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

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Purpose of work:

Development of a new continuous method for production of granular USP (urea-superphosphate) nitrogen and phosphate fertilizers and products based on it, in a continuous manner.

**USP – urea superphosphate**

EU fertilizer type: "NP fertilizer with calcium and sulfur"

**NP (CaO; SO₃) 20-10 (12;19)**

the main components of the fertilizer are: urea, ammonium sulphate, calcium phosphates, calcium sulphate
Flow diagram of semi-pilot plant at Research Centre at Ł-INS
Semi pilot plant – spatial view
A system of reactors for melts and slurries production
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A system of reactors for melts and slurries
A system of dosing raw material
Granulation unit

- Paddle granulator
- Pan granulator
- Drum granulator
- Pumping system
- Belt conveyors
Granulation unit
Granulation unit
Drying unit

ST - belt dryer
SB - drum dryer
ChT - belt cooler
KW - roller crusher
PW - vibrating screen
Drying unit - belt dryer

- 3 belt decks 5 m long and working width 70 cm,
- drying air from a steam heater,
- the flow of drying air distributed in a labyrinth through successive decks, starting from the lower one.
Drying unit – drum dryer

- a drum with a diameter of 70 cm and a length of 4 m,
- paddle system designed by our team,
- drying with air from a steam heater or natural gas fumes,
- optional co-current and counter-current drying air flow,
- adjustable rotation speed and tilt of the drum.
Raw materials used in a research

Pulp of USP:

- **Phosphate raw material (Algerian phosphate rock)**
  - $P_2O_5$ tot. – 30,18% wt.
  - CaO tot. – 48,49% wt.

- **Urea** – urea prill (from G.A. Z.A. „Puławy” S.A.)
  - N tot. – min. 46, 0% wt.
  - biuret – max. 1,2% wt.
  - % $H_2O$ – max. 0,3% wt.

- **Sulfuric acid**
  - concentration 94,0%

- **Water**
Raw materials used in a research

Other raw materials and additives:

• Magnesite
  – MgO tot. – 30-44%

• Ammonia (from G.A. Z.A. „Puławy” S.A.)

• SBO - antifoam agent
  (a mixture of dioctyl sodium sulfosuccinate, water and octyl alcohol)

• Water
Chemistry of the process

Three desirable reactions can be distinguished during the USP manufacturing process:

1. \[ \text{H}_2\text{SO}_4 + \text{CO(NH}_2\text{)}_2 \rightarrow \text{CO(NH}_2\text{)}_2\cdot\text{H}_2\text{SO}_4 \]  
   \[ \text{(1)} \]

2. \[ \text{CO(NH}_2\text{)}_2 + \text{CO(NH}_2\text{)}_2\cdot\text{H}_2\text{SO}_4 \rightarrow 2\text{CO(NH}_2\text{)}_2\cdot\text{H}_2\text{SO}_4 \]  
   \[ \text{(2)} \]

3. \[ \text{Ca}_{10}(\text{PO}_4)_6\text{F}_2 + 6[\text{H}_2\text{SO}_4 + 3,6\text{CO(NH}_2\text{)}_2] + 3\text{H}_2\text{O} \rightarrow 3\text{Ca(H}_2\text{PO}_4)_2\cdot\text{H}_2\text{O} + \text{CaF}_2 + 5,4[\text{CaSO}_4\cdot4\text{CO(NH}_2\text{)}_2] + 0,6[\text{CaSO}_4] \]  
   \[ \text{(3)} \]
Semi-technical trials – verification tests

The process parameters in the granulation tests were as follows:

- granulation pulp temperature 75-90°C
- recycle temperature: 60-70°C
- the temperature in the granulation unit: 55-70°C
- product temperature after drying: 85-95°C

The tests carried out to produce a urea superphosphate fertilizer were aimed at:

i) assessment of the transport and granulation properties of the pulps,

ii) determination of technological and process parameters,

iii) assessment of product physicochemical properties.
Product and process

Ø (2~5) mm
Research and analysis results

Table 1. Physicochemical analysis results of USP average sample prepared in tests on semi-technical scale.

<table>
<thead>
<tr>
<th>Total wt%</th>
<th>N</th>
<th>N$_{NH3}$</th>
<th>P$_2$O$_5$</th>
<th>P$_2$O$_5$ $(ws+c)$</th>
<th>P$_2$O$_5$ $(ws)$</th>
<th>CaO</th>
<th>SO$_4$</th>
<th>H$_2$O</th>
<th>Crush strength, kG/granule</th>
<th>Median diameter, mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>USP</td>
<td>20.15</td>
<td>0.89</td>
<td>9.16</td>
<td>7.61</td>
<td>5.52</td>
<td>12.31</td>
<td>23.43</td>
<td>1.77</td>
<td>4.5</td>
<td>3.81</td>
</tr>
</tbody>
</table>

$N_{tot}$ 20.15, $P_2O_5$ tot. 9.16, $P_2O_5$ $(ws+c)$ 7.61
Main parameters of the technological process

- Temperature in the reaction solution reactor – 65-70°C
- Temperature in the pulp reactor – 80-90°C
- Temperature in the granulation node – 60-70°C
- Water content in the granulate before the drying process 5-6% wt.
- Granulate temperature at the outlet from the dryer – 85 - 95°C
- Ratio of recycled to granulating pulp ~ 6:1
An industrial plant presented on the block diagram, 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.
Process diagram
Process diagram
Process diagram
Granulation pulp of USP
Summary

The production process is feasible.

The main advantages of the presented 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 phosphorite, the use of two slurry reactors operating alternately, is preferred.
Summary

The main advantages of the presented method are:

- the two-stage granulation method (granulator-mixer and flow granulator, i.e. drum granulator or pan granulator) allows to increase the share of the specific fraction of the product, which translates into reduced recycle and energy consumption of the process, as well as reduced conversion of urea in side reactions leading to the formation of products harmful to plants and the retardation of phosphorus.

- it is advisable to use additives supporting the process, i.e. magnesite and ammonia, they increase the thermal resistance of granules during the drying process, especially at elevated temperatures (after exceeding the product temperature of 100°C at the dryer outlet, the strength of the granules drastically decreases and they become brittle, and later plastic and sticky).
Thank you for attention!

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