in the storage facility (emission of fluorine compounds),
- safety threat by the course of exothermic reactions, mainly between urea and sulphuric acid.

In addition, the research shows that the time of aging and the properties of the semi-finished products depend on the type of phosphate raw material used and the composition of the reactant solution. The mentioned nuisances (construction of warehouses, additional operations) are serious disadvantages of the method and make it impossible to run the process continuously.

The method described below allows to avoid above mentioned disadvantages by using the suspension granulation method for the continuous production of USP type fertilizers or USP-based NPK fertilizers. According to this method, the process is carried out using the slurry method in a continuous manner in a typical process line for the production of compound fertilizer while maintaining precisely defined process parameters.

The following main raw materials were used in the production of the USP nitrogen-phosphorus fertilizer: phosphate rock (different types) - wt% P_2O_5,~ 30; sulfuric acid - concentration 95.0%; urea - N wt% 46.0; and additional raw materials: magnesite - wt% MgO 30-44; ammonia; SBO - surfactant; process water.

The study carried out research on the production of granulation pulps and fertilizer granulates on a laboratory scale and in a semi-technical scale plant for testing fertilizer's granulation.

Three desirable reactions can be distinguished during the USP manufacturing process: dissolving urea in a sulphuric acid solution:

$$H_2SO_4 + CO(NH_2)_2 = CO(NH_2)_2 \cdot H_2SO_4 \quad (1)$$

$$CO(NH_2)_2 \cdot H_2SO_4 + CO(NH_2)_2 = 2CO(NH_2)_2 \cdot H_2SO_4 \quad (2)$$

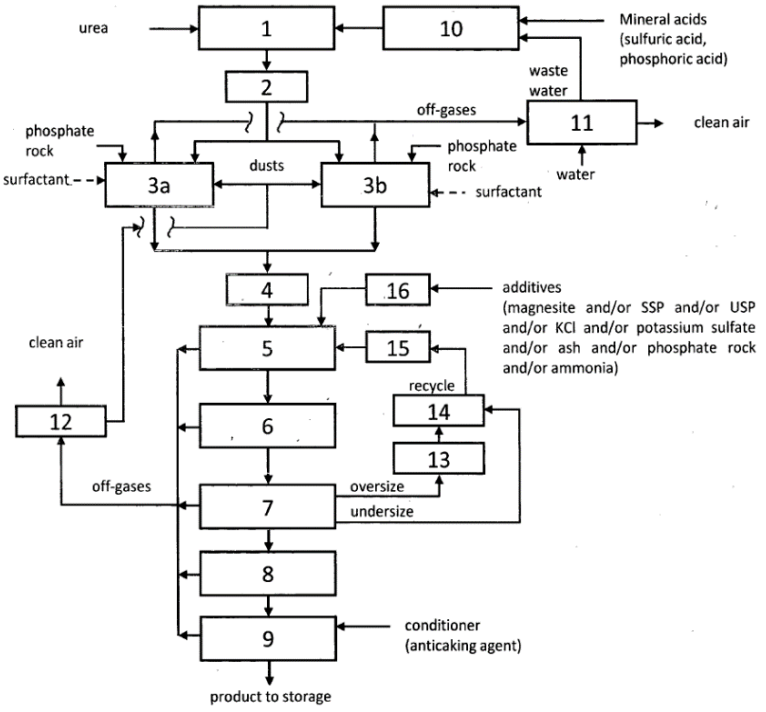
and the basic reaction described by the equation (3):

$$Ca_{10}(PO_4)_6F_2 + 6\,[H_2SO_4 + 3{,}6CO(NH_2)_2] + 3\,H_2O = 3Ca(H_2PO_4)_2 \cdot H_2O + CaF_2 + 5{,}4[CaSO_4 \cdot 4CO(NH_2)_2 + 0{,}6CaSO_4 \quad (3)$$

The rate of this reaction (3) depends on the type of phosphate raw material, its particle size distribution, chemical composition of the reactant solution (molar ratio $CO(NH_2)_2$: H_2SO_4 : H_2O and the possible participation of phosphoric acid), temperature, reactants mixing intensity, etc.
The tests carried out to produce a urea superphosphate fertilizer were aimed at: i) assessment of the transport and granulation properties of the pulps produced, ii) determination of technological and process parameters, iii) assessment of product physicochemical properties.
Table 1 Physicochemical analysis results of USP average sample prepared in tests on semi-technical scale.

Total wt%	N	N_{NH3}	P_2O_5	$P_2O_{5\,(ws+c)}$	$P_2O_{5\,(ws)}$	CaO	SO_4	H_2O	Crush strength, kG	Median diameter, mm
USP	20,15	0,89	9,16	7,61	5,52	12,31	23,43	1,77	4.5	3.81

As a result of the conducted research, a technological line for the USP fertilizer production process was developed using the pulp method with continuous dosing of raw materials and the parameters of the main process nodes were selected. The process parameters in the granulation tests were as follows: i) granulating pulp temperature in the range of 75-90°C, ii) temperature in the granulation unit about 60-75°C, iii) recycle (undersize and oversize particles after grinding) temperature around 60-80°C, iiii) finished product temperature: 75-110°C
Fig. 1. Block diagram of urea superphosphate fertilizer type with continuous feeding of raw materials.



An industrial plant presented on the block diagram (Fig. 1), consists of the following components:
- reactor-mixer 1 for the production of reactant solution,
- reactant solution dispenser 2,
- pulp reactors 3a,b,
- pulp dispenser 4,
- granulator set 5 consisting of a granulator-mixer and a plate granulator,
- rotary dryer 6 supplied co-currently with heated air,
– a set of sieves 7,
- cooler 8,
– conditioning drum 9,
- acid thinner/mixer 10,
- wet-treatment unit: scrubber 11,
- dry treatment unit: cyclone 12,
- oversized material mill 13,
- recycle buffer tank 14,
- recycle dispenser 15,
- additional raw materials dispenser 16.

Conclusions

The main advantages of the present method are:
- ability to conduct the continuous production, avoiding the necessity of additional storage and additional operations in the production process, related to the aging,
- possibility to use sulphuric acid with lower concentration, e.g. recovered from various industries,
- reduced process security risks by using the reactant solution with lower concentration and by completed reaction of sulphuric acid with rock phosphate during preparation of the process pulp,
- mitigation of the risk of formation of substances harmful to plants by using reactive solution with lower concentration,
- in order to avoid dependence on the type of raw material, the use of two slurry reactors operating alternately, is preferred.

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